INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

ANALYTICAL CHEMISTRY DIVISION
COMMISSION ON SOLUBILITY DATA

SOLUBILITY DATA SERIES

Volume 22

SCANDIUM, YTTRIUM, LANTHANUM AND LANTHANIDE HALIDES IN NONAQUEOUS SOLVENTS

SOLUBILITY DATA SERIES

Volume 1	H. L. Clever, *Helium and Neon*
Volume 2	H. L. Clever, *Krypton, Xenon and Radon*
Volume 3	M. Salomon, *Silver Azide, Cyanide, Cyanamides, Cyanate, Selenocyanate and Thiocyanate*
Volume 4	H. L. Clever, *Argon*
Volume 5/6	C. L. Young, *Hydrogen and Deuterium*
Volume 7	R. Battino, *Oxygen and Ozone*
Volume 8	C. L. Young, *Oxides of Nitrogen*
Volume 9	W. Hayduk, *Ethane*
Volume 10	R. Battino, *Nitrogen and Air*
Volume 11	B. Scrosati and C. A. Vincent, *Alkali Metal, Alkaline Earth Metal and Ammonium Halides. Amide Solvents*
Volume 12	C. L. Young, *Sulfur Dioxide, Chlorine, Fluorine and Chlorine Oxides*
Volume 13	S. Siekierski, T. Mioduski and M. Salomon, *Scandium, Yttrium, Lanthanum and Lanthanide Nitrates*
Volume 14	H. Miyamoto, M. Salomon and H. L. Clever, *Alkaline Earth Metal Halates*
Volume 15	A. F. M. Barton, *Alcohols with Water*
Volume 16/17	E. Tomlinson and A. Regosz, *Antibiotics: I. β-Lactam Antibiotics*
Volume 18	O. Popovych, *Tetraphenylborates*
Volume 19	C. L. Young, *Cumulative Index: Volumes 1–18*
Volume 20	A. L. Horvath and F. W. Getzen, *Halogenated Benzenes, Toluenes and Phenols with Water*
Volume 21	C. L. Young and P. G. T. Fogg, *Ammonia, Amines, Phosphine, Arsine, Stibine, Silane, Germane and Stannane in Organic Solvents*
Volume 22	T. Mioduski and M. Salomon, *Scandium, Yttrium, Lanthanum and Lanthanide Halides in Nonaqueous Solvents*

Selected Volumes in Preparation

T. P. Dirkse, *Copper, Silver, Gold, and Zinc, Cadmium and Mercury Oxides and Hydroxides*
W. Hayduk, *Propane, Butane and 2-Methylpropane*
H. L. Clever and W. Gerrard, *Hydrogen Halides in Nonaqueous Solvents*
H. Miyamoto, *Copper and Silver Halates*
J. W. Lorimer, *Beryllium, Strontium, Barium and Radium Sulfates*
H. L. Clever and C. L. Young, *Carbon Dioxide*
H. L. Clever and C. L. Young, *Methane*

SOLUBILITY DATA SERIES

Editor-in-Chief
A. S. KERTES

Volume 22

SCANDIUM, YTTRIUM, LANTHANUM AND LANTHANIDE HALIDES IN NONAQUEOUS SOLVENTS

Volume Editors

T. MIODUSKI
Institute of Nuclear Chemistry
and Technology, Warsaw, Poland

M. SALOMON
PO Box 254
Fair Haven, NJ, USA

PERGAMON PRESS
OXFORD · NEW YORK · TORONTO · SYDNEY · FRANKFURT

U.K.	Pergamon Press Ltd., Headington Hill Hall, Oxford OX3 0BW, England
U.S.A.	Pergamon Press Inc., Maxwell House, Fairview Park, Elmsford, New York 10523, U.S.A.
CANADA	Pergamon Press Canada Ltd., Suite 104, 150 Consumers Road, Willowdale, Ontario M2J 1P9, Canada
AUSTRALIA	Pergamon Press (Aust.) Pty. Ltd., P.O. Box 544, Potts Point, N.S.W. 2011, Australia
FEDERAL REPUBLIC OF GERMANY	Pergamon Press GmbH, Hammerweg 6, D-6242 Kronberg-Taunus, Federal Republic of Germany

First edition 1985

British Library Cataloguing in Publication Data

Scandium, yttrium, lanthanum and lanthanide halides in nonaqueous solvents. — (Solubility data series; v. 22)
1. Rare earth nitrates — Solubility — Tables
I. Mioduski, T. II. Salomon, M. III. Series
546′.4 QD172.R2
ISBN 0-08-030709-4

Printed in Great Britain by A. Wheaton & Co. Ltd., Exeter

CONTENTS

1. Scandium

2. Yttrium

3. Lanthanum

4. Cerium

5. Praseodymium

6. Neodymium

7. Samarium

8. Europium

9. Gadolinium

10. Terbium

11. Dysprosium

12. Holmium

13. Erbium

14. Thulium

15. Ytterbium

16. Lutetium

SOLUBILITY DATA SERIES

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY
IUPAC Secretariat: Bank Court Chambers, 2–3 Pound Way,
Cowley Centre, Oxford OX4 3YF, UK

FOREWORD

If the knowledge is
undigested or simply wrong,
more is not better

How to communicate and disseminate numerical data effectively in chemical science and technology has been a problem of serious and growing concern to IUPAC, the International Union of Pure and Applied Chemistry, for the last two decades. The steadily expanding volume of numerical information, the formulation of new interdisciplinary areas in which chemistry is a partner, and the links between these and existing traditional subdisciplines in chemistry, along with an increasing number of users, have been considered as urgent aspects of the information problem in general, and of the numerical data problem in particular.

Among the several numerical data projects initiated and operated by various IUPAC commissions, the *Solubility Data Project* is probably one of the most ambitious ones. It is concerned with preparing a comprehensive critical compilation of data on solubilities in all physical systems, of gases, liquids and solids. Both the basic and applied branches of almost all scientific disciplines require a knowledge of solubilities as a function of solvent, temperature and pressure. Solubility data are basic to the fundamental understanding of processes relevant to agronomy, biology, chemistry, geology and oceanography, medicine and pharmacology, and metallurgy and materials science. Knowledge of solubility is very frequently of great importance to such diverse practical applications as drug dosage and drug solubility in biological fluids, anesthesiology, corrosion by dissolution of metals, properties of glasses, ceramics, concretes and coatings, phase relations in the formation of minerals and alloys, the deposits of minerals and radioactive fission products from ocean waters, the composition of ground waters, and the requirements of oxygen and other gases in life support systems.

The widespread relevance of solubility data to many branches and disciplines of science, medicine, technology and engineering, and the difficulty of recovering solubility data from the literature, lead to the proliferation of published data in an ever increasing number of scientific and technical primary sources. The sheer volume of data has overcome the capacity of the classical secondary and tertiary services to respond effectively.

While the proportion of secondary services of the review article type is generally increasing due to the rapid growth of all forms of primary literature, the review articles become more limited in scope, more specialized. The disturbing phenomenon is that in some disciplines, certainly in chemistry, authors are reluctant to treat even those limited-in-scope reviews exhaustively. There is a trend to preselect the literature, sometimes under the pretext of reducing it to manageable size. The crucial problem with such preselection - as far as numerical data are concerned - is that there is no indication as to whether the material was excluded by design or by a less than thorough literature search. We are equally concerned that most current secondary sources, critical in character as they may be, give scant attention to numerical data.

On the other hand, tertiary sources - handbooks, reference books and other tabulated and graphical compilations - as they exist today are comprehensive but, as a rule, uncritical. They usually attempt to cover whole disciplines, and thus obviously are superficial in treatment. Since they command a wide market, we believe that their service to the advancement of science is at least questionable. Additionally, the change which is taking place in the generation of new and diversified numerical data, and the rate at which this is done, is not reflected in an increased third-level service. The emergence of new tertiary literature sources does not parallel the shift that has occurred in the primary literature.

With the status of current secondary and tertiary services being as briefly stated above, the innovative approach of the *Solubility Data Project* is that its compilation and critical evaluation work involve consolidation and reprocessing services when both activities are based on intellectual and scholarly reworking of information from primary sources. It comprises compact compilation, rationalization and simplification, and the fitting of isolated numerical data into a critically evaluated general framework.

The *Solubility Data Project* has developed a mechanism which involves a number of innovations in exploiting the literature fully, and which contains new elements of a more imaginative approach for transfer of reliable information from primary to secondary/tertiary sources. *The fundamental trend of the Solubility Data Project is toward integration of secondary and tertiary services with the objective of producing in-depth critical analysis and evaluation which are characteristic to secondary services, in a scope as broad as conventional tertiary services.*

Fundamental to the philosophy of the project is the recognition that the basic element of strength is the active participation of career scientists in it. Consolidating primary data, producing a truly critically-evaluated set of numerical data, and synthesizing data in a meaningful relationship are demands considered worthy of the efforts of top scientists. Career scientists, who themselves contribute to science by their involvement in active scientific research, are the backbone of the project. The scholarly work is commissioned to recognized authorities, involving a process of careful selection in the best tradition of IUPAC. This selection in turn is the key to the quality of the output. These top experts are expected to view their specific topics dispassionately, paying equal attention to their own contributions and to those of their peers. They digest literature data into a coherent story by weeding out what is wrong from what is believed to be right. To fulfill this task, the evaluator must cover *all* relevant open literature. No reference is excluded by design and every effort is made to detect every bit of relevant primary source. Poor quality or wrong data are mentioned and explicitly disqualified as such. In fact, it is only when the reliable data are presented alongside the unreliable data that proper justice can be done. The user is bound to have incomparably more confidence in a succinct evaluative commentary and a comprehensive review with a complete bibliography to both good and poor data.

It is the standard practice that the treatment of any given solute-solvent system consists of two essential parts: I. Critical Evaluation and Recommended Values, and II. Compiled Data Sheets.

The Critical Evaluation part gives the following information:

(i) a verbal text of evaluation which discusses the numerical solubility information appearing in the primary sources located in the literature. The evaluation text concerns primarily the quality of data after consideration of the purity of the materials and their characterization, the experimental method employed and the uncertainties in control of physical parameters, the reproducibility of the data, the agreement of the worker's results on accepted test systems with standard values, and finally, the fitting of data, with suitable statistical tests, to mathematical functions;
(ii) a set of recommended numerical data. Whenever possible, the set of recommended data includes weighted average and standard deviations, and a set of smoothing equations derived from the experimental data endorsed by the evaluator;
(iii) a graphical plot of recommended data.

The Compilation part consists of data sheets of the best experimental data in the primary literature. Generally speaking, such independent data sheets are given only to the best and endorsed data covering the known range of experimental parameters. Data sheets based on primary sources where the data are of a lower precision are given only when no better data are available. Experimental data with a precision poorer than considered acceptable are reproduced in the form of data sheets when they are the only known data for a particular system. Such data are considered to be still suitable for some applications, and their presence in the compilation should alert researchers to areas that need more work.

The typical data sheet carries the following information:

(i) components - definition of the system - their names, formulas and Chemical Abstracts registry numbers;
(ii) reference to the primary source where the numerical information is reported. In cases when the primary source is a less common periodical or a report document, published though of limited availability, abstract references are also given;
(iii) experimental variables;
(iv) identification of the compiler;
(v) experimental values as they appear in the primary source. Whenever available, the data may be given both in tabular and graphical form. If auxiliary information is available, the experimental data are converted also to SI units by the compiler.

Under the general heading of Auxiliary Information, the essential experimental details are summarized:

(vi) experimental method used for the generation of data;
(vii) type of apparatus and procedure employed;
(viii) source and purity of materials;
(ix) estimated error;
(x) references relevant to the generation of experimental data as cited in the primary source.

This new approach to numerical data presentation, formulated at the initiation of the project and perfected as experience has accumulated, has been strongly influenced by the diversity of background of those whom we are supposed to serve. We thus deemed it right to preface the evaluation/compilation sheets in each volume with a detailed discussion of the principles of the accurate determination of relevant solubility data and related thermodynamic information.

Finally, the role of education is more than corollary to the efforts we are seeking. The scientific standards advocated here are necessary to strengthen science and technology, and should be regarded as a major effort in the training and formation of the next generation of scientists and engineers. Specifically, we believe that there is going to be an impact of our project on scientific-communication practices. The quality of consolidation adopted by this program offers down-to-earth guidelines, concrete examples which are bound to make primary publication services more responsive than ever before to the needs of users. The self-regulatory message to scientists of the early 1970s to refrain from unnecessary publication has not achieved much. A good fraction of the literature is still cluttered with poor-quality articles. The Weinberg report (in 'Reader in Science Information', ed. J. Sherrod and A. Hodina, Microcard Editions Books, Indian Head, Inc., 1973, p. 292) states that 'admonition to authors to restrain themselves from premature, unnecessary publication can have little effect unless the climate of the entire technical and scholarly community encourages restraint...' We think that projects of this kind translate the climate into operational terms by exerting pressure on authors to avoid submitting low-grade material. The type of our output, we hope, will encourage attention to quality as authors will increasingly realize that their work will not be suited for permanent retrievability unless it meets the standards adopted in this project. It should help to dispel confusion in the minds of many authors of what represents a permanently useful bit of information of an archival value, and what does not.

If we succeed in that aim, even partially, we have then done our share in protecting the scientific community from unwanted and irrelevant, wrong numerical information.

A. S. Kertes

PREFACE

SCOPE OF THE VOLUME

This volume deals with the solubilities of the halides of scandium, yttrium, lanthanum, and lanthanides in nonaqueous solvents. The volume includes the solubilities of these "rare earth" halides in both organic and inorganic solvents, and in mixed solvents containing water where water is the minor constituent of the mixed solvent. The solubilities of rare earth halides in mixed water-organic solvents covering the entire composition range of 100 % water to 100 % organic component will be published in a forthcoming volume.

The literature on the solubilities of Sc, Y, La and the lanthanide halides in nonaqueous solvents has been covered through 1984, and only a few systems have not been compiled. The paper by Safonov et al. (1) dealing with the solubility of $NdCl_3$ in $SeOCl_2$ and $POCl_3$ has been rejected since it contains only qualitative limits of solubilities. Similarly we have rejected the data of Nikolaev et al. (2) who reported the solubilities of rare earth fluorides in water and liquid HF: the results were presented graphically. Data from (3) have also been omitted since the original data were reported in other publications which have been compiled in this volume. It is also noted that solubility data for *didymium chloride* in a number of ethers (4) have been rejected since didymium is a mixture of rare earths.

Most of the solubility data reported in the literature are either in mass % units or in units of mol kg^{-1}. A number of *Masters Theses* originating in the laboratory of Prof. Hopkins (University of Illinois) around 1930-1934 report solubilities in mol dm^{-3} units. Since density data are generally unavailable, direct comparisons of these early data with more recent results based on mass units are not possible. Hence we have not prepared critical evaluations for those systems for which two publications exist, and for which one publication reports solubilities in volume units and the other in mass units.

NATURE OF THE SOLID PHASE

For those researches which report analyses of equilibrated solid phases, we find considerable disagreement in the reported compositions. For example at 298.2 K Grigorovich (5) reports a solid phase of $NdCl_3 \cdot 3CH_3OH$ as opposed to $NdCl_3 \cdot 4CH_3OH$ reported by Merbach et al. (6). Since Grigorovich's solubility data are always significantly greater than those of all other workers, the evaluators assumed that Grigorovich's results might be based upon metastable equilibria. However it is always possible that errors exist in analyses of the solid phases which have escaped detection by the evaluators because of insufficient experimental descriptions in the source publications. Grigorovich's high solubilities could be due to a constant error in calculation. For example he reports a solubility of 32.04 mass % for $PrCl_3$ in 1-propanol at 298.2 K, and assuming this result to be based on the *anhydrous* $PrCl_3$ the evaluators calculated a solubility of 1.91 mol kg^{-1}. However if the solubility of 32.04 mass % is based on the solvate $PrCl_3 \cdot 2C_3H_7OH$, then the solubility calculated by the editors is 1.28 mol kg^{-1} which is close to the result of 1.24 mol kg^{-1} reported by Kirmse (7): note that Kirmse reports the solid phase to be $PrCl_3 \cdot C_3H_7OH$. Although all of Grigorovich's mass % results have been *assumed* by the evaluators to be based on the anhydrous salts, the reader should be aware that there is a high probability that Grigorovich's data are based upon mass % of the solvate, and that the reported compositions of the solvates may also be in error.

GENERAL COMMENTS

A major part of this volume consists of compilations from publications of Prof. E.M. Kirmse and her colleagues (in particular H. Dressler). Due to the fact that experimental details in Prof. Kirmse's publications are often incomplete, and due to the fact that her works form a principal part of this volume, it seems appropriate to combine the experimental details from all of her publications and present them in one place. The following therefore is a combined review of Prof. Kirmse's experimental techniques.

> "Anhydrous salts were prepared from the oxide or directly from the halide by the method of Taylor and Carter. Saturated solutions were prepared isothermally by mechanical agitation either by thermostating at 298 ± 0.2 K or at room temperature (291-298 K). Aliquots of saturated solution were treated with KOH solution to precipitate the rare earth hydroxide, and the rare earth content determined by complexometric titration. In several instances (e.g. with fluorides) the anion concentration was also determined. Anhydrous reagents were handled in a dry box containing P_2O_5, and the reported solubilities are mean values of 2-4 determinations. Solid phases were analysed from "wet" residues which were placed in a vacuum desiccator over P_2O_5 until constant mass was obtained. Both the rare earth content and the anion content of the solid phases were determined."

Noticeably absent in all of Prof. Kirmse's publications are the sources and purities of starting materials (oxides, halides, solvents). It is not clear why the rare earth fluorides prepared by Kirmse and Dressler by the wet method are not stoichiometric. It would appear that in spite of heating at around 580 K for 120 days, they obtained partially hydrated salts.

We conclude that the determination of the solubilities of anhydrous rare earth halides is subject to many sources of error. In spite of the large amount of data reported, significant disagreement exists, particularly with respect to composition of the solid phases. More rigorous thermodynamic treatment of these solublity data requires an exact knowledge of the compositions of the solid phases, and a precise determination of both stable and metastable equilibria. We hope that the present volume will serve as a useful guide in this respect.

The editors gratefully acknowledge the advice and encouragement of Prof. S. Siekierski. We also acknowledge the important comments by Prof. Kirmse, and her cooperation in sending us many reprints of her publications. Acknowledgements are also due Ms. Brenda Shanholtz, Reference Librarian, ERADCOM Technical Library, and Ms. Susanne Redalje, Assistant Chemistry Librarian, University of Illinois. Ms. Redalje kindly provided us with copies of numerous *Theses* on the solubilities of rare earth halides and nitrates.

REFERENCES

1. Safonov, V.V.; Fedorov, E.A.; Lebedev, V.G. *Zh. Neorg. Khim.* 1978, *23*, 1981.
2. Nikolaev, N.S.; Abdurakhmanov, Sh.A.; Dzhuraev, Kh.Sh. *Zh. Neorg. Khim.* 1974, *19*, 1133.
3. Zweitasch, K.J.; Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Kothen* 1971, *1*, 19.
4. Kirmse, E.M.; Dressler, H. *Z. Chem.* 1975, *15*, 239.
5. Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986.
6. Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44.
7. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200.

T. Mioduski
Institute for Nuclear Research
Warsaw, Poland

M. Salomon
PO Box 254
Fair Haven, NJ, USA

November 1984

INTRODUCTION TO THE SOLUBILITY OF SOLIDS IN LIQUIDS

Nature of the Project

The Solubility Data Project (SDP) has as its aim a comprehensive search of the literature for solubilities of gases, liquids, and solids in liquids or solids. Data of suitable precision are compiled on data sheets in a uniform format. The data for each system are evaluated, and where data from different sources agree sufficiently, recommended values are proposed. The evaluation sheets, recommended values, and compiled data sheets are published on consecutive pages.

This series of volumes includes solubilities of solids of all types in liquids of all types.

Definitions

A *mixture* (1,2) describes a gaseous, liquid, or solid phase containing more than one substance, when the substances are all treated in the same way.

A *solution* (1,2) describes a liquid or solid phase containing more than one substance, when for convenience one of the substances, which is called the *solvent* and may itself be a mixture, is treated differently than the other substances, which are called *solutes*. If the sum of the mole fractions of the solutes is small compared to unity, the solution is called a *dilute solution.*

The *solubility* of a substance B is the relative proportion of B (or a substance related chemically to B) in a mixture which is saturated with respect to solid B at a specified temperature and pressure. *Saturated* implies the existence of equilibrium with respect to the processes of dissolution and precipitation; the equilibrium may be stable or metastable. The solubility of a metastable substance is usually greater than that of the corresponding stable substance. (Strictly speaking, it is the activity of the metastable substance that is greater.) Care must be taken to distinguish true metastability from supersaturation, where equilibrium does not exist.

Either point of view, mixture or solution, may be taken in describing solubility. The two points of view find their expression in the quantities used as measures of solubility and in the reference states used for definition of activities and activity coefficients.

The qualifying phrase "substance related chemically to B" requires comment. The composition of the saturated mixture (or solution) can be described in terms of any suitable set of thermodynamic components. Thus, the solubility of a salt hydrate in water is usually given as the relative proportion of anhydrous salt in solution, rather than the relative proportions of hydrated salt and water.

Quantities Used as Measures of Solubility

1. *Mole fraction* of substance B, x_B:

$$x_B = n_B / \sum_{i=1}^{c} n_i \qquad (1)$$

where n_i is the amount of substance of substance i, and c is the number of distinct substances present (often the number of thermodynamic components in the system). *Mole per cent* of B is 100 x_B.

2. *Mass fraction* of substance B, w_B:

$$w_B = m'_B / \sum_{i=1}^{c} m'_i \qquad (2)$$

where m'_i is the mass of substance i. *Mass per cent* of B is 100 w_B. The equivalent terms weight fraction and weight per cent are not used.

3. *Solute mole (mass) fraction* of solute B (3,4):

$$x_{S,B} = n_B / \sum_{i=1}^{c'} n_i = x_B / \sum_{i=1}^{c'} x_i \qquad (3)$$

where the summation is over the solutes only. For the solvent A, $x_{S,A} = x_A$. These quantities are called *Jänecke mole (mass) fractions* in many papers.

4. *Molality* of solute B (1,2) in a solvent A:

$$m_B = n_B/n_A M_A \qquad \text{SI base units: mol kg}^{-1} \tag{4}$$

where M_A is the molar mass of the solvent.

5. *Concentration* of solute B (1,2) in a solution of volume V:

$$c_B = [B] = n_B/V \qquad \text{SI base units: mol m}^{-3} \tag{5}$$

The terms molarity and molar are not used.

Mole and mass fractions are appropriate to either the mixture or the solution points of view. The other quantities are appropriate to the solution point of view only. In addition of these quantities, the following are useful in conversions between concentrations and other quantities.

6. *Density*: $\rho = m/V$ SI base units: kg m^{-3} (6)

7. *Relative density*: d; the ratio of the density of a mixture to the density of a reference substance under conditions which must be specified for both (1). The symbol $d_{t'}^{t}$ will be used for the density of a mixture at t°C, 1 atm divided by the density of water at t'°C, 1 atm.

Other quantities will be defined in the prefaces to individual volumes or on specific data sheets.

Thermodynamics of Solubility

The principal aims of the Solubility Data Project are the tabulation and evaluation of: (a) solubilities as defined above; (b) the nature of the saturating solid phase. Thermodynamic analysis of solubility phenomena has two aims: (a) to provide a rational basis for the construction of functions to represent solubility data; (b) to enable thermodynamic quantities to be extracted from solubility data. Both these aims are difficult to achieve in many cases because of a lack of experimental or theoretical information concerning activity coefficients. Where thermodynamic quantities can be found, they are not evaluated critically, since this task would involve critical evaluation of a large body of data that is not directly relevant to solubility. The following discussion is an outline of the principal thermodynamic relations encountered in discussions of solubility. For more extensive discussions and references, see books on thermodynamics, e.g., (5-10).

Activity Coefficients (1)

(a) *Mixtures*. The activity coefficient f_B of a substance B is given by

$$RT\,\ell n(f_B x_B) = \mu_B - \mu_B^* \tag{7}$$

where μ_B is the chemical potential, and μ_B^* is the chemical potential of pure B at the same temperature and pressure. For any substance B in the mixture,

$$\lim_{x_B \to 1} f_B = 1 \tag{8}$$

(b) *Solutions*.

(i) *Solute substance*, B. The molal activity coefficient γ_B is given by

$$RT\,\ell n(\gamma_B m_B) = \mu_B - (\mu_B - RT\,\ell n\, m_B)^{\infty} \tag{9}$$

where the superscript ∞ indicates an infinitely dilute solution. For any solute B,

$$\gamma_B^{\infty} = 1 \tag{10}$$

Activity coefficients y_B connected with concentration c_B, and $f_{x,B}$ (called the *rational activity coefficient*) connected with mole fraction x_B are defined in analogous ways. The relations among them are (1,9):

$$\gamma_B = x_A f_{x,B} = V_A^*(1 - \sum_s c_s) y_B \tag{11}$$

or

$$f_{x,B} = (1 + M_A \sum_s m_s)\gamma_B = V_A{}^* y_B / V_m \tag{12}$$

or

$$y_B = (V_A + M_A \sum_s m_s V_s)\gamma_B / V_A{}^* = V_m f_{x,B} / V_A{}^* \tag{13}$$

where the summations are over all solutes, $V_A{}^*$ is the molar volume of the pure solvent, V_i is the partial molar volume of substance i, and V_m is the molar volume of the solution.

For an electrolyte solute $B \equiv C_{\nu+}A_{\nu-}$, the molal activity is replaced by (9)

$$\gamma_B m_B = \gamma_\pm{}^\nu m_B{}^\nu Q^\nu \tag{14}$$

where $\nu = \nu_+ + \nu_-$, $Q = (\nu_+{}^{\nu+}\nu_-{}^{\nu-})^{1/\nu}$, and $\gamma_\pm$ is the mean ionic molal activity coefficient. A similar relation holds for the concentration activity $y_B c_B$. For the mol fractional activity,

$$f_{x,B}\, x_B = \nu_+{}^{\nu_+} \nu_-{}^{\nu_-} f_\pm{}^\nu x_\pm{}^\nu \tag{15}$$

The quantities x_+ and x_- are the ionic mole fractions (9), which for a single solute are

$$x_+ = \nu_+ x_B / [1 + (\nu - 1) x_B]; \qquad x_- = \nu_- x_B / [1 + (\nu - 1) x_B] \tag{16}$$

(ii) *Solvent, A:*

The *osmotic coefficient*, ϕ , of a solvent substance A is defined as (1):

$$\phi = (\mu_A{}^* - \mu_A)/RT\, M_A \sum_s m_s \tag{17}$$

where $\mu_A{}^*$ is the chemical potential of the pure solvent.

The *rational osmotic coefficient*, ϕ_x, is defined as (1):

$$\phi_x = (\mu_A - \mu_A{}^*)/RT \ln x_A = \phi M_A \sum_s m_s / \ln(1 + M_A \sum_s m_s) \tag{18}$$

The activity, a_A, or the activity coefficient f_A is often used for the solvent rather than the osmotic coefficient. The activity coefficient is defined relative to pure A, just as for a mixture.

The Liquid Phase

A general thermodynamic differential equation which gives solubility as a function of temperature, pressure and composition can be derived. The approach is that of Kirkwood and Oppenheim (7). Consider a solid mixture containing c' thermodynamic components i. The Gibbs-Duhem equation for this mixture is:

$$\sum_{i=1}^{c'} x_i{}'(S_i{}'dT - V_i{}'dp + d\mu_i) = 0 \tag{19}$$

A liquid mixture in equilibrium with this solid phase contains c thermodynamic components i, where, usually, c ≥ c'. The Gibbs-Duhem equation for the liquid mixture is:

$$\sum_{i=1}^{c'} x_i(S_i dT - V_i dp + d\mu_i) + \sum_{i=c'+1}^{c} x_i(S_i dT - V_i dp + d\mu_i) = 0 \tag{20}$$

Eliminate $d\mu_1$ by multiplying (19) by x_1 and (20) $x_1{}'$. After some algebra, and use of:

$$d\mu_i = \sum_{j=2}^{c} G_{ij} dx_j - S_i dT + V_i dp \tag{21}$$

where (7)

$$G_{ij} = (\partial\mu_i/\partial x_j)_{T,P,x_i \neq x_j} \tag{22}$$

it is found that

$$\sum_{i=2}^{c'} \sum_{j=2}^{c} (x_i{}' - x_i x_1'/x_1) G_{ij} dx_j - (x_1{}'/x_1) \sum_{i=c'+1}^{c} \sum_{j=2}^{c} x_i G_{ij} dx_j$$

$$= \sum_{i=1}^{c'} x_i{}'(H_i - H_i{}')dT/T - \sum_{i=1}^{c'} x_i{}'(V_i - V_i{}')dp \tag{23}$$

where

$$H_i - H_i' = T(S_i - S_i') \tag{24}$$

is the enthalpy of transfer of component i from the solid to the liquid phase, at a given temperature, pressure and composition, and H_i, S_i, V_i are the partial molar enthalpy, entropy, and volume of component i. Several special cases (all with pressure held constant) will be considered. Other cases will appear in individual evaluations.

(a) Solubility as a function of temperature.
Consider a binary solid compound A_nB in a single solvent A. There is no fundamental thermodynamic distinction between a binary compound of A and B which dissociates completely or partially on melting and a solid mixture of A and B; the binary compound can be regarded as a solid mixture of constant composition. Thus, with c = 2, c' = 1, $x_A' = n/(n+1)$, $x_B' = 1/(n+1)$, eqn (23) becomes

$$(1/x_B - n/x_A)\{1 + (\frac{\partial \ell n f_B}{\partial \ell n x_B})_{T,P}\}dx_B = (nH_A + H_B - H_{AB}^{*})dT/RT^2 \tag{25}$$

where the mole fractional activity coefficient has been introduced. If the mixture is a non-electrolyte, and the activity coefficients are given by the expression for a simple mixture (6):

$$RT \ \ell n \ f_B = wx_A^{\,2} \tag{26}$$

then it can be shown that, if w is independent of temperature, eqn (25) can be integrated (cf. (5), Chap. XXIII, sect. 5). The enthalpy term becomes

$$\begin{aligned} nH_A + H_B - H_{AB}^{*} &= \Delta H_{AB} + n(H_A - H_A{*}) + (H_B - H_B{*}) \\ &= \Delta H_{AB} + w(nx_B^{\,2} + x_A^{\,2}) \end{aligned} \tag{27}$$

where ΔH_{AB} is the enthalpy of melting and dissociation of one mole of pure solid A_nB, and $H_A{*}$, $H_B{*}$ are the molar enthalpies of pure liquid A and B. The differential equation becomes

$$R \ d \ \ell n\{x_B(1-x_B)^n\} = -\Delta H_{AB} \ d(\frac{1}{T}) - w \ d(\frac{x_A^{\,2} + nx_B^{\,2}}{T}) \tag{28}$$

Integration from x_B,T to $x_B = 1/(1+n)$, T = T*, the melting point of the pure binary compound, gives:

$$\begin{aligned} \ell n\{x_B(1-x_B)^n\} \simeq{} & \ell n\{\frac{n^n}{(1+n)^{n+1}}\} - \{\frac{\Delta H_{AB}^{*} - T^{*}\Delta C_p^{*}}{R}\}\ (\frac{1}{T} - \frac{1}{T^{*}}) \\ & + \frac{\Delta C_p{}^{*}}{R}\ell n(\frac{T}{T^{*}}) - \frac{w}{R}\{\frac{x_A \ + nx_B}{T} - \frac{n}{(n+1)T^{*}}\} \end{aligned} \tag{29}$$

where $\Delta C_p{*}$ is the change in molar heat capacity accompanying fusion plus decomposition of the compound at temperature T*, (assumed here to be independent of temperature and composition), and ΔH_{AB}^{*} is the corresponding change in enthalpy at T = T*. Equation (29) has the general form

$$\ell n\{x_B(1-x_B)^n\} = A_1 + A_2/T + A_3 \ell nT + A_4(x_A^{\,2} + nx_B^{\,2})/T \tag{30}$$

If the solid contains only component B, n = 0 in eqn (29) and (30).

If the infinite dilution standard state is used in eqn (25), eqn (26) becomes

$$RT \ \ell n \ f_{x,B} = w(x_A^{\,2} - 1) \tag{31}$$

and (27) becomes

$$nH_A + H_B - H_{AB} = (nH_A{*} + H_B^{\infty} - H_{AB}^{*}) + n(H_A - H_A{*}) + (H_B - H_B^{\infty}) = \Delta H_{AB}^{\infty} + w(nx_B^{\,2} + x_A^{\,2} - 1) \tag{32}$$

where the first term, ΔH_{AB}^{∞}, is the enthalpy of melting and dissociation of solid compound A_nB to the infinitely dilute state of solute B in solvent A; H_B^{∞} is the partial molar enthalpy of the solute at infinite dilution. Clearly, the integral of eqn (25) will have the same form as eqn (29), with $\Delta H_{AB}^{\infty}(T^{*})$, $\Delta C_p^{\infty}(T^{*})$ replacing ΔH_{AB}^{*} and $\Delta C_p{*}$ and $x_A^{\,2} - 1$ replacing $x_A^{\,2}$ in the last term.

If the liquid phase is an aqueous electrolyte solution, and the solid is a salt hydrate, the above treatment needs slight modification. Using rational mean activity coefficients, eqn (25) becomes

$$R\nu(1/x_B-n/x_A)\{1+(\partial \ell n f_\pm/\partial \ell n x_\pm)_{T,P}\}dx_B/\{1+(\nu-1)x_B\}$$

$$= \{\Delta H^\infty_{AB} + n(H_A-H_A{}^*) + (H_B-H^\infty_B)\}d(1/T) \tag{33}$$

If the terms involving activity coefficients and partial molar enthalpies are negligible, then integration gives (cf. (11)):

$$\ell n\{\frac{x_B^\nu(1-x_B)^n}{1+(\nu-1)x_B{}^{n+\nu}}\} = \ell n\{\frac{n^n}{(n+\nu)^{n+\nu}}\} - \{\frac{\Delta H^\infty_{AB}(T^*)-T^*\Delta C_p{}^*}{R}\}\ (\frac{1}{T} - \frac{1}{T^*}) + \frac{\Delta C_p^*}{R}\ell n(T/T^*) \tag{34}$$

A similar equation (with $\nu=2$ and without the heat capacity terms) has been used to fit solubility data for some $MOH=H_2O$ systems, where M is an alkali metal; the enthalpy values obtained agreed well with known values (11). In many cases, data on activity coefficients (9) and partial molal enthalpies (8,10) in concentrated solution indicate that the terms involving these quantities are not negligible, although they may remain roughly constant along the solubility temperature curve.

The above analysis shows clearly that a rational thermodynamic basis exists for functional representation of solubility-temperature curves in two-component systems, but may be difficult to apply because of lack of experimental or theoretical knowledge of activity coefficients and partial molar enthalpies. Other phenomena which are related ultimately to the stoichiometric activity coefficients and which complicate interpretation include ion pairing, formation of complex ions, and hydrolysis. Similar considerations hold for the variation of solubility with pressure, except that the effects are relatively smaller at the pressures used in many investigations of solubility (5).

(b) Solubility as a function of composition.
At constant temperature and pressure, the chemical potential of a saturating solid phase is constant:

$$\mu^*_{A_nB} = \mu_{A_nB}(\text{sln}) = n\mu_A + \mu_B \tag{35}$$

$$= (n\mu^*_A + \nu_+\mu_+{}^\infty + \nu_-\mu_-{}^\infty) + nRT\ \ell n f_A x_A$$

$$+ \nu RT\ \ell n \gamma_\pm m_\pm Q_\pm \tag{36}$$

for a salt hydrate A_nB which dissociates to water, (A), and a salt, B, one mole of which ionizes to give ν_+ cations and ν_- anions in a solution in which other substances (ionized or not) may be present. If the saturated solution is sufficiently dilute, $f_A = x_A = 1$, and the quantity K^0_{s0} in

$$\Delta G^\infty \equiv (\nu_+\mu_+{}^\infty + \nu_-\mu_-{}^\infty + n\mu^*_A - \mu_{AB}{}^*)$$

$$= -RT\ \ell n\ K^0_{s0}$$

$$= -RT\ \ell n\ Q^\nu \gamma_\pm{}^\nu m_+{}^{\nu_+} m_-{}^{\nu_-} \tag{37}$$

is called the *solubility product* of the salt. (It should be noted that it is not customary to extend this definition to hydrated salts, but there is no reason why they should be excluded.) Values of the solubility product are often given on mole fraction or concentration scales. In dilute solutions, the theoretical behaviour of the activity coefficients as a function of ionic strength is often sufficiently well known that reliable extrapolations to infinite dilution can be made, and values of K^0_{s0} can be determined. In more concentrated solutions, the same problems with activity coefficients that were outlined in the section on variation of solubility with temperature still occur. If these complications do not arise, the solubility of a hydrate salt $C_{\nu_+}A_{\nu_-}\cdot nH_2O$ in the presence of other solutes is given by eqn (36) as

$$\nu\ \ell n\{m_B/m_B(0)\} = -\nu \ell n\{\gamma_\pm/\gamma_\pm(0)\} - n\ \ell n(a_{H_2O}/a_{H_2O}(0)) \tag{38}$$

where a_{H_2O} is the activity of water in the saturated solution, m_B is the molality of the salt in the saturated solution, and (0) indicates absence of other solutes. Similar considerations hold for non-electrolytes.

The Solid Phase

The definition of solubility permits the occurrence of a single solid phase which may be a pure anhydrous compound, a salt hydrate, a non-stoichiometric compound, or a solid mixture (or solid solution, or "mixed crystals"), and may be stable or metastable. As well, any number of solid phases consistent with the requirements of the phase rule may be present. Metastable solid phases are of widespread occurrence, and may appear as polymorphic (or allotropic) forms or crystal solvates whose rate of transition to more stable forms is very slow. Surface heterogeneity may also give rise to metastability, either when one solid precipitates on the surface of another, or if the size of the solid particles is sufficiently small that surface effects become important. In either case, the solid is not in stable equilibrium with the solution. The stability of a solid may also be affected by the atmosphere in which the system is equilibrated.

Many of these phenomena require very careful, and often prolonged, equilibration for their investigation and elimination. A very general analytical method, the "wet residues" method of Schreinemakers (12) (see a text on physical chemistry) is usually used to investigate the composition of solid phases in equilibrium with salt solutions. In principle, the same method can be used with systems of other types. Many other techniques for examination of solids, in particular X-ray, optical, and thermal analysis methods, are used in conjunction with chemical analyses (including the wet residues method).

COMPILATIONS AND EVALUATIONS

The formats for the compilations and critical evaluations have been standardized for all volumes. A brief description of the data sheets has been given in the FOREWORD; additional explanation is given below.

Guide to the Compilations

The format used for the compilations is, for the most part, self-explanatory. The details presented below are those which are not found in the FOREWORD or which are not self-evident.

Components. Each component is listed according to IUPAC name, formula, and Chemical Abstracts (CA) Registry Number. The formula is given either in terms of the IUPAC or Hill (13) system and the choice of formula is governed by what is usual for most current users: i.e. IUPAC for inorganic compounds, and Hill system for organic compounds. Components are ordered according to:

(a) saturating components;
(b) non-saturating components in alphanumerical order;
(c) solvents in alphanumerical order.

The saturating components are arranged in order according to a 18-column, 2-row periodic table:

Columns 1,2: H, groups IA, IIA;
3,12: transition elements (groups IIIB to VIIB, group VIII, groups IB, IIB);
13-18: groups IIIA-VIIA, noble gases.
Row 1: Ce to Lu;
Row 2: Th to the end of the known elements, in order of atomic number.

Salt hydrates are generally not considered to be saturating components since most solubilities are expressed in terms of the anhydrous salt. The existence of hydrates or solvates is carefully noted in the texts, and CA Registry Numbers are given where available, usually in the critical evaluation. Mineralogical names are also quoted, along with their CA Registry Numbers, again usually in the critical evaluation.

Original Measurements. References are abbreviated in the forms given by *Chemical Abstracts Service Source Index (CASSI)*. Names originally in other than Roman alphabets are given as transliterated by *Chemical Abstracts*.

Experimental Values. Data are reported in the units used in the original publication, with the exception that modern *names* for units and quantities are used; e.g., mass per cent for weight per cent; mol dm^{-3} for molar; etc. Both mass and molar values are given. Usually, only one type of value (e.g., mass per cent) is found in the original paper, and the compiler has added the other type of value (e.g., mole per cent) from computer calculations based on 1976 atomic weights (14). Errors in calculations and fitting equations in original papers have been noted and corrected, by computer calculations where necessary.

Method. Source and Purity of Materials. Abbreviations used in *Chemical Abstracts* are often used here to save space.

Estimated Error. If these data were omitted by the original authors, and if relevant information is available, the compilers have attempted to estimate errors from the internal consistency of data and type of apparatus used. Methods used by the compilers for estimating and reporting errors are based on the papers by Ku and Eisenhart (15).

Comments and/or Additional Data. Many compilations include this section which provides short comments relevant to the general nature of the work or additional experimental and thermodynamic data which are judged by the compiler to be of value to the reader.

References. See the above description for Original Measurements.

Guide to the Evaluations

The evaluator's task is to check whether the compiled data are correct, to assess the reliability and quality of the data, to estimate errors where necessary, and to recommend "best" values. The evaluation takes the form of a summary in which all the data supplied by the compiler have been critically reviewed. A brief description of the evaluation sheets is given below.

Components. See the description for the Compilations.

Evaluator. Name and date up to which the literature was checked.

Critical Evaluation

(a) Critical text. The evaluator produces text evaluating *all* the published data for each given system. Thus, in this section the evaluator review the merits or shortcomings of the various data. Only published data are considered; even published data can be considered only if the experimental data permit an assessment of reliability.

(b) Fitting equations. If the use of a smoothing equation is justifiable, the evaluator may provide an equation representing the solubility as a function of the variables reported on all the compilation sheets.

(c) Graphical summary. In addition to (b) above, graphical summaries are often given.

(d) Recommended values. Data are *recommended* if the results of at least two independent groups are available and they are in good agreement, and if the evaluator has no doubt as to the adequacy and reliability of the applied experimental and computational procedures. Data are reported as *tentative* if only one set of measurements is available, or if the evaluator considers some aspect of the computational or experimental method as mildly undesirable but estimates that it should cause only minor errors. Data are considered as *doubtful* if the evaluator considers some aspect of the computational or experimental method as undesirable but still considers the data to have some value in those instances where the order of magnitude of the solubility is needed. Data determined by an inadequate method or under ill-defined conditions are *rejected*. However references to these data are included in the evaluation together with a comment by the evaluator as to the reason for their rejection.

(e) References. All pertinent references are given here. References to those data which, by virtue of their poor precision, have been rejected and not compiled are also listed in this section.

(f) Units. While the original data may be reported in the units used by the investigators, the final recommended values are reported in S.I. units (1,16) when the data can be accurately converted.

References

1. Whiffen, D. H., ed., *Manual of Symbols and Terminology for Physicochemical Quantities and Units. Pure Applied Chem.* 1979, *51*, No. 1.
2. McGlashan, M.L. *Physicochemical Quantities and Units.* 2nd ed. Royal Institute of Chemistry. London. 1971.
3. Jänecke, E. *Z. Anorg. Chem.* 1906, *51*, 132.
4. Friedman, H.L. *J. Chem. Phys.* 1960, *32*, 1351.
5. Prigogine, I.; Defay, R. *Chemical Thermodynamics.* D.H. Everett, transl. Longmans, Green. London, New York, Toronto. 1954.
6. Guggenheim, E.A. *Thermodynamics.* North-Holland. Amsterdam. 1959. 4th ed.
7. Kirkwood, J.G.; Oppenheim, I. *Chemical Thermodynamics.* McGraw-Hill, New York, Toronto, London. 1961.
8. Lewis, G.N.; Randall, M. (rev. Pitzer, K.S.; Brewer, L.). *Thermodynamics.* McGraw Hill. New York, Toronto, London. 1961. 2nd ed.
9. Robinson, R.A.; Stokes, R.H. *Electrolyte Solutions.* Butterworths. London. 1959, 2nd ed.
10. Harned, H.S.; Owen, B.B. *The Physical Chemistry of Electrolytic Solutions.* Reinhold. New York. 1958. 3rd ed.
11. Cohen-Adad, R.; Saugier, M.T.; Said, J. *Rev. Chim. Miner.* 1973, *10*, 631.
12. Schreinemakers, F.A.H. *Z. Phys. Chem., stoechiom. Verwandschaftsl.* 1893, *11*, 75.
13. Hill, E.A. *J. Am. Chem. Soc.* 1900, *22*, 478.
14. IUPAC Commission on Atomic Weights. *Pure Appl. Chem.*, 1976, *47*, 75.

15. Ku, H.H., p. 73; Eisenhart, C., p. 69; in Ku, H.H., ed. *Precision Measurement and Calibration*. NBS Special Publication 300. Vol. 1. Washington. 1969.
16. *The International System of Units*. Engl. transl. approved by the BIPM of *Le Système International d'Unités*. H.M.S.O. London. 1970.

R. Cohen-Adad, Villeurbanne, France
J.W. Lorimer, London, Canada
M. Salomon, Fair Haven, New Jersey, U.S.A.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) Methanol; CH_4O; [67-56-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Z. Chem. 1961, *1*, 332-4.

VARIABLES:

One temperature: T/K = 298.2

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in methanol at 25°C was reported to be

45.5 mass %

The corresponding molality calculated by the compiler is

5.52 mol kg^{-1}

The solid phase was reported to be $ScCl_3.3CH_3OH$ which could be further dehydrated to $ScCl_3.2CH_3OH$ (see discussion below).

COMMENTS AND/OR ADDITIONAL DATA:

Since in the saturated solution there are almost 6 moles of solvent per mole of salt, the author suggests that $ScCl_3$ has a coordination number of 6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 10-15 cm^3 alcohol and $ScCl_3$ placed in glass stoppered bottles and mechanically rotated at 200 rpm in a thermostat for 14-15 days. After allowing the equilibrated slns to settle for 3 days, aliquots were removed for analyses. Sc determined gravimetrically by evaporation of solvent followed by ignition to Sc_2O_3. Identical results obtained by centrifuging the equilibrated slns prior to analyses.

Solid phase composition determined by analysis of wet residues. Samples dried in vacuum (11 ± 1 mm Hg) over P_2O_5 at 18 ± 1°C and weighed every 24 hours until the mass was constant. Sc and Cl analysed gravimetrically as Sc_2O_3 and AgCl. It is not clear if elemental C and H analyses were also carried out. After 28 days of drying, the solid phase composition was found to be $ScCl_3.3CH_3OH$. Additional drying in a desiccator or in a dry box yielded a solid phase of composition $ScCl_3.2CH_3OH$.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900-1000°C (1). Source and purity of Sc_2O_3 not specified.

Methanol was purified by fractional distillation from $CaSO_4.0.5H_2O$.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision $\pm$ 0.2 K.

REFERENCES:

1. Petru, F.; Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Comm.* 1957, *22*, 1534.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Z. Chem. 1961, *1*, 332-4.

VARIABLES:

One temperature: T/K = 298.2

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in ethanol at 25°C was reported as follows:

	aliquot 1 soly/mass %	aliquot 2 soly/mass %
first analyses	37.5	37.3
second analyses	37.0	37.4

The mean solubility is 37.3 mass %, and the corresponding (mean) molality calculated by the compiler is 3.93 mol kg^{-1}.

The solid phase was reported to be $ScCl_3.3CH_3CH_2OH$ which could be further dehydrated to $ScCl_3.2CH_3CH_2OH$.

COMMENTS AND/OR ADDITIONAL DATA:

Since in the saturated solution there are almost 6 moles of solvent per mole of salt, the author suggests that $ScCl_3$ has a coordination number of 6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 10-15 cm^3 alcohol and $ScCl_3$ placed in glass stoppered bottles and mechanically rotated at 200 rpm in a thermostat for 14-15 days. After allowing the equilibrated slns to settle for 3 days, aliquots were removed for analyses. Sc determined gravimetrically by evaporation of solvent followed by ignition to Sc_2O_3. Identical results obtained by centrifuging the equilibrated slns prior to analyses.

Solid phase composition determined by analysis of wet residues. Samples dried in vacuum (11 ± 1 mm Hg) over P_2O_5 at 18 ± 1°C and weighed every 24 hours until the mass was constant. Sc and Cl analysed gravimetrically as Sc_2O_3 and AgCl. It is not clear if elemental C and H analyses were also carried out After 57 days of drying, the solid phase composition was found to be $ScCl_33CH_3CH_2OH$. Additional drying in a desiccator or in a dry box yielded a solid phase of composition $ScCl_3.2CH_3CH_2OH$.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900-1000°C (1). Source and purity of Sc_2O_3 not specified.

Ethanol was dried with metallic sodium and distilled.

ESTIMATED ERROR:

Soly: std deviation about 0.2 mass % (compiler).

Temp: precision ± 0.2 K.

REFERENCES:

1. Petru, F.; Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Commn.* 1957, *22*, 1534.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) 1-Propanol; C_3H_8O; [71-23-8]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Z. Chem. 1961, *1*, 332-4.

VARIABLES:

One temperature: T/K = 298.2

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in 1-propanol at 25°C was reported as follows

	Run 1	Run 2	
	aliquot 1	aliquot 1	aliquot 2
	soly/mass %	soly/mass %	soly/mass %
first analyses	26.1	25.7	26.3
second analyses	26.2	26.1	---
third analyses	26.1	---	---

The mean solubility is 26.1 mass %, and the corresponding (mean) molality calculated by the compiler is 2.33 mol kg^{-1}.

The solid phase was reported to be $ScCl_3.4C_3H_7OH$ which could be further dehydrated to $ScCl_3.2C_3H_7OH$.

COMMENTS AND/OR ADDITIONAL DATA:

Since in the saturated solution there are almost 6 moles of solvent per mole of salt, the author suggests that $ScCl_3$ has a coordination number of 6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 10-15 cm^3 alcohol and $ScCl_3$ placed in glass stoppered bottles and mechanically rotated at 200 rpm in a thermostat for 14-15 days. After allowing the equilibrated slns to settle for 3 days, aliquots were removed for analyses. Sc determined gravimetrically by evaporation of solvent followed by ignition to Sc_2O_3. Identical results obtained by centrifuging the equilibrated slns prior to analyses.

Solid phase composition determined by analysis of wet residues. Samples dried in vacuum (11 ± 1 mm Hg) over P_2O_5 at 18 ± 1°C and weighed every 24 hours until the mass was constant. Sc and Cl analysed gravimetrically as Sc_2O_3 and AgCl. It is not clear if elemental C and H analyses were also carried out. After 28 days of drying, the solid phase composition was found to be $ScCl_3.4C_3H_7OH$. Additional drying in a desiccator or in a dry box yielded a solid phase of composition $ScCl_3.2C_3H_7OH$.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900-1000°C (1). Source and purity of Sc_2O_3 not specified.

1-Propanol was dried with freshly prepared CaO and distilled.

ESTIMATED ERROR:

Soly: std deviation 0.2 mass % (compiler).

Temp: precision ± 0.2 K.

REFERENCES:

1. Petru, F.; Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Commun.* 1957, *22*, 1534.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) 1-Butanol; $C_4H_{10}O$; [71-36-3]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Z. Chem. 1961, *1*, 332-4.

VARIABLES:

One temperature: T/K = 298.2

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in 1-butanol at 25°C was reported as follows:

	Run 1	Run 2	
	aliquot 1	aliquot 1	aliquot 2
	soly/mass %	soly/mass %	soly/mass %
first analyses	27.9	23.6	23.7
second analyses	27.7	23.7	---
third analyses	27.8	---	---

The mean solubility is 25.2 mass %, and the corresponding (mean) molality calculated by the compiler is 2.23 mol kg^{-1}.

The solid phase was reported to be $ScCl_3.3C_4H_9OH$ which could be further dehydrated to $ScCl_3.2C_4H_9OH$.

COMMENTS AND/OR ADDITIONAL DATA:

Since in the saturated solution there are almost 6 moles of solvent per mole of salt, the author suggests that $ScCl_3$ has a coordination number of 6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 10-15 cm^3 alcohol and $ScCl_3$ placed in glass stoppered bottles and mechanically rotated at 200 rpm in a thermostat for 14-15 days. After allowing the equilibrated slns to settle for 3 days, aliquots were removed for analyses. Sc determined gravimetrically by evaporation of solvent followed by ignition to Sc_2O_3. Identical results obtained by centrifuging the equilibrated slns prior to analyses.

Solid phase composition determined by analysis of wet residues. Samples dried in vacuum (11 ± 1 mm Hg) over P_2O_5 at 18 ± 1°C and weighed every 24 hours until the mass was constant. Sc and Cl analysed gravimetrically as Sc_2O_3 and AgCl. It is not clear if elemental C and H analyses were also carried out. After 29 days of drying, the solid phase composition was found to be $ScCl_3.3C_4H_9OH$. Additional drying in a desiccator or in a dry box yielded a solid phase of composition $ScCl_3.2C_4H_9OH$.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900-1000°C (1). Source and purity of Sc_2O_3 not specified.

1-Butanol was purified by fractional distillation.

ESTIMATED ERROR:

Soly: std deviation about 2.2 mass % (compiler).

Temp: precision ± 0.2 K.

REFERENCES:

1. Petru, F.; Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Commun.* 1957, *22*, 1534.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) 1-Pentanol; $C_5H_{12}O$; [71-41-0]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Z. Chem. 1961, *1*, 332-4.

VARIABLES:

One temperature: T/K = 298.2

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in 1-pentanol at 25°C was reported as follows:

	aliquot 1	aliquot 2
	soly/mass %	soly/mass %
first analyses	23.9	23.7
second analyses	23.7	23.6
third analyses	---	23.5

The mean solubility is 23.7 mass %, and the corresponding (mean) molality calculated by the compiler is 2.05 mol kg^{-1}.

The solid phase was reported to be $ScCl_3.4C_5H_{11}OH$ which could be further dehydrated $ScCl_3.2C_5H_{11}OH$.

COMMENTS AND/OR ADDITIONAL DATA:

Since in the saturated solution there are almost 6 moles of solvent per mole of salt the author suggests that $ScCl_3$ has a coordination number of 6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 10-15 cm^3 alcohol and $ScCl_3$ placed in glass stoppered bottles and mechanically rotated at 200 rpm in a thermostat for 14-15 days. After allowing the equilibrated slns to settle for 3 days, aliquots were removed for analyses. Sc determined gravimetrically by evaporation of solvent followed by ignition to Sc_2O_3. Identical results obtained by centrifuging the equilibrated slns prior to analyses.

Solid phase composition determined by analysis of wet residues. Samples dried in vacuum (11 ± 1 mm Hg) over P_2O_5 at 18 ± 1°C and weighed every 24 hours until the mass was constant. Sc and Cl analysed gravimetrically as Sc_2O_3 and AgCl. It is not clear if elemental C and H analyses were also carried out. After 57 days of drying, the solid phase composition was found to be $ScCl_3.4C_5H_{11}OH$. Additional drying in a desiccator or in a dry box yielded a solid phase of composition $ScCl_3.2C_5H_{11}OH$.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900-1000°C (1). Source and purity of Sc_2O_3 not specified.

1-Pentanol was purified by fractional distillation.

ESTIMATED ERROR:

Soly: std deviation about 0.15 mass % (compiler).

Temp: precision ± 0.2 K.

REFERENCES:

1. Petru, F.: Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Commun.* 1957, *22*, 1534.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) 1-Hexanol; $C_6H_{14}O$; [111-27-3]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Z. Chem. 1961, *1*, 332-4.

VARIABLES:

One temperature: T/K = 298.2

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in 1-hexanol at 25°C was reported as follows:

	aliquot 1 soly/mass %	aliquot 2 soly/mass %
first analyses	22.0	22.2
second analyses	21.4	21.1
third analyses	---	21.1

The mean solubility is 21.5 mass %, and the corresponding (mean) molality calculated by the compiler is 1.81 mol kg^{-1}.

The solid phase was reported to be $ScCl_3.3C_6H_{13}OH$ which could be further dehydrated to $ScCl_3.2C_6H_{13}OH$.

COMMENTS AND/OR ADDITIONAL DATA:

Since in the saturated solution there are almost 6 moles of solvent per mole of salt, the author suggests that $ScCl_3$ has a coordination number of 6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 10-15 cm^3 alcohol and $ScCl_3$ placed in glass stoppered bottles and mechanically rotated at 200 rpm in a thermostat for 14-15 days. After allowing the equilibrated slns to settle for 3 days, aliquots were removed for analyses. Sc determined gravimetrically by evaporation of solvent followed by ignition to Sc_2O_3. Identical results obtained by centrifuging the equilibrated slns prior to analyses.

Solid phase composition determined by analysis of wet residues. Samples dried in vacuum (11 ± 1 mm Hg) over P_2O_5 at 18 ± 1°C and weighed every 24 hours until the mass was constant. Sc and Cl analysed gravimetrically as Sc_2O_3 and AgCl. It is not clear if elemental C and H analyses were also carried out. After 57 days of drying, the solid phase composition was found to be $ScCl_3.3C_6H_{13}OH$. Additional drying in a desiccator or in a dry box yielded a solid phase of composition $ScCl_3.2C_6H_{13}OH$.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900-1000°C (1). Source and purity of Sc_2O_3 not specified.

1-Hexanol was purified by fractional distillation.

ESTIMATED ERROR:

Soly: std deviation about 0.5 mass % (compiler).

Temp: precision ± 0.2 K.

REFERENCES:

1. Petru, F.; Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Commun.* 1957, *22*, 1534.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Scandium chloride; $ScCl_3$; [10361-84-9] (2) 1-Heptanol; $C_7H_{16}O$; [111-70-6]	Kirmse, E. M. *Z. Chem.* 1961, *1*, 332-4.
VARIABLES:	**PREPARED BY:**
One temperature: T/K = 298.2	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in 1-heptanol at 25°C was reported as follows:

	aliquot 1	aliquot 2
	soly/mass %	soly/mass %
first analyses	19.2	19.4
second analyses	19.1	19.7

The mean solubility is 19.3 mass %, and the corresponding (mean) molality calculated by the compiler is 1.58 mol kg^{-1}.

The solid phase was reported to be $ScCl_3.4C_7H_{15}OH$ which could be further dehydrated to $ScCl_3.3C_7H_{15}OH$.

COMMENTS AND/OR ADDITIONAL DATA:

Since in the saturated solution there are almost 6 moles of solvent per mole of salt, the author suggests that $ScCl_3$ has a coordination number of 6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 10-15 cm^3 alcohol and $ScCl_3$ placed in glass stoppered bottles and mechanically rotated at 200 rpm in a thermostat for 14-15 days. After allowing the equilibrated slns to settle for 3 days, aliquots were removed for analyses. Sc determined gravimetrically by evaporation of solvent followed by ignition to Sc_2O_3. Identical results obtained by centrifuging the equilibrated slns prior to analyses.

Solid phase composition determined by analysis of wet residues. Samples dried in vacuum (11 ± 1 mm Hg) over P_2O_5 at 18 ± 1°C and weighed every 24 hours until the mass was constant. Sc and Cl analysed gravimetrically as Sc_2O_3 and AgCl. It is not clear if elemental C and H analyses were also carried out. After 59 days of drying, the solid phase composition was found to be $ScCl_3.4C_7H_{15}OH$. Additional drying in a desiccator or in a dry box yielded a solid phase of composition $ScCl_3.3C_7H_{15}OH$.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900-1000°C (1). Source and purity of Sc_2O_3 not specified.

1-Heptanol was purified by fractional distillation.

ESTIMATED ERROR:

Soly: std deviation about 0.3 mass % (compiler).

Temp: precision ± 0.2 K.

REFERENCES:

1. Petru, F.; Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Commun.* 1957, *22*, 1534.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) 1-Octanol; $C_8H_{18}O$; [111-87-5]

ORIGINAL MEASUREMENTS:
Kirmse, E. M.

Z. Chem. 1961, *1*, 332-4.

VARIABLES:

One temperature: T/K = 298.2

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in 1-octanol at 25°C was reported as follows:

	aliquot 1	aliquot 2
	soly/mass %	soly/mass %
first analyses	16.9	16.9
second analyses	17.0	17.0

The mean solubility is 16.9 mass %, and the corresponding (mean) molality calculated by the compiler is 1.34 mol kg^{-1}.

The solid phase was reported to be $ScCl_3.4C_8H_{17}OH$ which could be further dehydrated to $ScCl_3.3C_8H_{17}OH$.

COMMENTS AND/OR ADDITIONAL DATA:

Since in the saturated solution there are almost 6 moles of solvent per mole of salt, the author suggests that $ScCl_3$ has a coordination number of 6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
Isothermal method. About 10-15 cm^3 alcohol and $ScCl_3$ placed in glass stoppered bottles and mechanically rotated at 200 rpm in a thermostat for 14-15 days. After allowing the equilibrated slns to settle for 3 days, aliquots were removed for analyses. Sc determined gravimetrically by evaporation of solvent followed by ignition to Sc_2O_3. Identical results obtained by centrifuging the equilibrated slns prior to analyses.

Solid phase composition determined by analysis of wet residues. Samples dried in vacuum (11 ± 1 mm Hg) over P_2O_5 at 18 ± 1°C and weighed every 24 hours until the mass was constant. Sc and Cl analysed gravimetrically as Sc_2O_3 and AgCl. It is not clear if elemental C and H analyses were also carried out. After 114 days of drying the solid phase composition was found to be $ScCl_3.4C_8H_{17}OH$. Additional drying in a desiccator or in a dry box yielded a solid phase of composition $ScCl_3.3C_8H_{17}OH$.

SOURCE AND PURITY OF MATERIALS:
Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900-1000°C (1). Source and purity of Sc_2O_3 not specified.

1-Octanol was purified by fractional distillation.

ESTIMATED ERROR:
Soly: std deviation about 0.1 mass % (compiler).

Temp: precision ± 0.2 K.

REFERENCES:

1. Petru, F.; Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Commun.* 1957, *22*, 1534.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) 1-Nonanol; C_9H_2O; [143-08-8]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Z. Chem. 1961, *1*, 332-4.

VARIABLES:

One temperature: T/K = 298.2

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in 1-nonanol at 25°C was reported as follows:

first analyses	13.6 mass %
second analyses	13.4 mass %

The mean solubility is 13.5 mass %, and the corresponding (mean) molality calculated by the compiler is 1.03 mol kg^{-1}.

The solid phase was reported to be $ScCl_3.3C_9H_{19}OH$.

COMMENTS AND/OR ADDITIONAL DATA:

Since in the saturated solution there are almost 6 moles of solvent per mole of salt, the author suggests that $ScCl_3$ has a coordination number of 6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 10-15 cm^3 alcohol and $ScCl_3$ place in glass stoppered bottles and mechanically rotated at 200 rpm in a thermostat for 14-15 days. After allowing the equilibrated slns to settle for 3 days, aliquots were removed for analyses. Sc determined gravimetrically by evaporation of solvent followed by ignition to Sc_2O_3. Identical results obtained by centrifuging the equilibrated slns prior to analyses.

Solid phase composition determined by analysis of wet residues. Samples dried in vacuum (11 ± 1 mm Hg) over P_2O_5 at 18 ± 1°C and weighed every 24 hours until the mass was constant. Sc and Cl analysed gravimetrically as Sc_2O_3 and AgCl. It is not clear if elemental C and H analyses were also carried out. After 146 days of drying the solid phase composition was found to be $ScCl_3.3C_9H_{19}OH$. Additional drying in a desiccator or in a dry box did not change the composition of this solvate.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900-1000°C (1). Source and purity of Sc_2O_3 not specified.

1-Nonanol was purified by fractional distillation.

ESTIMATED ERROR:

Soly: std deviation about 0.1 mass % (compiler).

Temp: precision ± 0.2 K.

REFERENCES:

1. Petru, F.; Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Commun.* 1957, *22*, 1534.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) 2-Ethoxyethanol; $C_4H_{10}O_2$; [110-80-5]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in $C_2H_5OCH_2CH_2OH$ at 25°C was reported to be

26.3 mass %

The corresponding molality calculated by the compiler is

2.36 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified. On the basis of previous papers by the author, it appears that reaction mixtures were equilibrated for several days and that Sc was determined by complexometric titration using xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably, the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Scandium chloride; $ScCl_3$; [10361-84-9] (2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]	Kirmse, E. M.; Zwietasch, K. J. *Z. Chem.* 1967, *7*, 281.
VARIABLES:	PREPARED BY:
One temperature: T/K = 298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in $C_2H_5OCH_2CH_2OC_2H_5$ at 25°C was reported to be

1.22 mass %

The corresponding molatity calculated by the compiler is

0.0816 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
Isothermal method employed. The reaction mixtures were equilibrated in a dry atmosphere with frequent shaking. The solid phase was dried in a vacuum desiccator over P_2O_5. Sc was determined by complexometric titration using xylenol orange indicator. Cl^- determined by the Volhard titration method. In the solid phase the Sc:Cl:ether ratio was found to be 1:3.00:1.13.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was prepared by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:
Nothing specified.

REFERENCES:

1. Taylor, M.D .; Carter, C. P.
   J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Scandium chloride; $ScCl_3$; [10361-84-9] (2) 1-Methoxybutane (butyl methyl ether); $C_5H_{12}O$; [628-28-4]	Kirmse, E.M.; Dressler, H. *Z. Chem.* 1975, *15*, 239-40.
VARIABLES:	**PREPARED BY:**
Room Temperature : T/K = 293-298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in $CH_3(CH_2)_3OCH_3$ at 20-25°C was reported to be

6.5 mass %.

The corresponding molality calculated by the compiler is

0.46 mol kg^{-1}.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated (at room temperature) until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}. Sc was determined by complexometric titration using Xylenol Orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Scandium chloride; $ScCl_3$; [10361-84-9] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	Finke, A.; Kirmse, E. M. Z. Chem. 1965, 5, 193-4.
VARIABLES:	PREPARED BY:
One temperature: T/K = 298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in tetrahydrofuran at 25°C was reported to be

1.0 mass %

The corresponding molality calculated by the compiler is

0.067 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified. The compiler assumes that the method was similar to that described in ref (1).
The equilibrated solid phase was dried at reduced pressure and it was found to be $ScCl_3.2THF$ with $ScCl_3.3THF$ and $ScCl_3.2.5THF$ being intermediate products of drying.
IR spectra of the solution and the dry addition products are discussed. They indicate the formation of coordinate bonds of $ScCl_3$ with oxygen of THF.

SOURCE AND PURITY OF MATERIALS:

Nothing specified except that anhydrous components were used.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E. M.
Z. Chem. 1961, 1, 332.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) Ethyl acetate; $C_4H_8O_2$; [141-78-6]

ORIGINAL MEASUREMENTS:

Finke, A.; Kirmse, E. M.

Z. *Chem.* 1965, 5, 193-4.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in ethyl acetate at 25°C was reported to be

39.2 mass %

The corresponding molality calculated by the compiler is

4.26 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Not specified. The compiler assumes that the method was similar to that described in ref (1).
The equilibrated solid phase was dried at reduced pressure and it was found to be $ScCl_3.2S$ (S = ethyl acetate). IR spectra of the solution and the dry addition product are discussed. The spectra indicate the formation of coordinate bonds of $ScCl_3$ with oxygen of ethyl acetate.

SOURCE AND PURITY OF MATERIALS:

Nothing specified except that anhydrous components were used.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E. M.
Z. *Chem.* 1961, 1, 332.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) Amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Z. Chem. 1961, *1*, 334-7

VARIABLES:

One temperature: T/K = 298.2

PREPARED BY:

Mark Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a]		solid phase
			mass %	mol kg^{-1}	Sc:amine ratio
diethylamine ;	$C_4H_{11}N$;	[109-89-7]	0.034	$2.2_5 \times 10^{-3}$	1:3
dipentylamine;	$C_{10}H_{23}N$;	[2050-92-2]	0.157	1.04×10^{-2}	1:1
triethylamine;	$C_6H_{15}N$;	[121-44-8]	0.012	7.93×10^{-4}	1:3
tri-n-octylamine;	$C_{24}H_{51}N$;	[1116-76-3]	0.004	2.64×10^{-4}	---

[a] Molalities calculated by the compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. $ScCl_3$ and amine placed in glass stoppered bottles (sealed with glycerine) and rotated in a thermostat at 25 ± 0.2°C for 20 days. Scandium was determined gravimetrically as Sc_2O_3.

Samples of the solid phases were dried in vacuum (10 mm Hg) over P_2O_5 to constant weight. Scandium was determined gravimetrically as Sc_2O_3 and chloride determined gravimetrically as AgCl. The nitrogen, carbon and hydrogen contents were determined by "usual microanalytical methods."

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ScCl_3$ prepared by heating Sc_2O_3 and activated charcoal in a stream of chlorine at 900 - 1000°C (1). Source and purity of Sc_2O_3 not specified.

Comercially available amines were fractionated several times.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 0.2 K.

REFERENCES:

1. Petru, F; Hajek, B.; Prochazka, V.; Vit, J. *Collect. Czech. Chem. Comm.* 1957, *22*, 1534.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) Amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$ScCl_3$ solubility[a] mass %	$ScCl_3$ solubility[a] mol kg^{-1}
2-butanamine;	$C_4H_{11}N$;	[13952-84-6]	3.2	0.218
di-isobutylamine;	$C_8H_{19}N$;	[110-96-3]	0.1	0.0066

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to other works of the author which are compiled throughout this volume.

Nature of the solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor , M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$;
[10361-84-9]

(2) Dimethylformamide; C_3H_7NO;
[68-12-2]

ORIGINAL MEASUREMENTS:
Finke, A.; Kirmse, E. M.

Z. Chem. 1965, *5*, 193-4.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in $HCON(CH_3)_2$ at 25°C was reported to be

5.5 mass %

The corresponding molality calculated by the compiler is

0.385 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Not specified. The compiler assumes that the method is similar to that described in ref (1). The equilibrated solid phase was dried under reduced pressure and it was found to be $ScCl_3.2.5S$ (S = dimethylformamide). IR spectra of the saturated solution and of the equilibrated solid phase were studied. They indicate the formation of coordinate bonds of $ScCl_3$ with oxygen of C_3H_7NO.

SOURCE AND PURITY OF MATERIALS:

Nothing specified except that anhydrous components were used.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E. M.
Z. Chem. 1961, *1*, 332.

COMPONENTS:

(1) Scandium chloride; $ScCl_3$; [10361-84-9]

(2) Acetonitrile; C_2H_3N; [75-05-8]

ORIGINAL MEASUREMENTS:

Finke, A.; Kirmse, E. M.

Z. Chem. 1965, *5*, 193-4.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScCl_3$ in acetonitrile at 25°C was reported to be

3.7 mass %

The corresponding molality calculated by the compiler is

$0.254 \text{ mol kg}^{-1}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Not specified. The compiler assumes that the method was similar to that described in ref (1). The equilibrated solid phase was dried at reduced pressure and it was found to be unstable. No new IR bands found for the solution studied.

SOURCE AND PURITY OF MATERIALS:

Nothing specified except that anhydrous components were used.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E. M.
   Z. Chem. 1961, *1*, 332.

COMPONENTS:

(1) Scandium bromide; $ScBr_3$; [13465-59-3]

(2) 1,4-Dioxane (p-dioxane); $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ScBr_3$ in p-dioxane at 25°C was reported to be

1.0 mass %

The corresponding molality calculated by the compiler is

$0.035\ \text{mol kg}^{-1}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified. On the basis of previous papers of the author, it appears that reaction mixtures were equilibrated for several days and Sc determined by complexometric titration using xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably, the anhydrous bromide was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) Alcohols; ROH

(3) Benzene; C_6H_6; [71-43-2]

ORIGINAL MEASUREMENTS:

Golub, A.M.; Yankovich, V.N.
Ukr. Khim. Zh. 1977, *43*, 1139-42;

Ukr. J. Chem. (Engl. Transl.)
1977, *43*, 16-20.

VARIABLES:

Concentration of ROH

T/K = 295

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Numerical data were not given, but results were presented graphically and in the form of the equation

$$K = [YCl_3 \cdot nROH] / [ROH]^n \qquad [1]$$

In this equation $[YCl_3.nROH]$ is the solubility in units of mol dm^{-3}, [ROH] is the <u>total</u> alcohol concentration in units of mol dm^{-3}, and n is the solvate number <u>in solution</u> (see ref. 1). According to this equation, n is calculated from the slope of a plot of the logarithm of the solubility, log $[YCl_3.nROH]$, against log [ROH] . Thus the solubility of YCl_3 can be calculated as a function of ROH concentration using the reported values of n and K (see table below). The alcohol concentrations were varied from 1-5 mol dm^{-3}.

alcohol	n	-log K	nature of the solid phase
methanol; CH_4O; [67-56-1]	1	2.00	$YCl_3.3CH_3OH$
	3	2.90	"
ethanol; C_2H_6O; [64-17-5]	3	3.38	$YCl_3.2C_2H_5OH$
1-propanol; C_3H_8O; [71-23-8]	1	2.00	$YCl_3.3C_3H_7OH$
	3	2.78	"

For those systems where two values of n and K are reported, the overall solubility of YCl_3 is obtained by using the values for n-K in eq. [1] which give the greater solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous YCl_3 at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

Liquid phases were analysed for rare earth metal concentration (method not specified). At least 3 separate experiments were carried out for each system studied. In addition, the solid phases were analysed for several arbitrary points of each series of experiments (method not specified).

SOURCE AND PURITY OF MATERIALS:

Source and purity of YCl_3 not specified. Anhydrous YCl_3 prepared by method described in (2).

C.p. grade organic solvents were purified by "known" methods (3).

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K.

REFERENCES:

1. Golub, A.M.; Golovorushkin, V. I. *Zh. Neorg. Khim.* 1968, *13*, 3194.
2. Spedding, F.H.; Doan, A.H. *J. Am. Chem. Soc.* 1952, *74*, 2783.
3. Kolotyrkin, Ya.M. (ed). *Electrochemistry of Metals in Nonaqueous Solutions.* Khimiya Press. Moscow. 1974. p 440.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [19361-92-9]

(2) Methanol; CH_4O; [67-56-1]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M. N.; Jaccard, P.
Helv. Chim. Acta 1972, 55, 44-52.

Pittelound, M. N.
These. Faculte des Sciences de l'Universite des Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with $YCl_3.4CH_3OH$, the solubility was reported to be 4.38 mol kg^{-1}.

The equilibrated solid phase was analysed and found to contain 4.1 moles of methanol per mole of YCl_3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Yttrium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $YCl_3.4CH_3OH$ confirmed by ^1H NMR and x-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as *J. Inorg. Nucl. Chem.* 1958, 7, 224 (this is the reference to a paper by J. H. Freeman and M. L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Y_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The adduct $YCl_3.4CH_3OH$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and crystallization from methanol. The anhydrous salt was prepared by dehydration as described in (3).

Methanol was purified and dried according to the Vogel method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers)

Temp: precision probably at least ± 0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J. P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Platt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:	EVALUATOR:
(1) Yttrium chloride; YCl_3; [10361-92-9] (2) Ethanol; C_2H_6O; [64-17-5]	Mark Salomon USA ET & DL Ft. Monmouth, NJ, U.S.A.

CRITICAL EVALUATION:

The solubility of YCl_3 in ethanol has been reported only at 298.2 K by Kirmse (1) and by Merbach et al. (2).

Merbach et al. reported two values at 298.15 K as 2.92 mol kg^{-1} and 2.91 mol kg^{-1}. In the former determination, the solid phase analysed as $YCl_3 \cdot 4.1C_2H_5OH$. At 298.2 K Kirmse reported a solubility of 2.82 mol kg^{-1} and a solid phase of $YCl_3 \cdot C_2H_5OH$.

The difference between these two results (3 %) appears to be greater than the experimental precision. In addition it is doubtful that metastability could explain this difference since it is expected that the monosolvate (if it exists) would be metastable at 298 K and thus have a much greater solubility than the stable tetrasolvate. Thus we select the result of Merbach et al. for the *tentative* solubility at 298.15 K in the stable tetrasolvate system: i.e. 2.92 mol kg^{-1}.

REFERENCES

1. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200.

2. Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44: see also Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite Lausanne. 1971.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Yttrium chloride; YCl_3; [10361-92-9] (2) Ethanol; C_2H_6C; [64-17-5]	Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
T/K ≈ 298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in ethanol at 25°C was reported to be

35.5 mass %

The corresponding molality calculated by the compiler is

2.82 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified except that the solid phase was found to be $YCl_3.CH_3CH_2OH$. On the basis of previous papers by the author, it appears that reaction mixtures were isothermally equilibrated for several days and that Y was determined by complexometric titration in the presence of xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [19361-92-9]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M. N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44-52.

Pitteloud, M. N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Two results were reported for 25°C

1. Starting with the solid $YCl_3.4CH_3CH_2OH$, the solubility was reported to be 2.92 mol kg^{-1}. The equilibrated solid phase was analysed and found to contain 4.1 moles of ethanol per mole of YCl_3.
2. Starting with anhydrous YCl_3, the solubility was reported to be 2.91 mol kg^{-1}. The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Yttrium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $YCl_3.4C_2H_6O$ confirmed by ^1H NMR and x-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, *7*, 224 (this is the reference to a paper by J. H. Freeman and M. L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Y_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The adduct $YCl_3.4C_2H_6O$ prepared by dissolving the hydrate in a small excess of o-ethylformate followed by distillation and crystallization from ethanol. The anhydrous salt was prepared by dehydration as described in (3). Ethanol (Fluka) was used as received. Purity and the absence of water was confirmed by NMR.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinch, J. P.; Kalo, A. M. *Helv. Chim. Acta* 1964. *47*, 14.
2. Platt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, Yu.G; Ezhova, T.A.

Zh. Neorg. Khim. 1976, *21*, 551-4; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1976, *21*, 296-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $YCl_3.6H_2O$ in 96.8 % C_2H_5OH[a]

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	35.29	35.40	35.28	35.39	35.34	1.165
30	34.64	34.85	34.74	34.69	34.73	1.145
40	34.47	34.65	34.50	34.46	34.52	1.138
50	34.90	34.95	34.70	34.78	34.83	1.148
60	35.45	35.54	35.49	35.70	35.54	1.172

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$YCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analysed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 151.0 - 151.8°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.9 % (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) 2-Propanol; C_3H_8O; [67-63-0]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M. N.; Jaccard, P.
Helv. Chim. Acta 1972, 55, 44-52.

Pitteloud, M. N.
These. Faculte des Sciences de l'Universite des Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Two results were reported for 25°C

1. Starting with $YCl_3.3C_3H_8O$, the solubility was reported to be 0.91 mol kg^{-1}. The equilibrated solid phase was analysed and found to contain 3.3 moles isopropanol per mole of salt.

2. Starting with anhydrous YCl_3, the solubility was reported to be 0.93 mol kg^{-1}. The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Yttrium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $YCl_3.3C_3H_8O$ confirmed by ^1H NMR and x-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

SOURCE AND PURITY OF MATERIALS:

Y_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The adduct $YCl_3.3C_3H_8O$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and trans-s lvation of the CH_3OH complex with 2-propanol. The anhydrous salt was prepd by dehydration as described in (3).

Iso-propanol (Fluka) used as received. Purity and absence of water confirmed by NMR.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, *7*, 224 (this is the reference to a paper by J. H. Freeman and M. L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

REFERENCES:

1. Brunisholz, F.; Quinche, J. P.; Kalo, A. M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Platt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) 2-Methoxyethanol; $C_3H_8O_2$; [109-86-4]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in 2-methoxyethanol at 25°C was reported to be

2.7 mass %

The corresponding molality calculated by the compiler is

$0.142 \text{ mol kg}^{-1}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified except that the solid phase was found to be a solvate containing 2-3 moles solvent per mole of salt. On the basis of previous papers by the author, it seems that reaction mixtures were thermostated for several days, and that Y was determined by complexometric titration in the presence of xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably, the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) 2-Ethoxyethanol; $C_4H_{10}O_2$; [110-80-5]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teorl Rastvorov 1971, 200-6.

VARIABLES:

One temperature: $T/K = 298$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in 2-ethoxy-ethanol at 25°C was reported to be

6.9 mass %

The corresponding molality calculated by the compiler is

0.38 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified except that the solid phase was found to be $YCl_3.2C_4H_{10}O_2$. On the basis of previous papers by the author, it appears that reaction mixtures were equilibrated for several days and that Y was determined by complexometric titration using xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably, the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) 1-Ethoxy-2-methoxyethane; $C_5H_{12}O_2$; [5137-45-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.; Zwietasch, K. J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472-3:

Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in $C_2H_5OCH_2CH_2OCH_3$ at 25°C was reported to be

0.4 mass %

The corresponding molality calculated by the compiler is

0.02 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated until equilibrium was attained at 25°C or/and at room temperature (the difference found in the solubility was within the limits of experimental error). Y was determined by complexometric titration (no further details given).

SOURCE AND PURITY OF MATERIALS:

Nothing specified except that the anhydrous chloride was obtained by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3;
[10361-92-9]

(2) 1-Methoxybutane; $C_5H_{12}O$;
[628-28-4]

ORIGINAL MEASUREMENTS:
Schmalenberg, U. *Staatsexamenarbeit.*
Koethen. Paedag. Institut. 1969:
Kirmse, E. M.; Dressler, H.
Z. Chem. 1975, *15*, 239-40.

VARIABLES:

One temperature: T/K = 293-298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in 1-methoxybutane at 20-25°C was reported to be

29.7 mass %

The corresponding molality calculated by the compiler is

2.16 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were equilibrated isothermally at room temperature until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}. Y was determined by complexometric titration using xylenol orange indicator. The reported solubility is a mean value of four determinations. The hydrate $YCl_3.6H_2O$ was found to be insoluble in $C_5H_{12}O$.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) 1-Ethoxybutane; $C_6H_{14}O$; [628-81-9]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.; Zwietasch, K. J.; Tirschmann, J. *Wiss. Hefte, Paed. Inst. Koethen* 1968, *1*, 128-30:

Kirmse, E. M.; Zwietasch, K. J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472-3:

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in 1-ethoxybutane at 25°C was reported to be

0.03 mass %

The corresponding molality calculated by the compiler is

2.6×10^{-3} mol kg^{-1}

Note that the solubility for YCl_3 reported in *Z. Chem.* is 0.05 mass % (0.0015 mol kg^{-1}).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

YCl_3 was isothermally agitated at 25°C with $C_6H_{14}O$ until equilibrium was attained. Y was determined by complexometric titration using xylenol orange indicator. The solid phase is $YCl_3 \cdot 0.37C_6H_{14}O$. No other details available.

SOURCE AND PURITY OF MATERIALS:

The anhydrous chloride was prepared by the method of Taylor and Carter (1). No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:	EVALUATOR:
(1) Yttrium chloride; YCl_3; [10361-97-9] (2) 1-Methoxypentane; C_6H_4O; [628-80-8]	Mark Salomon USA ET & DL Ft. Monmouth, NJ, U.S.A.

CRITICAL EVALUATION:

The solubility of yttrium chloride in 1-methoxypentane has been reported in four publications by Kirmse et al. In all publications the solubility was determined at 298.2 K, and the nature of the solid phase was not identified.

The studies in (1-3) report the solubility of 0.158 mol kg^{-1}, and it is not known if these are independent measurements. The result from (4) is 0.083 mol kg^{-1}, and it is therefore apparent that one of these values is in error.

REFERENCES

1. Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J. *Wiss. Hefte, Paed. Inst. Koethen* 1968, *1*, 128.

2. Kirmse, E.M.; Zwietasch, K.J., Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472.

3. Kirmse, E.M. *Tr. II Vses. Konf. po Theor. Rastvorov* 1971, 200.

4. Kirmse, E.M.; Dressler, H. *Z. Chem.* 1975, *15*, 239.

COMPONENTS:

(1) Yttrium chloride; YCl_3;
[10361-92-9]

(2) 1-Methoxypentane; $C_6H_{14}O$;
[628-80-8]

ORIGINAL MEASUREMENTS:

1. Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J. *J. Wiss. Hefte Paed. Inst. Koethen* 1968, *1*, 128-30:
2. Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472-3:
3. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in 1-methoxypentane at 25° was reported to be

3.0 mass %

The corresponding molality calculated by the compiler is 0.158 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

YCl_3 was isothermally agitated at 25°C until saturation (no details given on how equilibrium was ascertained). Yttrium was determined by complexometric titration using Xylenol Orange indicator.

The solid phase was analysed and found to be $YCl_3.0.39C_5H_{14}O$.

No other details given.

SOURCE AND PURITY OF MATERIALS:

Anhydrous YCl_3 was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Dressler, H.

Z. *Chem.* 1975, *15*, 239-40.

VARIABLES:

Room temperature: T/K = 293-298

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

solvent			YCl_3 solubility[a]	
			mass %	mol kg^{-1}
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	1.6	0.083
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	2.7	0.14[b]
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	2.5	0.13
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	2.5	0.13
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	2.8	0.15

[a] Molalities calculated by the compilers.

[b] Solid phase is $YCl_3 \cdot C_8H_{18}O$ as determined by complexometric titration after drying in a vacuum desiccator over P_2O_5.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were agitated at room temperature until the solutions were saturated. The anhydrous reagents were handled in a dry box containing P_2O_5.

Yttrium was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations for each system.

SOURCE AND PURITY OF MATERIALS:

No information given.

ESTIMATED ERROR:

No information given.

REFERENCES:

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.; Auer-Welsbach, C.

Monatsh. Chem. 1965, *96*, 602-5.

VARIABLES:

Room temperature: T/K about 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in tetrahydrofuran at room temperature (about 20°C) was reported as

0.930 g/100 ml solution

The solid phase is $YCl_3 \cdot 3.59C_4H_8O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor for 60-80 hours at room temperature. Yttrium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. For the solid phase analysis, the solvent was determined by difference.

Anhydrous substances were handled in a dry box through which was passed a current of dry and CO_2-free nitrogen.

SOURCE AND PURITY OF MATERIALS:

Sources and purities not specified.

YCl_3 prepared by reaction of the oxide at high temperatures with an excess of NH_4Cl followed by heating the product in a current of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) 1,4-Dioxane (p-dioxane); $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.; Zwietasch, K. J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472-3.

Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in 1,4-dioxane at 25°C was reported to be

0.1 mass %

The corresponding molality calculated by the compiler is

5.1×10^{-3} mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were equilibrated isothermally by agitation at 25°C or/and at room temperature. (The difference found in the solubilities was within the limits of experimental error. Y was determined by complexometric titration. No other information given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous chloride was obtained by the method of Taylor and Carter (1). No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) Alcohols; ROH

(3) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Golub, A.M.; Yankovich, V.N.
Ukr. Khim. Zh. 1977, *43*, 1139-42;

Ukr. J. Chem. (Engl. Transl.)
1977, *43*, 16-20.

VARIABLES:

Concentration of ROH
T/K = 295

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Numerical data were not given, but results were presented graphically and in the form of the equation

$$K = [YCl_3.nROH] / [ROH]^n \qquad [1]$$

In this equation $[YCl_3.nROH]$ is the solubility in units of mol dm^{-3}, [ROH] is the total alcohol concentration in units of mol dm^{-3}, and n is the solvate number in solution (see ref. 1). According to this equation, n is calculated from the slope of a plot of the logarithm of the solubility, log $[YCl_3.nROH]$, against log [ROH] . Thus the solubility of YCl_3 can be calculated as a function of ROH concentration using the reported values of n and K (see table below). The alcohol concentrations were varied from 1-5 mol dm^{-3}.

alcohol	n	-log K	nature of the solid phase
methanol; CH_4O; [67-56-1]	1	1.40	$YCl_3.3CH_3OH$
	2	1.80	
ethanol; C_2H_6O; [64-17-5]	2	2.67	$YCl_3.2C_2H_5OH$
	3	3.17	
1-propanol; C_3H_8O; [71-23-8]	1	1.65	$YCl_3.3C_3H_7OH$
	2	2.00	

For the systems, two values of n and K are reported, and the overall solubility of YCl_3 is obtained by using the values for n-K in eq. [1] which give the greater solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous YCl_3 at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

Liquid phases were analysed for rare earth metal concentration (method not specified). At least 3 separate experiments were carried out for each system studied. In addition, the solid phases were analysed for several arbitrary points of each series of experiments (method not specified).

SOURCE AND PURITY OF MATERIALS:

Source and purity of YCl_3 not specified. Anhydrous YCl_3 prepared by method described in (2).

C.p. grade organic solvents were purified by "known" methods (3).

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K

REFERENCES:

1. Golub, A.M.; Golovorushkin, V. I. *Zh. Neorg. Khim.* 1968, *13*, 3194.
2. Spedding, F.H.; Doan, A. H. *J. Am. Chem. Soc.* 1952, *74*, 2783.
3. Kolotyrkin, Ya.M. (ed). *Electrochemistry of Metals in Nonaqueous Solutions.* Khimiya Press. Moscow. 1974. p 440.

COMPONENTS: (1) Yttrium chloride; YCl_3; [10361-92-9] (2) Diethylamine; $C_4H_{11}N$; $C_4H_{11}N$; [109-89-7]	ORIGINAL MEASUREMENTS: Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: One temperature: T/K = 298	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in diethylamine, $(C_2H_5)_2NH$, at 25°C was reported to be

1.2 mass %

The corresponding value recalculated by the compiler is

0.062 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: Nothing specified. On the basis of previous papers by the author, it seems that reaction mixtures were thermostated for several days, and that Y was determined by complexometric titration in the presence of xylenol orange indicator. For the solid phase, the ratio Y:Cl:amine was found to be 1:2.9:2.6.	SOURCE AND PURITY OF MATERIALS: Nothing specified. Presumably, the anhydrous chloride was prepared by the method of Taylor and Carter (1). ESTIMATED ERROR: Nothing specified. REFERENCES: 1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS: (1) Yttrium chloride; YCl_3; [10361-92-9] (2) 1-Propanamine (propylamine); C_3H_9N; [107-10-8]	ORIGINAL MEASUREMENTS: Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: One temperature: T/K = 298	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in $CH_3CH_2CH_2NH_2$ at 25°C was reported to be

15.0 mass %

The corresponding molality calculated by the compiler is

0.904 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: Nothing specified. On the basis of previous papers by the author, it seems that reaction mixtures were thermostated for several days, and that Y was determined by complexometric titration in the presence of xylenol orange indicator.	SOURCE AND PURITY OF MATERIALS: Nothing specified. Presumably, the anhydrous chloride was prepared by the method of Taylor and Carter (1).
	ESTIMATED ERROR: Nothing specified.
	REFERENCES: 1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS: (1) Yttrium chloride; YCl_3; [10361-92-9] (2) 2-Propanamine (isopropylamine); C_3H_9N; [75-31-0]	ORIGINAL MEASUREMENTS: Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: One temperature: T/K = 298	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in $(CH_3)_2CHNH_2$ at 25°C was reported to be

14.1 mass %

The corresponding molality calculated by the compiler is

0.84 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified. On the basis of previous papers by the author, it seems that reaction mixtures were thermostated for several days, and that Y was determined by complexometric titration in the presence of xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably, the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Yttrium chloride; YCl_3; [10361-92-9] (2) 2-Propen-1-amine (allylamine); C_3H_7N; [107-11-9]	Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	PREPARED BY:
One temperature: T/K = 298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in $H_2C{=}CHCH_2NH_2$ at 25°C was reported to be

35.6 mass %

The corresponding molality calculated by the compiler is

2.83 mol kg^{-1}

[a] In the original paper, only the formula $C_3H_5NH_2$ is given, but on the compiler's request the author kindly specified the solvent name.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified. On the basis of previous works by the author, it appears that solute-solvent mixtures were equilibrated isothermally for several days and that Y was determined by complexometric titration using xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably anhydrous YCl_3 was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) 1-Butanamine (n-butylamine); $C_4H_{11}N$; [109-73-9]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in $CH_3(CH_2)_3NH_2$ at 25°C was reported to be

21.1 mass %

The corresponding molality calculated by the compiler is

1.37 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified. On the basis of previous papers by the author, it seems that reaction mixtures were thermostated for several days, and that Y was determined by complexometric titration in the presence of xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably, the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) 2-Butanamine (sec-butylamine); $C_4H_{11}N$; [13952-84-6]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in $C_2H_5CH(NH_2)CH_3$ at 25°C was reported to be

11.3 mass %

The corresponding molality calculated by the compiler is

0.652 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified. On the basis of previous papers by the author, it seems that reaction mixtures were thermostated for several days, and that Y was determined by complexometric titration in the presence of xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Yttrium chloride; YCl_3; [10361-92-9]

(2) Di-isobutylamine; $C_8H_{19}N$; [110-96-3]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II. Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YCl_3 in $(CH_3)_2CHCH_2\ _2NH$ at 25°C was reported to be

0.6 mass %.

The corresponding molality calculated by the compiler is

0.03 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified. On the basis of previous papers by the author, it seems that reaction mixtures were thermostated for several days, and that Y was determined by complexometric titration in the presence of xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably, the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Yttrium bromide; YBr_3;
[13469-98-2]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$;
[629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YBr_3 in $C_2H_5OCH_2CH_2OC_2H_5$ at 25°C was reported to be

1.2 mass %

The corresponding molality calculated by the compiler is

$0.037 \text{ mol kg}^{-1}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified. On the basis of previous papers by the author, it seems that reaction mixtures were thermostated for several days, and that Y was determined by complexometric titration in the presence of xylenol orange indicator.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably the anhydrous bromide was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Yttrium bromide; YBr_3; [13469-98-2]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. 1966, 97, 1357-64.

VARIABLES:

Room temperature: T/K about 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YBr_3 in tetrahydrofuran at 21-23°C was reported to be

0.16 g/100 ml solution

The solid phase is $YBr_3.3.5C_4H_8O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor for 60-80 hours at room temperature. Yttrium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. For the solid phase analysis, the solvent was determined by difference.

Anhydrous substances were handled in a dry box through which was passed a current of dry and CO_2-free nitrogen.

SOURCE AND PURITY OF MATERIALS:

Sources and purities not specified. YBr_3 prepared by reaction of the oxide at high temperatures with an excess of NH_4Br followed by heating the product in a current of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Yttrium bromide; YBr_3; [13469-98-2]

(2) 1,4-Dioxane (p-dioxane); $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.; Zwietasch, K. J.; Tirschmann, J.; Oelsner, L.; Niedergessaess, U. *Z. Chem.* 1968, *8*, 472-3

Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

VARIABLES:

One temperature: T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YBr_3 in 1,4-dioxane at 25°C was reported to be

1.6 mass %

The corresponding molality calculated by the compiler is

0.049 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or/and at room temperature (the difference found in the solubility was within the limits of experimental error). Y was determined by complexometric titration. No other information given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous bromide was obtained by the method of Taylor and Carter (1). No other details given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Yttrium iodide; YI_3; [13470-38-7]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Kachkimbaeva, S. A.; Chalova, E. P.; Bleshinskii, S. V.

Khim. Kompleks. Soedin. Redk. Soputstvuyushchikh Elem. 1970, 122-6.

VARIABLES:

One temperature: T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YI_3 in tetrahydrofuran at 20°C was reported to be

$$1.53\ \text{g dm}^{-3}\quad (3.26 \times 10^{-3}\ \text{mol dm}^{-3},\ \text{compiler}).$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were equilibrated isothermally by agitation. The phases separated by decantation and in some cases by centrifuging. Y determined by the oxalate method. I determined by titration with an $AgNO_3$ solution (the Volhard method).

SOURCE AND PURITY OF MATERIALS:

YI_3 prepared from "c.p." grade I_2 and excess powdered metal (Y-0-Sort) by heating in an ampule to 1200°C. YI_3 sublimated from the hot to the cold part of the ampoule. The product was analysed for Y and I (results not given).

"C.p." grade tetrahydrofuran, b.p.=65.6°C, dried with NaOH and distilled from metallic sodium.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Bis-(2-ethylhexyl)phosphoric acid; $C_{16}H_{35}O_4P$; [298-07-7]

(3) Petroleum ether

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in 1 m solution of di(2-ethylhexl)phosphoric acid in petroleum ether at room temperature was reported to be

0.02 mass %

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be about 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temp for 100 hours. Samples of satd solution for analyses were obtaine by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. The $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The other components were purified and dried by standard methods.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Halongenated organic solvents

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen. GDR. 1980.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			LaF_3 solubility[a] mass %	mol kg^{-1}	solid phase La:F:solvent ratio
1-(chloromethoxy)propane;	C_4H_9ClO;	[3587-57-3]	0.026	1.33×10^{-3}	1:2.70:0.52
1-(chloromethoxy)butane;	$C_5H_{11}ClO$;	[2351-69-1]	0.045 0.05	2.30×10^{-3} 2.6×10^{-3}	b
1-(chloromethoxy)pentane;	$C_6H_{13}ClO$;	[19416-65-0]	0.022	1.12×10^{-3}	1:2.41:0.40
1-(bromomethoxy)butane;	$C_5H_{11}BrO$;	[59375-51-8]	0.053	2.71×10^{-3}	1:3.19:0.40
1-(bromomethoxy)pentane;	$C_6H_{13}BrO$;		0.055	2.81×10^{-3}	1:2.43:0.22
Nonafluoro-1-butanesulfonyl fluoride;	$C_4F_{10}SO_2F$;	[375-72-4]	0.060	3.06×10^{-3}	1:3.12:0.12
heptadecafluoro-1-octanesulfonyl fluoride;	$C_8F_{18}O_2S$;	[307-35-7]	0.011	5.62×10^{-4}	1:2.90:0.06

[a] Molalities calculated by the compilers.

[b] La:F:ether:H_2O ratio reported as 1:2.96:0.23:0.54

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the La:F:H_2O ratio was 1:3.01:0.20.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E. M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Benzene; C_6H_6; [71-43-2]

(3) Substituted benzenes

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen. GDR. 1980.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

The solubility of LaF_3 was determined in three substituted benzene solvents containing 0.5 mol dm^{-3} benzene.

solvent (component 3)			LaF_3 solubility[a] mass %	LaF_3 solubility[a] mol kg^{-1}	solid phase La:F:solvent ratio
1,3-dinitrobenzene;	$C_6H_4N_2O_4$;	[99-65-0]	0.026	1.33×10^{-3}	1:2.89:0.16
1-fluoro-2,4-dinitrobenzene;	$C_6H_3FN_2O_4$;	[70-34-8]	0.011	5.62×10^{-4}	1:3.02:0.20
1-chloro-2,4-dinitrobenzene;	$C_6H_3ClN_2O_4$;	[97-00-7]	0.015	7.66×10^{-4}	1:3.05:0.17

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the La:F:H_2O ratio was 1:3.01:0.20.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E. M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Benzenamine (aniline); C_6H_7N; [62-53-3]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in aniline at room temperature was reported as

0.03 mass %.

The corresponding molality calculated by the compiler is

$$1.5 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum fluoride; LaF_3; [13709-38-1] (2) Methanol; CH_4O; [67-56-1]	Kirmse, E. M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, 2, 85-90.
VARIABLES: Room temperature	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in methanol was reported to be

0.02 mass %

The corresponding molality calculated by the compiler is

1.0×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Urea; CH_4N_2O; [57-13-6]

(3) Methanol; CH_4O; [67-56-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature : T/K = 291-295

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in methanol saturated with urea at room temperature was reported to be

$$1.7 \times 10^{-2} \text{ mass } \%$$

The urea content was not given.

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be about 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temp (18-22°C) for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 20 % KOH solution for 3 hours to obtaine quantitative separation of solid $La(OH)_3$ and a basic F^- solution. The $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The other components were purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum fluoride; LaF_3; [13709-38-1] (2) Ethanol; C_2H_6O; [64-17-5]	Kirmse, E. M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES:	PREPARED BY:
Room temperature	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in ethanol was reported to be

0.03 mass %

The corresponding molality calculated by the compiler is

1.5×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lenthanum fluoride; LaF_3; [13709-38-1]

(2) Urea; CH_4N_2O; [57-13-6]

(3) Ethanol; C_2H_6O; [67-17-5]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss, Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in ethanol saturated with urea at room temperature was reported to be

$$5 \times 10^{-3} \text{ mass \%.}$$

The urea content was not given.

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be about 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 g of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The other components were purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) 1,2-Ethanediol (ethylene glycol); $C_2H_6O_2$; [107-21-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, 2, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in ethylene glycol at room temperature was reported to be

0.02 mass %

The corresponding molality calculated by the compiler is

$$1.0 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3;
[13709-38-1]

(2) 2-Methyl-1-propanol (isobutanol);
$C_4H_{10}O$; [78-83-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in isobutanol at room temperature was reported to be

0.03 mass %

The corresponding molality calculated by the compiler is

1.5×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temp for 50 hours. Samples of saturated solution for analyses were obtained by decantation. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

The reported solubility is the mean of at least two determinations.

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3.0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS: (1) Lanthanum fluoride; LaF_3; [13709-38-1] (2) 2-Butanol; $C_4H_{10}O$; [78-92-2]	ORIGINAL MEASUREMENTS: Kirmse, E. M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES: Room temperature	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in 2-butanol at room temperature was reported to be

0.02 mass %

The corresponding molality calculated by the compiler is

1.0×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La determined several times by complexometric titration using a potentiometric method (2). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) 2-Methyl-2-butanol (t-pentanol); $C_5H_{12}O$; [75-85-4]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss, Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in t-pentanol at room temperature was reported to be

$$0.02_5 \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$1.3 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was dtermined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen. GDR. 1980.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			LaF_3 solubility[a] mass %	mol kg^{-1}	solid phase La:F:solvent ratio
1,2-dimethoxyethane;	$C_4H_{10}O_2$;	[110-71-4]	0.017	8.68×10^{-4}	1:2.82:0.31
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.005	2.6×10^{-4}	1:2.87:0.26
1-methoxy-2-methylpropane;	$C_5H_{12}O$;	[625-44-5]	0.002	1.0×10^{-4}	1:2.97:0.36
1-methoxybutane;	$C_5H_{12}O$;	[628-28-4]	0.003	1.5×10^{-4}	b
1-methoxypentane	$C_6H_{14}O$;	[628-80-8]	0.005	2.6×10^{-4}	1:3.20:0.33
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	0.004	2.0×10^{-4}	1:2.88:0.22
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	0.003	1.5×10^{-4}	c
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	0.020	1.02×10^{-3}	1:2.98:0.11

[a] Molalities calculated by the compilers.

[b] La:F:ether:H_2O ratio reported as 1:2.87:0.20:0.58

[c] La:F:ether:H_2O ratio reported as 1:2.99:0.18:0.52

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the La:F:H_2O ratio was 1:3.01:0.20.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E. M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum fluoride; LaF_3; [13709-38-1] (2) Ethyl acetate (acetic acid ethyl ester); $C_4H_8O_2$; [141-78-6]	Kirmse, E. M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES:	PREPARED BY:
Room temperature	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in ethyl acetate at room temperature was reported to be

0.01 mass %

The corresponding molality calculated by the compiler is

5×10^{-4} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum fluoride; LaF_3; [13709-38-1] (2) Tributyl phosphate ; $C_{12}H_{27}O_4P$; [126-73-8]	Kirmse, E. M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES:	PREPARED BY:
Room temperature	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in tributyl phosphate at room temperature was reported to be

0.03 mass %

The corresponding molality calculated by the compiler is

$$1.5 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtaine quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Dimethylsulfoxide; C_2H_6OS; [67-68-5]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in dimethylsulfoxide at room temperature was reported to be

0.03 mass %

The corresponding molality calculated by the compiler is

1.5×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3.0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) 2-Aminoethanol (ethanolamine); C_2H_7NO; [141-43-5]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in ethanolamine at room temperature was reported to be

0.03 mass %

The corresponding molality calculated by the compiler is

$$1.5 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were hearted with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Triethylamine ; $C_6H_{15}N$; [121-44-8]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature: T/K about 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in triethylamine at room temperature was reported to be

$$0.01 \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$5\ 10^{-4} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature (about 25°C) for 100 h Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH sln for 3-5 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. The $La(OH)_3$ was filtered, washed and dissolved in HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3;
[13709-38-1]

(2) 2-Propanamine ;
C_3H_9N; [75-31-0]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in isopropylamine at room temperature was reported to be

0.02 mass %

The corresponding molality calculated by the compiler is

1.0×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt (LaF_3.0.5H_2O) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3;
[13709-38-1]

(2) Di-isobutylamine; $C_8H_{19}N$;
[110-96-3]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in di-isobutylamine at room temperature was reported to be

$$0.01_5 \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$7.7 \times 10^{-4} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanide fluoride; LaF_3; [13709-38-1]

(2) Pyridine; C_5H_5N; [110-86-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.; Jordan, M.
Z. Chem. 1977, *17*, 75-6.

VARIABLES:

Temperature not specified: presumably room temperature.

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in pyridine was reported to be

$$4 \times 10^{-2} \text{ mass } \%$$

The corresponding value in molal units calculated by the compiler is

$$2 \times 10^{-3} \text{ mol kg}^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. A sample of saturated solution was evaporated to dryness, and about 30 mg of LaF_3 heated with about 10 cm^3 of 10 % KOH solution for 1 hour to obtain quantitative separation of the precipitated $La(OH)_3$ and the fluoride solution. The precipitate was filtered, washed and dissolved in dil HCl. La was determined by complexometric titration with potentiometric end-point detection. The fluoride content of the filtrate was determined photometrically using Al-Eriochrome cyanine color lake.

SOURCE AND PURITY OF MATERIALS:

Technical grade LaF_3 (VEB Fluowerke Dohna) contained 3.5 % Pr_6O_{11} and 14.5 % Nd_2O_3.

The salt ($LaF_3 . 0.5H_2O$) was dehydrated by washing with acetone and drying at 300°C (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Karpenko, L. I.; Fadeeva, L.A.; Shevchenko, L. D. *Zh. Anal. Khim.* 1975, *30*, 1330.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3;
[13709-38-1]

(2) Pyridine; C_5H_5N; [110-86-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in pyridine at room temperature was reported to be

$$0.03_5 \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$1.8 \times 10^{-3} \text{ mol kg}^{-1}.$$

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be very close to 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Urea; CH_4N_2O; [57-13-6]

(3) Pyridine; C_5H_5N; [110-86-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature : T/K = 291-294

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 at room temperature in pyridine saturated with urea was reported to be

$$1.2 \times 10^{-2} \text{ mass \%}$$

The urea content was not given.

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be about 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature (18-22°C) for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH to obtaine quantitative separation of solid $La(OH)_3$ and a basic F^- solution. The solid $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method.(1).

The reported solubility is the mean of at least two determinations.

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The other components were purified and dried by standard methods.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-]]

(2) Acidic nitrosyl fluoride; NOF.3HF; [14947-17-2]

ORIGINAL MEASUREMENTS:

Galkin, N. P.; Shishkov, Yu. D.
Khomyakov, V. I.
Radiokhimiya 1978, *20*, 136-41;
Soviet Radiochem. (Engl. Transl.) 1978, *20*, 109-13.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in acidic nitrosyl fluoride at room temperature was reported to be

0.05 mass %

The molality calculated by the compiler is

$$2.6 \times 10^{-3}\ \text{mol kg}^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solute-solvent mixture was placed in a Teflon vessel and mechanically agitated at room temperature for 10 h. The reaction mixture was allowed to settle for 24 h and the supernatant saturated solution was analysed for the La content. An aliquot was evaportated to dryness under vacuum at 100-150°C, and the dry residue dissolved and analysed (the method of analysis not specified).

The solid phase is LaF_3 as found by analyses for F,N,HF and La.

SOURCE AND PURITY OF MATERIALS:

LaF_3 was at least 99 % pure.

NOF.3HF prepared by saturation of liquid HF with NOF, and was distilled twice at 95°C before use. The melting point of acidic nitrosyl fluoride was 3.8°C.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Lanthanum fluoride; LaF_3; [13709-38-1]

(2) Urea; CH_4N_2O; [57-13-6]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of LaF_3 in 46% aqueous solution of urea at room temperature was reported to be

$$9 \times 10^{-3} \text{ mass } \%$$

The solid phase was dried in a desiccator over P_4O_{10} and its La:F ratio was found to be about 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg LaF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-3 hours to obtain quantitative separation of solid $La(OH)_3$ and a basic F^- solution. $La(OH)_3$ was filtered, washed and dissolved with HCl. La was determined several times by complexometric titration using a potentiometric method (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

SOURCE AND PURITY OF MATERIALS:

La_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated with HF. The ppt ($LaF_3 \cdot 0.5H_2O$) was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The other components were purified and dried by standard methods.

ESTIMATED ERROR:

Soly: results for which rel error exceeded 50% were rejected. The reported value is a mean of at least two detns.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E. M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E. M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Hexachloro-1,3-butadiene; C_4Cl_6; [87-68-3]

ORIGINAL MEASUREMENTS:

Shevtsova, Z.N.; Korshunov, B.G.; Safonov, V.V.; Kogan, L.M.; Gudkova, V.I.

Zh. Neorg. Khim. 1968, *13*, 3096-9; *Russ, J. Inorg. Chem. (Engl. Transl.)* 1968, *13*, 1596-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition, densities, viscosities and refractive indices of saturated solutions.

	solubility[a]					nature of the
$t/^{\circ}C$	mass %	mol kg^{-1}	$d/g\ cm^{-3}$	η/P	n_D^{20}	solid phase
25	0.040	0.00163	1.681	0.0382	1.5564	$LaCl_3.4H_2O$
50	0.042	0.00171	1.642	0.0305	1.5554	$LaCl_3.4H_2O$
75	0.057	0.00233	1.614	0.0244	1.5547	$LaCl_3.2H_2O$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Depending on temp, equil was established after 12 d at 25°C, 10 d at 50°C, and 7 d at 75°C.

Chloride was detd by the Volhard method, and lanthanum detd gravimetrically by precipitating as the oxalate and igniting to the oxide. Lanthanum was also detd by titration with Trilon B with Xylene Orange indicator.

The composition of the solid phase was established by chemical analysis, and confirmed by X-ray analysis.

Samples of the solid phases were also studied thermographically after removal of excess solvent by washing with absolute ethyl ether which is claimed not to change the composition of hydrate.

Authors state that at 110°C the equil solid phase is the monohydrate, but no solubility data are given. At 120°C partial hydrolysis takes place with formation of LaOCl.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3.7H_2O$ prepd by dissolving La_2O_3 in HCl, evaporating and cooling, and then recrystallized and dried in a desiccator. La_2O_3, 99.9% pure, contained oxide impurities of other rare earth metals, Fe (0.01%), Ca (0.01-0.05 %), and Cu (0.01%). Analysis of the heptahydrate gave the following (in mass % units): La 37.42; Cl 28.67; H_2O 33.91.

Purified solvent (method not specified) had the following properties: d_4^{20} = 1.6807 g/ml, and n_D^{20} = 1.5543.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: accuracy ± 0.1 K (authors).

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum chloride; $LaCl_3$; [10099-58-8] (2) Methanol; CH_4O; [67-56-1] (3) Benzene; C_6H_6; [71-43-2]	Golub, A.M.; Yankovich, V.N. *Ukr. Khim. Zh.* 1977, *43*, 1139-42; *Ukr. J. Chem. (Engl. Transl.)* 1977, *43*, 16-20.
VARIABLES:	**PREPARED BY:**
Concentration of CH_3OH T/K = 295	T. Mioduski

EXPERIMENTAL VALUES:

Initial Concn Methanol mol dm^{-3}	$LaCl_3$ solubility[a] mol dm^{-3}
1.0	0.00225
1.5	0.00580
2.0	0.01017
2.5	0.01600
3.0	0.02350
3.5	0.03155
4.0	0.04169

[a] Solid phase is $LaCl_3 \cdot CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous $LaCl_3$ at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

Liquid phases were analysed for rare earth metal concentration (method not specified). At least 3 separate experiments were carried out for each system studied. In addition, the solid phases were analysed for several arbitrary experimental points (method not specified).

SOURCE AND PURITY OF MATERIALS:

Source and purity of $LaCl_3$ not specified. Anhydrous $LaCl_3$ prepared by method described in (2).

C.p. grade organic solvents were purified by "known" methods (3).

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K.

REFERENCES:

1. Golub, A.M.; Golovorushkin, V. I. *Zh. Neorg. Khim.* 1968, *13*, 3194.
2. Spedding, F.H.; Doan, A.H. *J. Am. Chem. Soc.* 1952, *74*, 2783.
3. Kolotyrkin, Ya.M. (ed). *Electrochemistry of Metals in Nonaqueous Solutions.* Khimiya Press. Moscow, 1974. p 440.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Alcohols; ROH

(3) Benzene; C_6H_6; [71-43-2]

ORIGINAL MEASUREMENTS:

Golub, A.M.; Yankovich, V. N.
Ukr. Khim. Zh. 1977, *43*, 1139-42;

Ukr. J. Chem. (Engl. Transl.) 1977, *43*, 16-20.

VARIABLES:

Concentration of ROH
T/K = 295

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Numerical data were given only for the $LaCl_3$ - CH_3OH - C_6H_6 system (see the compilation for this system). The remaining data were presented graphically and in the form of the equation

$$K = [LaCl_3.nROH] / [ROH]^n \qquad [1]$$

In this equation $[LaCl_3.nROH]$ is the solubility in units of mol dm^{-3}, [ROH] is the total alcohol concentration in units of mol dm^{-3}, and n is the solvate number in solution (see ref. 1). According to this equation, n is calculated from the slope of a plot of the logarithm of the solubility, log $[LaCl_3.nROH]$, against log [ROH]. Thus the solubility of $LaCl_3$ can be calculated as a function of ROH concentration using the reported values of n and K (see table below). The alcohol concentrations were varied from 1-5 mol dm^{-3}.

alcohol	n	-log K	nature of the solid phase
methanol; CH_4O; [67-56-1]	2	2.58	$LaCl_3.CH_3OH$
ethanol; C_2H_6O; [64-17-5]	1	2.05	$LaCl_3.3C_2H_5OH$
	3	2.57	
1-propanol; C_3H_8O; [71-23-8]	1	2.10	$LaCl_3.3C_3H_7OH$
	3	3.29	

For those systems where two values of n and K are reported, the overal solubility of $LaCl_3$ is obtained by using the values for n-K in eq. [1] which give the greater solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous $LaCl_3$ at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

Liquid phases were analysed for rare earth metal concentration (method not specified). At least 3 separate experiments were carried out for each system studied. In addition, the solid phases were analysed for several arbitrary points of each series of experiments (method not specified).

SOURCE AND PURITY OF MATERIALS:

Source and purity of $LaCl_3$ not specified. Anhydrous $LaCl_3$ prepared by method described in (2).

C.p. grade organic solvents were purified by "known" methods (3).

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K

REFERENCES:

1. Golub, A.M.; Golovorushkin, V.I. *Zh. Neorg. Khim.* 1968, *13*, 3194.
2. Spedding, F.H.; Doan, A. H. *J. Am. Chem. Soc.* 1952, *74*, 2783.
3. Kolotyrkin, Ya.M. (ed). *Electrochemistry of Metals in Nonaqueous Solutions.* Khimiya Press. Moscow. 1974. p 440.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Methanol; CH_4O; [67-56-1]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M. N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44-52.

Pitteloud, M.N.
These. Faculte des Sciences de l'Universite des Lausanne. 1971.

VARIABLES:

T/K = 273.2, 298.2, 323.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	solubility $LaCl_3$/mol kg^{-1} a	solubility $LaCl_3$/mol kg^{-1} b
0	---	2.23
25	2.45	2.44
50	---	2.98

[a] Initial solid was $LaCl_3.4CH_3OH$. Equilibrated solid phase analysed and found to contain 4.0 moles methanol per mole of salt.

[b] Initial solid was anhydrous $LaCl_3$. Equilibrated solid phase not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Lanthanum determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $LaCl_3.4CH_3OH$ confirmed by ^1H NMR and x-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, *7*, 224 (this is the reference to a paper by J. H. Freeman and M. L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

La_2O_3 of at least 99.9% purity dissolved in HCl to produce the heptahydrate. The adduct $LaCl_3.4CH_3OH$ prepared by dissolving the heptahydrate in a small excess of o-methylformate followed by distillation and crystallization from methanol. The anhydrous salt prepared by dehydration as described in (3).

Methanol was purified and dried by the Vogel method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J. P.; Kalo, A. M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Platt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

King, F. E.

Masters Thesis.

University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

t/°C	g La_2O_3 in 10 cc satd sln experimental	average[a]	density/g cm^{-3} exptl	av[a]	solubility of $LaCl_3$ [a,b] mol dm^{-3}	mol kg^{-1}
0	1.8753	1.8525	1.0487	------	1.1372	1.0844
0	1.8297		------			
10	2.0314	2.0431	1.0774	-----	1.2541	1.1640
10	2.0547					
15	2.1682	2.1350	------	-----	1.3106	------
15	2.1017					
20	2.8246	2.8292	1.1751	1.1749	1.7367	1.4782
20	2.8337		1.1747			
25	2.7874	2.7704	1.1697	1.1693	1.7006	1.4544
25	2.7534		1.1689			
25[c]	2.9355	2.9304	1.1839	1.1837	1.7989	1.5196
25[c]	2.9252		1.1835			
30	2.7338	2.7361	1.1510	1.1510	1.6796	1.4593
30	2.7384		1.1509			
40	2.8743	2.8725	1.1719	1.1719	1.7633	1.5047
40	2.8707		1.1718			
40[c]	3.6374	------	------	------	2.2328	------
50	4.4695	4.4721	1.4100	1.4101	2.7452	1.9468
50	4.4747		1.4102			

a,b Calculated by compilers. b. From average values of mass La_2O_3 and satd sln densities.

c. Repeat analyses after lowering temp: compilers presume that after reaching 50°C, the temp was lowered first to 40°C, then to 25°C. These points probably represent metastable equilibria.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Salt and ethanol placed in 250 cc stoppered bottles and mechanically agitated in a thermostat for at least 24h. Bottles were sealed by placing rubber tubing over the stopper and neck of each bottle. Slns allowed to settle for at least 12 h and duplicate 10 cc aliquots removed with pipet previously rinsed with the sln. Analyses performed by evapn of ethanol, addn of water, and pptn of lanthanum with oxalic acid. The oxalate was filtered and ignited to const mass as the oxide. Densities measured with a pycnometer, but author states loss by evapn resulted in slightly low values. Soly detns using single bottle by (1) starting at 0°C and raising the temp for the next detn, and (2) by cooling the bottle to a lower temp for a second analysis. Salt and ethanol added to the bottle as needed. The results of the second duplicate analysis (i.e. by cooling) resulted in higher soly values (see table). Several samples of the solid were taken for analyses, but *temperature not specified*. These samples were dried in vac over P_2O_5, weighed, converted to the oxalate and ignited to the oxide. Two analyses gave 2.77 and 1.39 molecules of crystallization.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd from spectro-pure La_2O_3 by addn of aq HCl, and evapn to the point of crystn. Crystals dried in atm of dry HCl for 24 h at room temp followed by slow heating in dry HCl until anhydr salt obtained. The salt was stored in a vac desiccator over P_2O_5. The salt was analysed for H_2O by gravimetric analysis (oxalate-oxide method), but results not given: presumably little or no water found. Ethanol obtained from the stock room (i.e. source and purity unknown) was dried with anhydr Na_2SO_4.

ESTIMATED ERROR:

Soly: precision no better than ± 5% (compilers)

Temp: precision ± 1 K.

REFERENCES:

1. Some of the data from King's Thesis were published in graphical form by Hopkins, B. S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M. N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44-52.

Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite des Lausanne. 1971.

VARIABLES:

T/K = 273.2, 298.2, 323.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

	Solubility $LaCl_3$/mol kg^{-1}	
t/°C	a	b
0	---	1.14
25	1.26	1.34
50	---	1.97

a. Initial solid was $LaCl_3.3C_2H_6O$. Equilibrated solid phase analysed and found to contain 3.6 moles ethanol per mole of $LaCl_3$.

b. Initial solid was anhydrous $LaCl_3$. The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Lanthanum determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $LaCl_3 \cdot 3C_2H_6O$ confirmed by ^1H NMR and x-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, 7, 224. (this is the reference to a paper by J. H. Freeman and M. L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

La_2O_3 of at least 99.9% purity dissolved in HCl to produce the heptahydrate. The adduct $LaCl_3 \cdot 3C_2H_6O$ prepared by dissolving the hydrate in a small excess of o-ethylformate followed by distillation and crystallization from ethanol. The anhydrous salt was prepared by dehydration as described in (3).

Ethanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J. P.; Kalo, A. M. *Helv. Chim. Acta* 1964, 47, 14.
2. Platt, R. *Chimia* 1952, 6, 62.
3. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, 24, 387 (see COMMENTS at left).

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, N.N.; Sakharova, Yu.G.; Ezhova, T.A.; Izmailova, A.A.

Zh. Neorg. Khim. 1975, *20*, 1479-83; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 830-2.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $LaCl_3.6H_2O$ in 96.8 % $C_2H_5OH^a$

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g^b	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1c}
20	40.05	40.23	39.82	40.23	40.08	1.134
30	44.30	44.25	44.18	44.21	44.23	1.252
40	48.70	48.73	48.99	48.66	48.69	1.378
50	56.42	56.38	56.59	56.61	56.50	1.599
60	71.25	71.14	71.14	71.36	71.23	2.016

[a] It is not clearly stated whether the mixture is 96.8 mass % ethanol or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

The hexahydrate melted at 93.9 - 94.5°C.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analysed for the metal by titrn with Trilon B, and for Cl by the Volhard method. Found (%) for La: 39.20, 39.39 (calcd 39.33). Found (%) for Cl: 30.10, 30.08 (calcd 30.16). 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.8% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) 1,2-Ethanediol (ethylene glycol); $C_2H_6O_2$; [107-21-1]

ORIGINAL MEASUREMENTS:

Racster, L.V.
Masters Thesis.
University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salamon and T. Mioduski

EXPERIMENTAL VALUES:

	g La_2O_3 in 10 cc of satd sln		density/g cm^{-3}		solubility of $LaCl_3$ [a,b]	
t/°C	experimental	average[a]	exptl	av[a]	mol dm^{-3}	mol kg^{-1}
10	1.9275	1.8843	1.3580	------	1.1567	0.8518
10	1.8410		------			
15	2.1165	2.1058	1.3856	------	1.2926	0.9329
15	2.0950		------			
20	1.1443	1.1466	1.2597	1.2597	0.7038	0.5587
20	1.1488		1.2597			
30	2.7345	2.7578	1.4357	1.4356	1.6929	1.1792
30	2.7810		1.4355			
40	3.1325	3.0523	1.4535	1.4570	1.8736	1.2860
40	2.9720		1.4605			
50	3.0810	3.0188	1.4410	1.4413	1.8531	1.2858
50	2.9565		1.4415			
60	2.5923	2.6082	------	------	1.6010	------
60	2.6240		------			

a. Calculated by compilers.
b. Calculated by compilers from average mass La_2O_3 and average density of satd sln.

The solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

$LaCl_3$ and solvent placed in 250 cc glass stoppered bottle and mechanically agitated for 24 h. Rubber tubing placed over stopper and neck of bottle and end of tubing sealed with a rubber stopper to prevent water from entering the bottle. Slns allowed to settle for 12-18 h, but slight turbidity persisted, particularly at the lowest and highest temps. At 60°C sln turbidity was significant and appeared different leading author to speculate possible reaction between solute and solvent. Results at 50°C and 60°C said to be approximate. Duplicate 10 cc aliquots pipetted from the bottle for each temp. Each aliquot diluted with 25 cc H_2O and the rare earth pptd as the oxalate with oxalic acid. The oxalate was filtered, ignited and weighed as the oxide. Densities of satd slns detd pycnometrically using pycnometer calibrated at each temp. Densities at 10°C and 15°C may be high due to the condensation of atm water on the surface of the pycnometer.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd by addn of HCl to spectro-pure La_2O_3, and evapn of solvent until crystn. Crystals dehydrated by method of Kremers (2). Salt analysed for presence of H_2O gravimetrically by conversion to oxalate and ignition to the oxide. No water of crystn was found. Ethylene glycol (source and purity not specified) was distilled and initial 5% of distillate discarded. The distilled solvent was stored in a flask sealed with paraffin.

ESTIMATED ERROR:

Soly: Precision no better than ± 3%, and accuracy is probably poor (compilers).

Temp: Not specified.

REFERENCES:

1. Some data from Racster's Thesis were published in graphical form by Hopkins, B.S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

2. Kremers, H.C. *J. Am. Chem. Soc.* 1925 *17*, 298.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) 1-Propanol; C_3H_8O; [71-23-8]

ORIGINAL MEASUREMENTS:

West, D.H.
Masters Thesis.
University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g La_2O_3 in 10 cc of saturated sln			solubility of $LaCl_3$[a,b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	1.8057	1.8263	1.8160[c]	1.1148
20	2.0097	2.0251	2.0174	1.2384
30	2.3641	2.3763	2.3702	1.4550
40	2.8013	2.7879	2.7946	1.7115

a. Calculated by compilers.

b. Calculated by compilers from average mass La_2O_3.

c. Author gives 1.8146 g for this average.

The solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 cc of alcohol and excess salt placed in 250 cc glass stoppered bottle, and rubber tubing placed over the stopper and neck of the bottle and a rubber bung fitted into the open end of the tubing to prevent leakage of water into the bottle. The bottle was immersed in a thermostat and mechanically agitated for at least 12 h. The saturated solutions were then permitted to settle for a minimum of 12 h, and duplicate 10 cc aliquots removed with a pipet Water was added to the aliquots and the sln heated and oxalic acid added to precipitate the rare earth oxalate. The precipitate was filtered, washed with distilled water, and ignited and weighed as the oxide.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd by addn of HCl to spectro-pure La_2O_3, and evaporating the sln to a paste which crystallized upon cooling. The hydrate was dried in a stream of dry HCl by slowly increasing the temp. The anhyd salt was stored in cork-stoppered bottles in a desiccator over P_2O_5. Analysis by conversion to the oxalate and ignition to the oxide showed the salt to be anhydr. Commercial alcohol placed over CaO for 1 week and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was negative.

ESTIMATED ERROR:

Soly: precision probably within ± 3 % (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. Some data from West's Thesis were published in graphical form by Hopkins, B.S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) 2-Propanol; C_3H_8O; [67-63-0]

ORIGINAL MEASUREMENTS:

West, D.H.
Masters Thesis.
University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g La_2O_3 in 10 cc of saturated sln			solubility of $LaCl_3$ [a,b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	0.0283	0.0269	0.0276	0.0169
20	0.0335	0.0319	0.0327	0.0201
30	0.0656	0.0642	0.0649	0.0398
40	0.0884	0.0906	0.0895	0.0549

a. Calculated by compilers.

b. Calculated by compilers from average mass La_2O_3.

The solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 cc of alcohol and excess salt placed in 250 cc glass stoppered bottle, and rubber tubing placed over the stopper and neck of the bottle and a rubber bung fitted into the open end of the tubing to prevent leakage of water into the bottle. The bottle was immersed in a thermostat and mechanically agitated for at least 12 h. The saturated solutions were then permitted to settle for a minimum of 12 h, and duplicate 10 cc aliquots removed with a pipet. Water was added to the aliquots and the sln heated and oxalic acid added to precipitate the rare earth oxalate. The precipitate was filtered, washed with distilled water, and ignited and weighed as the oxide.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd by addn of HCl to spectro-pure La_2O_3, and evaporating the sln to a paste which crystallized upon cooling. The hydrate was dried in a stream of dry HCl by slowly increasing the temp. The anhyd salt was stored in cork-stoppered bottles in a desiccator over P_2O_5. Analysis by conversion to the oxalate and ignition to the oxide showed the salt to be anhydr. Commercial alcohol placed over CaO for 1 week and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was negative.

ESTIMATED ERROR:

Soly: precision probably within ± 3 % (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. Some data from West's Thesis were published in graphical form by Hopkins, B.S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, 19, 64.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) 2-Propanol; C_3H_8O; [67-63-0]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M. N.; Jaccard, P.
Helv. Chim. Acta 1972, 55, 44-52.

Pitteloud, M.N.
These. Faculte des Sciences de l'Universite des Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Two results were reported for 25°C.

1. Starting with $LaCl_3.3C_3H_8O$, the solubility was reported to be 0.004 mol kg^{-1}. The equilibrated solid phase was analysed and found to contain a 4.5 - 5.1 moles of iso-propanol per mole of $LaCl_3$.

2. Starting with the anhydrous salt, the solubility was reported to be 0.016 mol kg^{-1}. The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Lanthanum determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $LaCl_3.3C_3H_8O$ confirmed by ^1H NMR and x-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, *7*, 224 (this is the reference to a paper by J. H. Freeman and M. L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

La_2O_3 of at least 99.9% purity dissolved in HCl to produce the hepthahydrate.. The adduct $LaCl_3.3C_3H_8O$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and trans-solvation of the methanol complex with 2-propanol. The anhydrous salt was prepared by dehydration as described in (3).

Iso-propanol (Fluka) was used as received. Purity and absence of water confirmed by NMR.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

REFERENCES:

1. Brunisholz, P.; Quinche, J. P.; Kalo, A. M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Platt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M. D.; Carter, C. P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) 1,2,3-propanetriol (glycerol); $C_3H_8O_3$; [56-81-5]

ORIGINAL MEASUREMENTS:

Dawson, L. R.
Masters Thesis.
University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g La_2O_3 in 25 cc satd sln		density/g cm^{-3}		solubility of $LaCl_3$[a,b]	
t/°C	experimental	average	exptl	ave[c]	mol dm^{-3}	mol kg^{-1}
10	0.2423	0.2495	1.2732	1.2681	0.0613	0.0483
10	0.2566		1.2630			
20	2.6962	2.6918	1.3592	1.3595	0.6609	0.4862
20	2.6873		1.3598			
25	2.0927	2.0891	1.3330	1.3332	0.5130	0.3848
25	2.0855		1.3334[d]			
30	1.3266	1.3277	1.3114	1.3123	0.3260	0.2484
30	1.3287		1.3131			
40	2.3058	2.3049	1.3639	1.3642	0.5659	0.4149
40	2.3039		1.3645			
50	2.4841	2.4812	1.3514	1.3494	0.6092	0.4515
50	2.4782		1.3474			
60	2.5302	2.5213	------	------	0.6191	------
60	2.5123					

a. Calculated by compilers.
b. Based on average mass of La_2O_3.
c. Recalculated by compilers.
d. Value tabulated in Thesis is 1.334, but this is obviously a typographical error.

The solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

About 175 cc glycerol placed in 250 cc glass stoppered bottles and "liberal amounts" of salt added. Mixtures mechanically agitated in thermostat for 24 h, and even after 15 h of standing turbidity was present. All analyses carried out on turbid slns. For analyses, duplicate 25 cc aliquots (from each bottle) were taken and the rare earth pptd as the oxalate. The ppt was filtered, ignited, and weighed as the oxide. Author states the presence of turbidity has small effect on the overall accuracy of the soly detns. Densities of satd slns determined by withdrawing samples from the bottles, placing them into a pycnometer, and weighing "as quickly as possible."

COMMENTS AND/OR ADDITIONAL DATA:

Since there is a sharp rise in soly from 10°C to 20°C followed by a sharp decrease to 30°C at which point the soly begins to rise again, it is evident that the solid phase in equil with the satd slns is changing. Unsuccessful attempts were made to isolate and identify the solid phases.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd by adding HCl to spectro-pure La_2O_3, and evaporating the solvent to the point of crystallization. Dehydration was carried out in a stream of dry HCl first at room temp 24 h, then at 100°C for ~ 12 h, 110°C for ~ 6 h, and 200°C for 3-4 h. HCl prepd from NaCl + H_2SO_4 and passed through H_2SO_4 drying towers. Glycerol (presumably c.p. or A.R. grade: compilers) distilled at reduced pressure and the "first portion" rejected (no other details given).

ESTIMATED ERROR:

Soly: based upon precision in analyses and temp control, overall precision in soly around ± 3%. Error in accuracy due to turbidity is unknown.

Temp: precision ± 0.5K except for the 10°C run where precision was ± 1.5 K.

REFERENCES:

1. Some of the data from Dawson's Thesis were published in graphical form by Hopkins, B. S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) 2-Methoxyethanol (methyl cellosolve); $C_3H_8O_2$; [109-86-4]

ORIGINAL MEASUREMENTS:

McCarty, C.N.

Masters Thesis. University of Illinois. Urbana, IL. 1933.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

	La_2O_3 [a]	$LaCl_3$ [b]	$LaCl_3$ [b]
t/°C	g/25 cc	g/dm^3	mol/dm^3
0	0.5793	34.89	0.1422
10	0.9068	54.61	0.2227
20	1.1026	66.40	0.2707
30	1.2228	73.64	0.3002
40	1.3077	78.75	0.3211
50	1.5886	95.67	0.3901

a. Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $LaCl_3$ from preparations 1 and 2.

b. Recalculated by the compilers using 1977 IUPAC recommended atomic masses (1).

The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in bottles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber bung fitted into the upper end of the tubing. After equilibration, the solutions were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

ESTIMATED ERROR:

Soly: precision probably within 3 % (compilers).

Temp: precision ± 0.2 K (author).

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 week and then distilled. A middle portion (fraction not specified) was retained and stored in a stoppered flask: b.p. 123°C. La salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide (no details) and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystallized salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extraction the chloride was carried out with HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 and by passing the resulting HCl through H_2SO_4 drying towers.

REFERENCES:

1. IUPAC Commission on Atomic Weights, *Pure Appl. Chem.* 1979, *51*, 405.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) 2-Ethoxyethanol (ethyl cellosolve); $C_4H_{10}O_2$; [110-80-5]

ORIGINAL MEASUREMENTS:

McCarty, C.N.

Masters Thesis. University of Illinois. Urbana, IL. 1933.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	La_2O_3 [a] g/25 cc	$LaCl_3$ [b] g/dm^3	$LaCl_3$ [b] mol/dm^3
0	0.2746	16.54	0.0674
10	0.4968	29.92	0.1220
20	0.7331	44.15	0.1800
30	0.9991	60.17	0.2453
40	1.1125	67.00	0.2732
50	1.3391	80.64	0.3288

a. Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $LaCl_3$ from preparations 1 and 2.

b. Recalculated by the compilers using 1977 IUPAC recommended atomic masses (1).

The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in bottles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber bung fitted into the upper end of the tubing. After equilibration, the solutions were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet, two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 week and distilled. A middle portion (fraction not specified) was retained and stored in a stoppered flask; b.p. 134°C. La salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide (no details) and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystallized salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extraction the chloride was carried out with HCl satd ether, and the resulting chloride heated at 60°C first in a stream of dry HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 and by passing the resulting HCl through H_2SO_4 drying towers.

ESTIMATED ERROR:

Soly: precision probably within 3 % (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. IUPAC Commission on Atomic Weights, *Pure Appl. Chem.* 1979, *51*, 405.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum chloride; $LaCl_3$; [10099-58-8] (2) Ethers	Kirmse, E.M.; Dressler, H. Z. *Chem.* 1975, *15*, 239-40.
VARIABLES:	PREPARED BY:
Room temperature: T/K = 293-298	Mark Salomon and Tomasz Mioduski

EXPERIMENTAL VALUES:

solvent			solubility[a,b] mass %	solubility[a,b] mol kg^{-1}
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	0.6	0.02_5
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	0.1	0.004

[a] Molalities calculated by the compilers.

[b] Solid phase ratios La:ether found to be 1: > 2.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at room temperature until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}.

La was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U.
Z. Chem. 1968, *8*, 472-3.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

Mark Salomon and Tomasz Mioduski

EXPERIMENTAL VALUES:

solvent			$LaCl_3$ solubility[a,b]	
			mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O$;	[5137-45-1]	0.4	0.016
1,3-dioxolane;	$C_3H_6O_2$;	[646-06-0]	0.5	0.020
1,4-dioxane;	$C_4H_8O_2$;	[123-81-1]	0.02	0.0008

[a] Molalities calculated by the compilers.
[b] Nature of solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubilities was within experimental error limits.

La determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was preapred by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Methanol; CH_4O; [67-56-1]

(3) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Golub, A.M.; Yankovich, V. N.
Ukr. Khim. Zh. 1977, *43*, 1139-42;

Ukr. J. Chem. (Engl. Transl.) 1977, *43*, 16-20.

VARIABLES:

Concentration of CH_3OH
T/K = 295

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

Initial Concn Methanol mol dm^{-3}	$LaCl_3$ solubility[a] mol dm^{-3}
1.5	0.01320
2.0	0.02972
2.5	0.05482
3.0	0.09300
3.5	0.13804
4.0	0.21380

[a] Solid phase is $LaCl_3.CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous $LaCl_3$ at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

Liquid phases were analysed for rare earth metal concentration (method not specified). At least 3 separate experiments were carried out for each system studied. In addition, the solid phases were analysed for several arbitrary experimental points (method not specified).

SOURCE AND PURITY OF MATERIALS:

Source and purity of $LaCl_3$ not specified. Anhydrous $LaCl_3$ prepared by method described in (2).

C.p. grade organic solvents were purified by "known" methods (3).

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K.

REFERENCES:

1. Golub, A. M.; Golovorushkin, V. I. *Zh. Neorg. Khim.* 1968, *13*, 3194.
2. Spedding, F.H.; Doan, A.H. *J. Am. Chem. Soc.* 1952, *74*, 2783.
3. Kolotyrkin, Ya.M. (ed). *Electrochemistry of Metals in Nonaqueous Solutions.* Khimiya Press. Moscow. 1974. p 440.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum chloride; $LaCl_3$; [10099-58-8] (2) Alcohols; ROH (3) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]	Golub, A.M.; Yankovich, V.N. *Ukr. Khim. Zh.* 1977, *43*, 1139-42; *Ukr. J. Chem. (Engl. Transl.)* 1977, *43*, 16-20.
VARIABLES:	PREPARED BY:
Concentration of ROH T/K - 295	M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Numerical data were given only for the $LaCl_3$ - CH_3OH - $C_4H_8O_2$ system (see the compilation for this system). The remaining data were presented graphically and in the form of the equation

$$K = [LaCl_3.nROH] / [ROH]^n \qquad [1]$$

In this equation $[LaCl_3.nROH]$ is the solubility in units of mol dm^{-3}, [ROH] is the total alcohol concentration in units of mol dm^{-3}, and n is the solvate number in solution (see ref. 1). According to this equation, n is calculated from the slope of a plot of the logarithm of the solubility, log $[LaCl_3.nROH]$, against log [ROH] . Thus the solubility of $LaCl_3$ can be calculated as a function of ROH concentration using the reported values of n and K (see table below). The alcohol concentrations were varied from 1-5 mol dm^{-3}.

alcohol	n	-log K	nature of the solid phase
methanol; CH_4O; [67-56-1]	3	2.36	$LaCl_3.CH_3OH$
ethanol; C_2H_6O; [64-17-5]	3	3.10	$LaCl_3.3C_2H_5OH$
1-propanol; C_3H_8O; [71-23-8]	1 2	2.90 3.45	$LaCl_3.3C_3H_7OH$

For the last system where two values of n and K are reported, the overall solubility of $LaCl_3$ is obtained by using the values for n-K in eq. [1] which give the greater solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous $LaCl_3$ at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses. Liquid phases were analysed for rare earth metal concentration (method not specified). At least 3 separate experiments were carried out for each system studied. In addition, the solid phases were analysed for several arbitrary points of each series of experiments (method not specified).	Source and purity of $LaCl_3$ not specified. Anhydrous $LaCl_3$ prepared by method described in (2). C.p. grade organic solvents were purified by "known" methods (3). ESTIMATED ERROR: Soly: nothing specified. Temp: precision ± 1 K

REFERENCES:

1. Golub, A.M.; Golovorushkin, V. I. *Zh. Neorg. Khim.* 1968, *13*, 3194.
2. Spedding, F.H.; Doan, A.H. *J. Am. Chem. Soc.* 1952, *74*, 2783.
3. Kolotyrkin, Ya.M. (ed). *Electrochemistry of Metals in Nonaqueous Solutions.* Khimiya Press. Moscow. 1974. p 440.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$;
[10099-58-8]

(2) Hexamethylphosphorotriamide;
$C_6H_{18}N_3OP$;
[680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1761-6;
Russ. J. Inrog. Chem. (Engl. Transl.) 1977, *22*, 955-8.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

Starting with anhydrous $LaCl_3$, the solubility at 25 ± 3°C was given as

$$0.106 \pm 0.002 \text{ mol dm}^{-3}$$

Starting with the solvate $LaCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C was given as

$$0.107 \pm 0.002 \text{ mol dm}^{-3}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature (25 ± 3°C) until eqilibrium was reached. Aliquots were withdrawn periodically and analysed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analysed and found to be $LaCl_3.3C_6H_{18}N_3OP$.

The solvate was analysed for metal content by complexometric titrn, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate also carried out by x-ray analysis.

SOURCE AND PURITY OF MATERIALS:

Anhyd $LaCl_3$ prepd similar to that in (1) by subliming NH_4Cl from a mixt of $LaCl_3$ and 6 moles of NH_4Cl in a stream of inert gas at 200-400°C (LaOCl content less than 3%). The solvent was purified as in (2).

$LaCl_3.3C_6H_{18}N_3OP$ prepd by dissolving the hydrate in $C_6H_{18}N_3OP$ and heating to 140-145° C for 5 m. The solvate was pptd by addition of abs ether, washing 7 times with ether, and drying over P_2O_5 in a stream of dry nitrogen. Yield was about 90%.

ESTIMATED ERROR:

Soly: precision ± 0.002 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.
2. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E. I.; Batyaev, I. M.
Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

T/K = 293

Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	La_2O_3 solubility[a] moles La dm^{-3}
0	0	0.012
1:250	0.035	0.14
1:100	0.085	0.26
1:50	0.17	0.30
1:25	0.33	0.27
1:15	0.59	0.22
1:10	0.78	0.21

[a] This is also the solubility of $LaCl_3$ in the $SnCl_4$-$POCl_3$ mixtures because the oxide is quantitatively converted to the chloride according to

$$La_2O_3 + 6POCl_3 = 2LaCl_3 + 3P_2O_3Cl_4$$

Thus the equilibrated solutions should actually be considered to be a four component system containing $SnCl_4$, $LaCl_3$, $P_2O_3Cl_4$ and $POCl_3$ (the compiler assumes $P_2O_3Cl_4$ is soluble).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and La_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20°C to 2 hours.

La was determined by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

La_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum bromide; $LaBr_3$; [13536-79-3] (2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]	Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesass, U. *Z. Chem.* 1968, *8*, 472-3.
VARIABLES:	**PREPARED BY:**
Room temperature: T/K around 298	Mark Salomon and Tomasz Mioduski

EXPERIMENTAL VALUES:

The solubility of $LaBr_3$ in p-dioxane at about 25°C was given as

0.2 mass %

The corresponding molality calculated by the compilers is

0.005_3 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixture was isothermally agitated at 25°C or room temperature. The authors state that the difference found for the solubility was within experimental error limits.

La was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Lanthanum bromide; $LaBr_3$; [13536-79-3]

(2) Alcohols; ROH

(3) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Golub, A.M.; Yankovich, V. N.
Ukr. Khim. Zh. 1977, *43*, 1139-42;

Ukr. J. Chem. (Engl. Transl.)
1977, *43*, 16-20.

VARIABLES:

Concentration of ROH

T/K = 295

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Numerical data were not given, but results were presented graphically and in the form of the equation

$$K = [LaCl_3.nROH] / [ROH]^n \qquad [1]$$

In this equation $[LaBr_3.nROH]$ is the solubility in units of mol dm^{-3}, [ROH] is the total alcohol concentration in units of mol dm^{-3}, and n is the solvate number in solution (see ref. 1). According to this equation, n is calculated from the slope of a plot of the logarithm of the solubility, log $[LaBr_3.nROH]$, against log [ROH]. Thus the solubility of $LaBr_3$ can be calculated as a function of ROH concentration using the reported values of n and K (see table below). The alcohol concentrations were varied from 1-5 mol dm^{-3}.

alcohol	n	-log K	nature of the solid phase
methanol; CH_4O; [67-56-1]	1	0.89	$LaBr_3.2CH_3OH$
	2	1.16	
1-propanol; C_3H_8O; [71-23-8]	1	1.71	$LaBr_3.2C_3H_7OH$
	2	2.16	

In the above systems, two values of n and K are reported, and the overall solubility of $LaBr_3$ is obtained by using the values for n-K in eq. [1] which give the greater solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous $LaBr_3$ at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

Liquid phases were analysed for rare earth metal concentration (method not specified). At least 3 separate experiments were carried out for each system studied. In addition, the solid phases were analysed for several arbitrary points of each series of experiments (method not specified).

SOURCE AND PURITY OF MATERIALS:

Source and purity of $LaBr_3$ not specified. Anhydrous $LaBr_3$ prepared by method described in (2).

C.p. grade organic solvents were purified by "known" methods (3).

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K

REFERENCES:

1. Golub, A.M.; Golovorushkin, V.I. *Zh. Neorg. Khim.* 1968, *13*, 3194.
2. Spedding, F. H.; Doan, A. H. *J. Am. Chem. Soc.* 1952, *74*, 2783.
3. Kolotyrkin, Ya.M. (ed). *Electrochemistry of Metals in Nonaqueous Solutions.* Khimiya Press. Moscow. 1974. p 440.

COMPONENTS:

(1) Lanthanum iodide; LaI_3; [13813-22-4]

(2) N,N-Dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Moeller, T.; Galasyn, V.

J. Inorg. Nucl. Chem. 1960, *12*, 259-65.

VARIABLES:

$T/K = 298.15$

PREPARED BY:

M. Salomon

EXPERIMENTAL VALUES:

The solubility of LaI_3 in $HCON(CH_3)_2$ at 25°C was reported as

$$578.7 \text{ g dm}^{-3}$$

and as

$$0.5240 \text{ mol dm}^{-3}$$

The solid phase is the solvate $LaI_3.8HCON(CH_3)_2$. The melting point (sealed tube method) of this solvate given as 73.0-75.0°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Authors state that solubilities were determined by analysis of aliquots after equilibration at 25 ± 0.025°C, and that techniques were generally similar to those described in (1).

The rare earth content was determined by complexometric titration with EDTA at 60°C. Iodide was determined by the Volhard method, and carbon, hydrogen, and nitrogen by usual microanalytical techniques.

SOURCE AND PURITY OF MATERIALS:

The initial material was the rare earth oxide of 99.9+% purity. Iodides were prepd by two methods. 1. Acetyl iodide method (2) where the hydrated acetate is treated with acetyl iodide in benzene. Acetyl iodide prepd as in (3). 2. The iodide was prepd by metathesis by reaction of the hydrated $LaCl_3$ with KI in DMF followed by addition of benzene and distillation of the benzene-water azeotrope.

For both preparations the solvate $LaI_3.8DMF$ was recrystallized from DMF by addition of ether.

The solvent, DMF, was prepared as in (4,5), and its electrolytic conductance was $\geq 3.7 \times 10^{-7}$ S cm^{-1} at 25°C.

ESTIMATED ERROR:

Soly: precision around ± 0.1% (compiler).

Temp: precision ± 0.025 K (authors).

REFERENCES:

1. Moeller, T.; Cullen, G.W. *J. Inorg. Nucl. Chem.* 1959, *10*, 148.
2. Watt, G.W.; Gentile, P.S.; Helvenston, E.P. *J. Am. Chem. Soc.* 1955, *77*, 2752.
3. Biltz, H.; Biltz, W. *Laboratory Methods of Inorganic Chemistry (2nd Edition)*. John Wiley. N.Y. 1928.
4. Leader, G.R.; Gormley, J.F. *J. Am. Chem. Soc.* 1951, *73*, 5731.
5. Thomas, A.B.; Rochow, E.G. *J. Am. Chem. Soc.* 1957, *79*, 1843.

COMPONENTS: (1) Cerium fluoride; CeF_3; [7758-88-5] (2) Acidic nitrosyl fluoride; NOF.3HF; [14947-17-2]	ORIGINAL MEASUREMENTS: Galkin, N. P.; Shishkov, Yu.D. Khomyakov, V.I. *Radiokhimiya* 1978, *20*, 136-41; *Soviet Radiochem.* (*Engl. Transl.*) 1978, *20*, 109-13.
VARIABLES: Room temperature	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

The solubility of CeF_3 in acidic nitrosyl fluoride at room temperature was reported to be

0.09 mass %

The molality calculated by the compiler is

4.6×10^{-3} mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solute-solvent mixture was placed in a Teflon vessel and mechanically agitated at room temperature for 10 h. The reaction mixture was allowed to settle for 24 h and the supernatant saturated solution was analysed for the Ce content. An aliquot was evaporated to dryness under vacuum at 100-150°C, and the dry residue dissolved and analysed (the method of analysis not specified).

The solid phase is CeF_3 as found by analyses for F,N,HF and Ce.

SOURCE AND PURITY OF MATERIALS:

CeF_3 was at least 99 % pure.

NOF.3HF prepared by saturation of liquid HF with NOF, and was distilled twice at 95°C before use. The melting point of acidic nitrosyl fluoride was 3.8°C

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Cerium chloride; $CeCl_3$; [7790-86-5]

(2) Hexachloro-1,3-butadiene; C_4Cl_6; [87-68-3]

ORIGINAL MEASUREMENTS:

Shevtsova, Z.N.; Korshunov, B.G.; Safonov, V.V.; Kogan, L.M.; Gudkova, V.I.

Zh. Neorg. Khim. 1968, *13*, 3096-9; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1968, *13*, 1596-8

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition, densities, viscosities and refractive indices of saturated solutions.

$t/^\circ C$	solubility[a] mass %	solubility[a] mol kg^{-1}	$d/g\ cm^{-3}$	η/P	n_D^{20}	nature of the solid phase
25	0.036	0.00146	1.679	0.0385	1.5563	$CeCl_3.4H_2O$
50	0.043	0.00175	1.645	0.0308	1.5556	"
75	0.062	0.00252	1.616	0.0247	1.5549	$CeCl_3.2H_2O$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Equilibrium attained after 12 d at 25°C, 10 d at 50°C, and 7 d at 75°C.

Initial salt, liquid phases and solid phases analyzed for Ce by the oxalate method or by titration with Trilon B using Xylene Orange indicator, and for chloride by the Volhard method. Presumably water was found by difference. Solid phase compositions confirmed by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$CeCl_3.7H_2O$ prepd by dissolving 99.8% Ce_2O_3 in HCl, evaporating and cooling, recrystallizing, and drying in a desiccator. The oxide contained oxide impurities of other rare earths and Fe (0.01%), Ca (0.01-0.05%), and Cu (0.01%). The product was analysed for metal and halide (mass %): Ce 37.45%, Cl 28.75%, H_2O 33.77%.

Purified solvent (method not specified) had the following properties:

$d_4^{20} = 1.6807\ g\ cm^{-3}$, and $n_D^{20} = 1.5543$.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: accuracy ± 0.1 K (authors).

REFERENCES:

COMPONENTS:

(1) Cerium chloride; $CeCl_3$; [7790-86-5]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water: H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, N.N.; Sakharova, Yu.G.; Ezhova, T.A.; Izmailova, A.A.

Zh. Neorg. Khim. 1975, *20*, 1479-83;*Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1975, *20*, 830-2.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $CeCl_3.6H_2O$ in 96.8 % $C_2H_5OH^a$

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g^b	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1c}
20	43.97	44.10	44.36	44.15	44.15	1.245
30	48.91	48.89	48.90	48.92	48.9^d	1.379
40	55.20	54.98	54.87	54.93	54.99	1.551
50	68.41	68.55	68.66	68.38	68.50	1.932
60	84.53	84.61	84.47	84.51	84.53	2.384

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

[d] Compilers calculated 48.91 g/100 g solvent. The molality for this solution was calculated from this value of the solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content of each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

The hexahydrate melted at 95.5 - 96.5°C.

SOURCE AND PURITY OF MATERIALS:

$CeCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analysed for the metal by titrn with Trilon B, and for Cl by the Volhard method. Found (%) for Ce: 39.20, 39.34 (calcd 39.51). Found (%) for Cl: 30.10, 29.95 (calcd 30.04). 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.8% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS: (1) Cerium chloride; $CeCl_3$; [7790-86-5] (2) 2-Methoxyethanol; $C_3H_8O_2$; [109-86-4]	ORIGINAL MEASUREMENTS: Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: T/K = 298	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $CeCl_3$ in 2-methoxyethanol at 25°C was reported to be

10.7 mass %

The corresponding molality calculated by the compiler is

0.486 mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing specified except that the solid phase was found to be $CeCl_3.nC_3H_8O_2$ where n = 2-3.

On the basis of previous papers by the author compiled elsewhere in this volume, it is assumed that the solutions were prepared isothermally and equilibrated for several days, and that Ce determined by complexometric titration.

SOURCE AND PURITY OF MATERIALS:

Nothing specified. Presumably, the anhydrous chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Cerium chloride; $CeCl_3$; [7790-86-5]

(2) 1,1'-Oxybis-ethane (diethyl ether); $C_4H_{10}O$; [60-29-7]

ORIGINAL MEASUREMENTS:

Dzhuraev, Kh. Sh.; Mirsaidov, U.; Kurbanbekov, A.; Rakhimova, A.

Dokl. Akad. Nauk Tadzh. SSR 1976, *19*, 32-4.

VARIABLES:

T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $CeCl_3$ in diethyl ether at 20°C was reported to be

$$7.3 \times 10^{-3} \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$2.96 \times 10^{-4} \text{ mol kg}^{-1}.$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrium was attained within 24 h and was verified by constancy in the Ce concentration. Both the saturated solution and the equilibrated solid phase were analysed. Ce determined by complexometric titration using methylthymol blue indicator and urotropine buffer. Cl determined by titration with $AgNO_3$. The solid phase corresponded to $CeCl_3 \cdot 0.1C_4H_{10}O$ (the solvate was dried under vacuum at 40°C prior to analysis). The pyrolysis product obtained by heating to 500°C is CeOCl.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $CeCl_3$ prepared by the ethanol solvate method (no details given). Diethyl ether was dried with Na and distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cerium chloride; $CeCl_3$; [7790-86-5] (2) Alkyl ethers	Kirmse, E.M.; Dressler, H. *Z. Chem.* 1975, *15*, 239-40.

VARIABLES:	PREPARED BY:
Room temperature T/K = 293-298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$CeCl_3$ solubility[a] mass %	mol kg^{-1}
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	0.1	4×10^{-3} [b]
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	0.1	4×10^{-3} [c]
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	0.12	4.9×10^{-3} [c]

[a] Molalities calculated by the compilers.

[b] Solid phase dried in a vacuum desiccator over P_2O_5 and analysed. The $C_8H_{18}O:CeCl_3$ ratio was less than 2

[c] Solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at room temperature. Method of ascertaining equilibrium not specified.

The anhydrous reagents were handled in a dry box containing P_2O_5.

Cerium was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Cerium chloride; $CeCl_3$; [7790-86-5]

(2) 1,3-Dioxolane; $C_3H_6O_2$; [646-06-0]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Niedergesaess, U.

Z. Chem. 1968, *8*, 472-3;

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $CeCl_3$ in dioxolane at 25°C was reported to be

1.0 mass %.

The corresponding molality calculated by the compiler is

0.041 mol kg^{-1}.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature: authors state that the difference found for the solubility was within experimental error limits.

Ce was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous chloride was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Cerium chloride; $CeCl_3$; [7790-86-5]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.; Auer-Welsbach, C.

Monatsh. Chem. 1965, *96*, 602-5.

VARIABLES:

Room Temperature: T/K about 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $CeCl_3$ in tetrahydrofuran at 20°C (room temperature) was reported to be

0.593 g per 100 ml of solution

(0.024 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Cerium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $CeCl_3.1.46C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $CeCl_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Cl followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cerium chloride; $CeCl_3$; [7790-86-5] (2) Pyridine; C_5H_5N; [110-86-1]	Mueller, R. *Z. Anorg. Allg. Chem.* 1925, *142*, 130-2.
VARIABLES:	PREPARED BY:
T/K = 273	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $CeCl_3$ in pyridine at 0°C was reported to be

1.58 g of anhydrous salt per 100 ml of solution

(0.0641 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Isothermal method employed. The solute-solvent mixture was thermostated at 0°C for 48 h. No other information given.	Nothing specified.
	ESTIMATED ERROR:
	Nothing specified.
	REFERENCES:

COMPONENTS:

(1) Cerium chloride; $CeCl_3$; [7790-86-5]

(2) Hexamethylphosphorotriamide;
$C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.;
Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1761-6;
Russ, J. Inorg. Chem. (Engl. Transl.)
1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of the anhydrous salt at 25 ± 3°C was given as

$$0.109 \pm 0.003 \text{ mol dm}^{-3}$$

Starting with the solvate $CeCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.107 \pm 0.003 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at rooom temperature (25 ± 3°C).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature (25 ± 3°C) until equilibrium was reached. Aliquots were withdrawn periodically and analysed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analysed and found to be $CeCl_3.3C_6H_{18}N_3OP$.

The solvate was analysed for metal content by complexometric titrn, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

Anhyd $CeCl_3$ prepd similarly to that in (1) by subliming NH_4Cl from a mixt of $CeCl_3$ and 6 moles of NN_4Cl in a stream of inert gas at 200-400°C (CeOCl content less than 3%). The solvent was purified as in (2).

$CeCl_3.3C_6H_{18}N_3OP$ prepd by dissolving the hydrate in $C_3H_{18}N_3OP$ and heating to 140-150° C for 5 m. The solvate was pptd by addition of abs ether, washing 7 times with ether, and drying over P_2O_5 in a stream of dry nitrogen. Yield was about 90 %.

ESTIMATED ERROR:

Soly: precision ± 0.003 mol dm^{-3} at a 95 % level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.
2. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Cerium chloride; $CeCl_3$; [7790-86-5]

(2) Hydrazine; N_2H_4; [302-01-2]

ORIGINAL MEASUREMENTS:

Welsh, T.W.B.; Broderson, H.J.

J. Am. Chem. Soc. 1915, 37, 816-24.

VARIABLES:

Room temperature (not specified)

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

The solubility of $CeCl_3$ at room temperature was reported to be

0.03 g/cc

The compilers have not attempted to convert this value to mol kg^{-1} units for several reasons. First we do not know the temperature of the measurements and hence cannot obtain a value for the density of hydrazine. Second we do not know whether or not the initial salt was anhydrous or the heptahydrate as the authors did not provide this information. If the heptahydrate was used, then the results are meaningless for the obvious reasons. Third, the authors admit to problems with oxidation of the solvent and some contamination with water. Fourth, the experimental technique is so crude that in addition to the other sources of experimental error, it hardly seems justifiable to estimate the solubility in units of mol kg^{-1}.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The soly was determined in small tubes which were loosely sealed with a cork covered with tin foil. Capillary tubing sealed to the bottom of the tube served for the passage of dry N_2. 1 cc of N_2H_4 was added to the tube and small weighed portions of powdered $CeCl_3$ added : N_2 was allowed to bubble through the sln to insure adequate mixing. $CeCl_3$ was added in this manner until no more solute would dissolve: the solubility was taken as the total weight of the added salt which dissolved up to this point. Weighings were made to a precision in the 10 mg range, and temperature was not controlled. Authors state that slight oxidation of N_2H_4 probably occurred, and that "slight amounts" of moisture probably were introduced into the solution. Gassing was noted upon introduction of the solid into the solvent.

SOURCE AND PURITY OF MATERIALS:

Commercial N_2H_5 was dehydrated and distilled as described in (1). Analysis for N_2H_4 yielded 99.7% purity. $CeCl_3$ was "an ordinary pure chemical of standard manufacture." It is not clearly stated whether or not the salt was dehydrated. Authors state "water of crystallization was removed wherever it was possible to do so without decomposition." Since many salts were studied in this work, the compilers cannot determine absolutely if the $CeCl_3$ starting material was anhydrous.

ESTIMATED ERROR:

Soly: precision no better than 50%, and accuracy may be much poorer (compilers).

Temp: unknown

REFERENCES:

1. Welsh, T.W.B. *J. Am. Chem. Soc.* 1915, 37, 497.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cerium (IV) chloride; $CeCl_4$; [14986-52-8] (2) Tetrachlorostannate; $SnCl_4$; [7646-78-8] (3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]	Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, *45*, 1176-8.
VARIABLES:	PREPARED BY:
T/K = 293	T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	CeO_2 solubility[a] moles Ce dm^{-3}
1:50	0.17	0.007

[a] Since the solubility is reported for CeO_2 in terms of moles of Ce dm^{-3}, this also corresponds to the solubility of $CeCl_4$ in units of mol dm^{-3} assuming that no reduction of CeO_2 and $CeCl_4$ takes place. Presumably all CeO_2 present in the initial mixture reacts with $POCl_3$ to form the chloride $CeCl_4$ (e.g. see the compilation for the $LaCl_3$-$SnCl_4$-$POCl_3$ system by the same authors).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and CeO_2 were placed in sealed ampoules, heated to 120°C for 2 hours to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20°C to 2 hours.

Ce was determined by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

CeO_2 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS: (1) Cerium bromide; $CeBr_3$; [14457-87-5] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	ORIGINAL MEASUREMENTS: Rossmanith, K. *Monatsh. Chem.* 1966, 97, 1357-64.
VARIABLES: Room Temperature: T/K = 294-296	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $CeBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.60 g per 100 ml of solution (0.016 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Cerium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $CeBr_3.4C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $CeBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Cerium bromide; $CeBr_3$; [14457-87-5]

(2) Pyridine; C_5H_5N; [110-86-1]

ORIGINAL MEASUREMENTS:

Müller, R.

Z. Elektrochem. 1932, *38*, 227-32.

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

solubilities from run number 2[a]

t/°C	mass %	mol %	mol kg^{-1}	nature of the solid phase
-5	0.436	0.0909	0.01153	$CeBr_3.3C_5H_5N$
0	0.907	0.149	0.02410	"
4	1.034	0.216	0.02751	$2CeBr_3.3C_5H_5N$
8	1.213	0.255	0.03233	$CeBr_3.2C_5H_5N$
12	2.138	0.4523	0.05752	"
22	2.615	0.5551	0.07070	$CeBr_3.C_5H_5N$
28	1.414	0.312	0.03776	"
35	0.801	0.167	0.02126	$3CeBr_3.2C_5H_5N$
50	0.719	0.1502	0.01907	"
70	0.692	0.1456	0.01835	"

[a] Molalities calculated by compilers, and mol % calculated by the author (see COMMENTS on next page).

continued......

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solvent and excess solid were equilibrated in glass tubes for 48 h with constant agitation. The saturated solutions were separated from the solid phases by rapid filtration using a heated or cooled filter apparatus. The filtrates were collected in weighing bottles, and the solid phases were also placed in weighing bottles after drying by suction. Cerium in both the filtrates and solid phases was determined gravimetrically by conversion to the oxalate and ignition to CeO_2.

Two separate experimental runs were carried out.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of inorganic materials not specified. Anhydrous $CeBr_3$ prepared by dissolving metallic Ce in sulfuric acid followed by reduction of $Ce_2(SO_4)_3$ with H_2S and high temperature reaction with HBr.

Pyridine (Kahlbaum) was carefully dehydrated with fused KOH and fractionated.

ESTIMATED ERROR:

Soly: Average reproducibility about ± 0.5% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cerium bromide; $CeBr_3$; [14457-87-5] (2) Pyridine; C_5H_5N; [110-86-1]	Müller, R. Z. *Elektrochem.* 1932, *38*, 227-32.

EXPERIMENTAL VALUES: continued...

solubilities from run number 1[a]

t/°C	mass %	mol %	mol kg^{-1}	nature of the solid phase
-5	0.437	0.0912	0.01156	$CeBr_3.3C_5H_5N$
-2	0.713	0.1491	0.01891	"
0	0.907	0.1944	0.02410	"
3	1.33	0.281	0.0355	$CeBr_3.3C_5H_5N + 2CeBr_3.3C_5H_5N$
4	1.02	0.214	0.0271	$2CeBr_3.3C_5H_5N$
5	0.768	0.1606	0.02038	$2CeBr_3.3C_5H_5N + CeBr_3.2C_5H_5N$
8	1.218	0.255	0.03246	$CeBr_3.2C_5H_5N$
10	1.63	0.3451	0.0436	"
12	2.15	0.4562	0.0578	"
15	2.91	0.6195	0.0789	"
18	3.53	0.7552	0.0963	$CeBr_3.2C_5H_5N + CeBr_3.C_5H_5N$
22	2.613	0.555	0.07064	$CeBr_3.C_5H_5N$
25	2.12	0.4485	0.0570	"
28	1.403	0.309	0.03746	"
30	0.821	0.1719	0.02179	$CeBr_3.C_5H_5N + 3CeBr_3.2C_5H_5N$
35	0.802	0.1678	0.02129	$3CeBr_3.2C_5H_5N$
40	0.791	0.165	0.02099	"
50	0.753[b]	0.1576	0.01997	"
60	0.721	0.1503	0.01912	"
70	0.691	0.145	0.01832	"
80	0.653	0.136	0.0173	"

[a] Molalities calculated by the compilers.

[b] Source publication gives 0.453 mass % units which is probably a typographical error. The value probably should be 0.753 mass % units which is consistent with the mol % value calculated by the author.

COMMENTS AND/OR ADDITIONAL DATA:

Since the experimental solubilities are those reported in mass % units, the compilers used these data to calculate the molalities. The author's calculations for mol % differ by around ± 0.5 % from those calculated by the compilers using 1977 IUPAC recommended atomic masses. The compilers' calculations for mol % units are not given in any of the above tables.

COMPONENTS:

(1) Cerium iodide; CeI_3; [7790-87-6]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Yastrebova, L.F.; Grigor, T.I.; Kuznetsova, G.P.; Stepin, B.D.

Zh. Neorg. Khim. <u>1981</u>, *26*, 2238-9; *Russ. J. Inorg. Chem. (Engl. Transl.)*, <u>1981</u>, *26*, 1203-4.

VARIABLES:

Composition at 273 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility at 0°C

	$CeI_3.9H_2O$	CeI_3 [a]		
solvent	mass %	mass %	mol kg^{-1}	solid phase
C_2H_5OH [b]	78.57	59.92	2.879	$CeI_3.9H_2O$
H_2O	89.51	68.26	4.129	"

[a] Results for the anhydrous salt calculated by the compilers.

[b] Authors' original results reported in terms of the solubility of nonohydrate in the pure alcohol. Accounting for the waters of hydration, the compilers calculate that at equilibrium, the solvent contains 53.46 mass % alcohol and 46.34 mass % water.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. No information was given on how equilibrium was ascertained. Aliquots of saturated solution were withdrawn and analyzed for the metal complexometrically, for iodide by a potentiometric volumetric argentometric method, and for water by the Karl Fischer method. The alcohol and water contents in the mixtures[c] were found by quantitative gas chromatography. Solid phase compositions were determined by Schreinemakers' method of residues.[c]

[c] These statements indicate that the authors studied the ternary system over a wide range of compositions. However no phase diagram was given, and the only numerical results reported are those given in the data table above. The phase diagram is stated to be similar to that for the $NdI_3 - H_2O - C_4H_9OH$ system (see the compilation for this system).

SOURCE AND PURITY OF MATERIALS:

The nonohydrate, $CeI_3.9H_2O$, was synthesized according to (1,2).

The alcohol was dried and purified by "recommended" methods.

The source and purity of water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Yakimova, Z.P.; Kuznetsova, G.P.; Yastrebova, L.F.; Stepin, B.D. *Zh. Neorg. Khim.* <u>1977</u>, *22*, 251.
2. Belousova, A.P.; Kuznetsova, G.P.; Rukk, N.S.; Stepin, B.D. *Zh. Neorg. Khim.* <u>1979</u>, *24*, 1410.

COMPONENTS:

(1) Cerium iodide; CeI_3; [7790-87-6]

(2) 1-Butanol; $C_4H_{10}O$; [71-36-3]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Yastrebova, L. F.; Grigor, T. I.; Kuznetsova, G. P.; Stepin, B. D.

Zh. Neorg. Khim. <u>1981</u>, *26*, 2238-9; *Russ. J. Inorg. Chem. (Engl. Transl.)*, <u>1981</u>, *26*, 1203-4.

VARIABLES:

Composition at 273 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility at 0°C

	$CeI_3.9H_2O$	CeI_3[a]		
solvent[b]	mass %	mass %	mol kg^{-1}	solid phase
n-C_4H_9OH	55.90	42.63	1.427	$CeI_3.9H_2O$
H_2O	89.51	68.26	4.129	"

[a] Results for the anhydrous salt calculated by the compilers.

[b] Authors' original results reported in terms of the solubility of the nonohydrate in the pure alcohol. Accounting for the waters of hydration, the compilers calculate that at equilibrium the solvent contains 76.87 mass % alcohol and 23.13 mass % water.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. No information was given on how equilibrium was ascertained. Aliquots of saturated solution were withdrawn and analyzed for the metal complexometrically, for iodide by a potentiometric volumetric argentometric method, and for water by the Karl Fischer method. The alcohol and water contents in the mixtures[c] were found by quantitative gas chromatography. Solid phase compositions were determined by Schreinemakers' method of residues.[c]

[c] These statements indicate that the authors studied the ternary system over a wide range of compositions. However no phase diagram was given, and the only numerical results reported are those given in the data table above. The Phase diagram is stated to be similar to that for the NdI_3 - H_2O - C_4H_9OH system (see the compilation for this system).

SOURCE AND PURITY OF MATERIALS:

The nonohydrate, $CeI_3.9H_2O$, was synthesized according to (1,2).

The alcohol was dried and purified by recommended methods.

The source and purity of water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Yakimova, Z.P.; Kuznetsova, G.P.; Yastrebova, L.F.; Stepin, B.D. *Zh. Neorg. Khim.* <u>1977</u>, *22*, 251.
2. Belousova, A.P.; Kuznetsova, G.P.; Rukk, N.S.; Stepin, B.D. *Zh. Neorg. Khim.* <u>1979</u>, *24*, 1410.

COMPONENTS:

(1) Cerium iodide; CeI_3; [7790-87-6]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Kachkimbaeva, S.A.; Chalova, E.P.; Bleshinskii, S.V.

Khim. Kompleks. Soedin. Redk. Soputstvuyushchikh Elem. 1970, 122-6.

VARIABLES:

T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of CeI_3 in tetrahydrofuran at 20°C was reported to be

0.14 g dm^{-3} (2.69 x 10^{-4} mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were equilibrated isothermally by agitation. The phases were separated by decantation, and in some cases by centrifuging. Ce determined by the oxalate method. Iodide determined by titration with an $AgNO_3$ solution (the Volhard method).

SOURCE AND PURITY OF MATERIALS:

CeI_3 prepared by heating "cp" grade I_2 with excess metallic Ce (Ce-E-1) in a sealed ampoule, and the CeI3 collected by sublimation to the cold part of the ampoule. Analyses for Ce and I revealed the presence of CeI_2. The I/Ce ratio was 2.82.

"C.p." grade tetrahydrofuran (GDR), b.p. = 65.6°C was treated with NaOH and Na, and then distilled from metallic sodium.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Praseodymium fluoride; PrF_3; [13709-46-1] (2) Methanol; CH_4O; [67-56-1]	Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen* <u>1978</u>, *2*, 85-90.
VARIABLES:	**PREPARED BY:**
Room temperature	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of PrF_3 in methanol at room temperature was reported to be

0.01 mass %

The corresponding molality calculated by the compiler is

5.1×10^{-4} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and the Pr:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of PrF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Pr(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Pr determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Pr_6O_{11} (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $PrF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* <u>1974</u>, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* <u>1975</u>, *20*, 33.

COMPONENTS:

(1) Praseodymium fluoride; PrF_3; [13709-46-1]

(2) 1-(Chloromethoxy) butane; $C_4H_{11}ClO$; [2351-69-1]

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen. GDR. 1980.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of PrF_3 in chloromethyl n-butyl ether was reported to be

0.03 mass %

and

1.52×10^{-4} mol/100 g of solution.

The corresponding molality calculated by the compiler is

1.52×10^{-3} mol kg^{-1}

In the solid phase the Pr:F:ether ratio was found to be

1:2.90:0.23.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the Pr:F:H_2O ratio was 1:3.00:0.38.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen.* 1978, 2, 85.

COMPONENTS:

(1) Praseodymium fluoride; PrF_3; [13709-46-1]

(2) 1-Methoxydecane ; $C_{11}H_{24}O$; [7289-52-3]

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen. GDR. 1980.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of PrF_3 in methyl n-decyl ether was reported to be

0.02 mass %

and

1.01×10^{-4} mol/100 g sln

The corresponding molality calculated by the compiler is

1.01×10^{-3} mol kg^{-1}.

In the solid phase the Pr:F:ether ratio was found to be

1:2.91:0.10.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the Pr:F:H_2O ratio was 1:3.00:0.38.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen.* 1978, *2*, 85.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Praseodymium fluoride; PrF_3 [13709-46-1] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	Rossmanith, K. *Monatsh. Chem.* 1966, 97, 1357-64.
VARIABLES:	**PREPARED BY:**
Room Temperature: T/K - 294-296	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $PrBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.62 g per 100 ml of solution (0.016 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Praseodymium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $PrBr_3.4C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $PrBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Praseodymium fluoride; PrF_3; [13709-46-1]

(2) Tributyl phosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of PrF_3 in $[CH_3(CH_2)_3]_3P(O)$ at room temperature was given as

0.02 mass %

The corresponding molality calculated by the compiler is

$$1.0 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Pr:F ratio determined to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of PrF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Pr(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Pr determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Pr_6O_{11} (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $PrF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Preseodymium fluoride; PrF_3; [13709-46-1]

(2) Dimethylsulfoxide; C_2H_6OS; [67-68-5]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of PrF_3 in $(CH_3)_2SO$ at room temperature was given as

0.03 mass %

The corresponding molality calculated by the compiler is

1.5×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and the Pr:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of PrF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Pr(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Pr determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Pr_6O_{11} (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $PrF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Praseodymium fluoride; PrF_3; [13709-46-1] (2) Pyridine; C_6H_5N; [110-86-1]	Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES:	**PREPARED BY:**
Room temperature	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of PrF_3 in pyridine at room temperature was reported to be

$$0.07_5 \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$3.8 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{11} and the Pr:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of PrF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Pr(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Pr determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Pr_6O_{11} (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $PrF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Praseodymium fluoride; PrF_3; [13709-46-1] (2) Acidic nitrosyl fluoride; NOF.3HF; [14947-17-2]	Galkin, N.P.; Shishkov, Yu.D. Khomyakov, V.I. *Radiokhimiya* 1978, *20*, 136-41; *Soviet Radiochem.* (*Engl. Transl.*) 1978, *20*, 109-13.
VARIABLES:	**PREPARED BY:**
Room temperature	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of PrF_3 in acidic nitrosyl fluoride at room temperature was reported to be

0.06 mass %

The molality calculated by the compiler is

3.0×10^{-3} mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solute-solvent mixture was placed in a Teflon vessel and mechanically agitated at room temperature for 10 h. The reaction mixture was allowed to settle for 24 h and the supernatant saturated solution was analyzed for the Pr content. An aliquot was evaporated to dryness under vacuum at 100-150°C, and the dry residue dissolved and analyzed (the method of analysis not specified).

Presumably, the solid phase is anhydrous PrF_3 (compiler).

SOURCE AND PURITY OF MATERIALS:

PrF_3 was at least 99% pure.

NOF.3HF prepared by saturation of liquid HF with NOF, and was distilled twice at 95°C before use. The melting point of acidic nitrosyl fluoride was 3.8°C.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) Hexachloro-1,3-butadiene; C_4Cl_6; [87-68-3]

ORIGINAL MEASUREMENTS:

Shevtsova, Z.N.; Korshunov, B.G.; Safonov, V.V.; Kogan, L.M.; Gudkova, V.I.

Zh. Neorg. Khim. 1968, *13*, 3096-9; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1968, *13*, 1596-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition, densities, viscosities and refractive indices of saturated solutions.

	solubility[a]					nature of the
t/°C	mass %	mol kg^{-1}	d/g cm^{-3}	η/p	n_D^{20}	solid phase
25	0.039	0.00158	1.679	0.0386	1.5565	$PrCl_3.4.5H_2O$
50	0.041	0.00166	1.646	0.0309	1.5554	"
75	0.062	0.00251	1.612	0.0249	1.5550	$PrCl_3.2H_2O$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Equilibrium attained after 12 d at 25°C, 10 d at 50°C, and 7 d at 75°C.

Initial salt, liquid phases and solid phases analysed for Pr by the oxalate method or by titration with Trilon B using Xylene Orange indicator, and for chloride by the Volhard method. Presumably water was found by difference. Solid phase compositions confirmed by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$PrCl_3.7H_2O$ prepd by dissolving 99.5% Pr_2O_3 in HCl, evaporating and cooling, recrystallizing, and drying in a desiccator. The oxide contained oxide impurities of other rare earths and Fe (0.01%), Ca ((0.01-0.05%), and Cu (0.01%). The product was analysed for metal and halide (mass %): Pr 37.70%, Cl 28.85%, H_2O 33.45%.

Purified solvent (method not specified) had the following properties:

$d_4^{20} = 1.6807$ g cm^{-3}, and $n_D^{20} = 1.5543$.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: accuracy ± 0.1 K (authors).

REFERENCES:

COMPONENTS: (1) Praseodymium chloride; $PrCl_3$; [10361-79-2] (2) Methanol; CH_4O; [67-56-1]	**ORIGINAL MEASUREMENTS:** Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986-9.
VARIABLES: Temperature	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
t/°C	mass %	mol kg^{-1}
0	45.02	3.312
25	53.69	4.689
50	59.94	6.051

[a] Molalities calculated by the compiler. At 25°C the solid phase is $PrCl_3.3.5CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $PrCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$PrCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: authors claim accuracy to be about 0.05 %.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, 7, 286.

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Grigorovich, Z. I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
t/°C	mass %	mol kg^{-1}
0	37.29	2.405
25	43.76	3.147
50	47.56	3.668

[a] Molalities calculated by the compiler. At 25°C the solid phase is $PrCl_3.2.5C_2H_5OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $PrCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$PrCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: authors claim accuracy to be about 0.05%.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, 7, 286.

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water: H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, N.N.; Sakharova, Yu.G.; Ezhova, T.A.; Izmailova, A.A.

Zh. Neorg. Khim. 1975, *20*, 1479-83; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 830-2.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $PrCl_3.6H_2O$ in 96.8 % $C_2H_6OH^a$

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1c}
20	43.22	43.49	43.26	43.08	43.32	1.219
30	45.01	45.21	44.90	45.00	45.03	1.267
40	48.35	48.46	48.23	48.14	48.29	1.359
50	54.42	54.41	54.20	54.46	54.37	1.530
60	63.02	62.98	63.11	63.06	63.04	1.774

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table were obtained after 3 h of equilibration, and the remaining two data points were obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

The hexahydrate melted at 113.4 - 114°C.

SOURCE AND PURITY OF MATERIALS:

$PrCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analysed for the metal by titrn with Trilon B, and for Cl by the Volhard method. Found (%) for Pr: 39.50, 39.55 (calcd 39.64). Found (%) for Cl; 29.80, 30.10 (calcd 29.97). 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn determined refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.7 % (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Praseodymium chloride; $PrCl_3$; [10361-79-2] (2) 1-Propanol; C_3H_8O; [71-23-8]	Tomasz Mioduski Institute of Nuclear Research Warsaw, Poland and Mark Salomon USA ET & DL Ft. Monmouth, NJ, U.S.A.

CRITICAL EVALUATION:

The solubility of $PrCl_3$ in 1-propanol has been reported by Kirmse (1) for 298.2 K, and by Grigorovich (2) for 273.2 K, 298.2 K, and 323.2 K. The data reported by these two authors for 298.2 K are in serious disagreement with one another. For example Kirmse reported a solubility of 1.24 mol kg^{-1} and a solid phase of $PrCl_3 \cdot C_3H_7OH$ whereas Grigorovich reported a solubility of 1.907 mol kg^{-1} and a solid phase of $PrCl_2 \cdot 2C_3H_7OH$.

The source of this disagreement is unknown, and thus the more accurate data cannot be identified. Thus we are not able to propose any *tentative* solubility data for $PrCl_3$ in 1-propanol at this time.

REFERENCES

1. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200.

2. Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986.

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) Alcohols

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$PrCl_3$ solubility[a] mass %	mol kg^{-1}	nature of the solid phase
1,2-ethanediol;	$C_2H_6O_2$;	[107-21-1]	30.2	1.75	$PrCl_3.3C_2H_6O_2$
1-propanol;	C_3H_8O;	[71-23-8]	23.5	1.24	$PrCl_3.C_3H_8O$
2-propen-1-ol[b];	C_3H_6O;	[107-18-6]	39.5	2.64	$PrCl_3.C_3H_6O$

[a] Molalities calculated by the compilers.

[b] In the original paper the solvent was specified simply as C_3H_5OH. Upon request, the author kindly identified the solvent as allyl alcohol.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Only the nature of the solid phase was reported. Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) 1-Propanol; C_3H_8O; [71-23-8]

ORIGINAL MEASUREMENTS:

Grigorovich, Z. I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
t/°C	mass %	mol kg^{-1}
0	30.08	1.740
25	32.04	1.907
50	33.10	2.001

[a] Molalities calculated by the compiler. At 25°C the solid phase is $PrCl_3.2.0C_3H_7OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $PrCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$PrCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: authors claim accuracy to be about 0.5 %.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, 7, 286.

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) 1-Butanol; $C_4H_{10}O$; [71-36-3]

ORIGINAL MEASUREMENTS:

Grigorovich, Z.I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
$t/^{\circ}C$	mass %	mol kg^{-1}
0	15.42	0.737
25	23.62	1.251
50	31.78	1.884

[a] Molalities calculated by the compiler. At 25°C the solid phase is $2PrCl_3.3C_4H_9OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $PrCl_3$ (no details were given).

The alochol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$PrCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: authors claim accuracy to be about 0.05 %.

Temp: nothing specified.

REFERENCES:

1. Freeman, I. H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) 1-Pentanol; $C_5H_{12}O$; [71-41-0]

ORIGINAL MEASUREMENTS:

Grigorovich, Z.I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
$t/^\circ C$	mass %	mol kg^{-1}
0	17.48	0.857
25	24.35	1.302
50	23.37	1.233

[a] Molalities calculated by the compiler. At 25°C the solid phase is $PrCl_3.3C_5H_{11}OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $PrCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$PrCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: authors claim accuracy to be about 0.5 %.

Temp: nothing specified.

REFERENCES:

1. Freeman, I. H. *J. Inorg. Nucl. Chem.* 1958, 7, 286.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Praseodymium chloride; $PrCl_3$; [10361-79-2] (2) Ethers	Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472-3. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
Room temperature: T/K around 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

			$PrCl_3$ solubility[a,b]	
solvent			mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.04	0.0016
1,3-dioxolane;	$C_3H_6O_2$;	[646-06-0]	0.9	0.037
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.04	0.0016

[a] Molalities calculated by the compilers.

[b] Nature of solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Pr was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS: (1) Praseodymium chloride; $PrCl_3$; [10361-79-2] (2) Alkoxy-ethanols	ORIGINAL MEASUREMENTS: Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: T/K = 298	PREPARED BY: T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$PrCl_3$ solubility[a] mass %	mass kg^{-1}	nature of the solid phase
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	12.8	0.594	$PrCl_3.nC_3H_8O_2$ (n = 2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	16.2	0.782	$PrCl_3.2C_4H_{10}O_2$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the authors the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Kirmse, E. M.; Dressler, H.

Z. Chem. 1975, *15*, 239-40.

VARIABLES:

Room Temperature (293-298 K)

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

			$PrCl_3$ solubility[a]	
solvent			mass %	mol kg^{-1}
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	0.8 [b]	0.033
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	0.07	0.0028
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	0.06	0.0024

[a] Molalities calculated by the compilers.

[b] Solid phase dried in a vacuum desiccator over P_2O_5. Analysis yielded the composition $PrCl_3.2C_8H_{18}O$. Compositions of other solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated (at room temperature) until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}. Pr was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Praseodymium chloride; $PrCl_3$; [10361-79-2] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	Rossmanith, K.; Auer-Welsbach, C. *Monatsch. Chem.* 1965, *96*, 602-5.
VARIABLES:	**PREPARED BY:**
Room Temperature: T/K about 293	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $PrCl_3$ in tetrahydrofuran at 20°C (room temperature) was reported to be

0.590 g per 100 ml of solution

(0.0239 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Praseodymium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $PrCl_3.2.00C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $PrCl_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Cl followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.
Zh. Neorg. Khim. 1975, *20*, 908-14;
Russ. J. Inorg. Chem. Engl. Transl. 1975, *20*, 508-11.

VARIABLES:

One Temperature: 25°C

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solution

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg^{-1} (compiler)	density/g cm^{-3}
30.0	1.21	356.5	1.45	1.73	1.24

The solid phase is $PrCl_3$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and pptd with aq NH_3. The pptd $Pr(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous and only anhydrous $PrCl_3$ was found. All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complextion between TBP and $PrCl_3$ in solution. Additional studies with unsaturated solutions (IR spectra, viscosity, molar conductivities) are discussed in the source paper.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $PrCl_3$ prepared by chlorination of Pr_2O_3 with CCl_4 vapor (1,2). Source and purity of materials not given. Pr was analyzed gravimetrically and Cl by Volhard's method. Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* 1964, *9*, 1427.
2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* 1965, *38*, 1160.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Praseodymium chloride; $PrCl_3$ [10361-79-2] (2) Alkyl amines	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* <u>1971</u>, 200-6.

VARIABLES:	PREPARED BY:
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$PrCl_3$ solubility[a] mass %	mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	22.2	1.15
2-propanamine;	iso-C_3H_9N;	[75-31-0]	0.08	0.0032
2-propen-1-amine[b]	C_3H_7N;	[107-11-9]	0.05	0.0020

[a] Molalities calculated by the compilers.

[b] The original paper simply specifies the solvent as $C_3H_5NH_2$, and upon request the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* <u>1962</u>, *24*, 387.

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1761-6; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of the anhydrous salt at 25 ± 3°C was given as

$$0.113 \pm 0.003 \text{ mol dm}^{-3}$$

Starting with the solvate $PrCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.128 \pm 0.001 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature (25 ± 3°C) until equilibrium was reached. Aliquots were withdrawn periodically and analysed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $PrCl_3.3C_6H_{18}N_3OP$. The solvate was analysed for metal content by complexometric titrn, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

Anhyd $PrCl_3$ prepd similarly to that in (1) by subliming NH_4Cl from a mixt of $PrCl_3$ and 6 moles of NN_4Cl in a stream of inert gas at 200-400°C (PrOCl content less than 3 %). The solvent was purified as in (2).

$PrCl_3.3C_6H_{18}N_3OP$ prepd by dissolving the hydrate in $C_3H_{18}N_3OP$ and heating to 140-145°C for 5 m. The solvate was pptd by addition of abs ether, washing 7 times with ether, and drying over P_2O_5 in a stream of dry nitrogen. Yield was about 90 %.

ESTIMATED ERROR:

Soly: precision ± 0.001 mol dm^{-3} at a 95 % level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.
2. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Praseodymium chloride; $PrCl_3$; [10361-79-2]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

T/K = 293

Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	Pr_2O_3 solubility[a] moles Pr dm^{-3}
0	0	0.013
1:250	0.035	0.10
1:100	0.085	0.20
1:50	0.17	0.31
1:25	0.33	0.27
1:15	0.59	0.12
1:10	0.78	0.11

[a] This is also the solubility of $PrCl_3$ in the $SnCl_4$-$POCl_3$ mixtures because the oxide is quantitatively converted to the chloride according to

$$Pr_2O_3 + 6POCl_3 = 2PrCl_3 + 3P_2O_3Cl_4$$

Thus the equilibrated solutions should actually be considered to be a four component system containing $SnCl_4$, $PrCl_3$, $P_2O_3Cl_4$ and $POCl_3$ (the compiler assumes $P_2O_3Cl_4$ is soluble).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Pr_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20° C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20° to 2 hours.

Pr was determined by colorimetric analysis, and in some cases by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Pr_6O_{11} of "the first sort" was reduced to Pr_2O_3 with hydrogen at 950°C.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS:

(1) Praseodymium bromide; $PrBr_3$; [13536-53-3]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

The solubility of $PrBr_3$ in 1,2-diethoxyethane at 25°C was given as

0.6 mass %

The corresponding value of the molality calculated by the compiler is

$0.016 \text{ mol kg}^{-1}$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Praseodymium bromide; $PrBr_3$; [13536-53-3]

(2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U.
Z. Chem. 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $PrBr_3$ in p-dioxane at around 25°C was given as

$$0.3_5 \text{ mass \%}$$

The corresponding molality calculated by the compiler is

$$9.2 \times 10^{-3} \text{ mol kg}^{-1}$$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Pr was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Praseodymium bromide; $PrBr_3$; [13536-53-3]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$PrBr_3$ solubility[a] mass %	mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	21.6	0.724
2-propanamine;	iso-C_3H_9N;	[75-31-0]	9.6	0.279
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	9.2	0.266
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	26.5	0.947
di-2-butylamine;	(sec-$C_4H_9)_2NH$;	[626-23-3]	0.04	0.0011

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Praseodymium iodide; PrI_3; [13813-23-5]

(2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. *Z. Chem.* 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of PrI_3 in p-dioxane at about 25°C was given as

2.7 mass %

The corresponding molality calculated by the compiler is

0.053 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Pr was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS: (1) Praseodymium iodide; PrI_3; [13813-23-5] (2) Alkyl amines	**ORIGINAL MEASUREMENTS:** Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: T/K = 298	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			PrI_3 solubility^a mass %	mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	15.5	0.352
2-propanamine;	iso-C_3H_9N;	[75-31-0]	5.3	0.107
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	19.6	0.467
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	2.5	0.049

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Praseodymium iodide ; PrI_3; [13813-23-5]

(2) N,N-Dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Moeller, T.; Galasyn, V.

J. Inorg. Nucl. Chem. 1962, *12*, 259-65.

VARIABLES:

T/K = 298.15

PREPARED BY:

M. Salomon

EXPERIMENTAL VALUES:

The solubility of PrI_3 in $HCON(CH_3)_2$ at 25°C was reported as

$$735.8 \text{ g dm}^{-3}$$

and as

$$0.6650 \text{ mol dm}^{-3}$$

The solid phase is the solvate $PrI_3.8HCON(CH_3)_2$. The melting point (sealed tube method) of this solvate given as 90.5 - 92.5°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Authors state that solubilities were determined by analysis of aliquots after equilibration at 25 ± 0.025°C, and that techniques were generally similar to those described in (1).

The rare earth content was determined by complexometric titration with EDTA at 60°C. Iodide was determined by the Volhard method, and carbon, hydrogen, and nitrogen by usual microanalytical techniques.

SOURCE AND PURITY OF MATERIALS:

The initial material was the rare earth oxide of 99.9+% purity. Iodides were prepd by two methods. 1. Acetyl iodide method (2) where the hydrated acetate is treated with acetyl iodide in benzene. Acetyl iodide prepd as in (3). 2. The iodide was prepd by metathesis by reaction of the hydrated $PrCl_3$ with KI in DMF followed by addition of benzene and distillation of the benzene-water azeotrope.

For both preparations the solvate $PrI_3.8DMF$ was recrystallized from DMF by addition of ether.

The solvent, DMF, was prepared as in (4,5), and its electrolytic conductance was $\geq 3.7 \times 10^{-7}$ S cm^{-1} at 25°C.

ESTIMATED ERROR:

Soly: precision around ± 0.1% (compiler).

Temp: precision ± 0.025 K (authors).

REFERENCES:

1. Moeller, T.; Cullen, G.W. *J. Inorg. Nucl. Chem.* 1959, *10*, 148.
2. Watt, G.W.; Gentile, P.S.; Helvenston, E. P. *J. Am. Chem. Soc.* 1955, *77*, 2752.
3. Biltz, H.; Biltz, W. *Laboratory Methods of Inorganic Chemistry (2nd Edition)*. John Wiley. N.Y. 1928.
4. Leader, G.R.; Gormley, J.F. *J. Am. Chem. Soc.* 1951, *73*, 5731.
5. Thomas, A.B.; Rochow, E.G. *J. Am. Chem. Soc.* 1957, *79*, 1843.

COMPONENTS: (1) Neodymium fluoride; NdF_3; [13709-42-7] (2) Alcohols	**ORIGINAL MEASUREMENTS:** Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES: Room temperature	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			NdF_3 solubility[a,b] mass %	mol kg^{-1}
methanol ;	CH_4O;	[67-56-1]	0.02	1×10^{-3}
ethanol ;	C_2H_6O;	[64-17-5]	0.02	1×10^{-3}

[a] Molalities calculated by the compilers.

[b] Solid phases were dried in a desiccator over P_4O_{10} and the Nd:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of NdF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging.

The sln was heated with about 10 cm^3 of 10% KOH solution for 3-5 h to obtain solid $Nd(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Nd determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined as described in (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $NdF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvents were dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Neodymium fluoride; NdF_3; [13709-42-7]

(2) Ethers

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen, GDR. 1980.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility mass %	solubility mol/100 g sln
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	0.01[a]	5.0×10^{-5}
1-(chloromethoxy)butane;	$C_5H_{11}ClO$;	[2351-69-1]	0.02[b]	9.9×10^{-5}

[a] Solid phase. Nd:F:ether:H_2O ratio found to be 1:3.03:0.06:0.24.

[b] Solid phase. Nd:F:ether:H_2O ratio found to be 1:2.89:0.51:0.25

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the Nd:F:H_2O ratio was 1:3.01:0.45.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen.* 1978, *2*, 85.

COMPONENTS:

(1) Neodymium fluoride; NdF_3;
[13709-42-7]

(2) Tributyl phosphate; $C_{12}H_{27}O_4P$;
[126-73-8]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of NdF_3 in $[CH_3(CH_2)_3]_3P(O)$ at room temperature was given as

0.04 mass %

The corresponding molality calculated by the compiler is

2.0×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and the Nd:F ratio determined to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of NdF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging.

The sln was heated with about 10 cm^3 of 10% KOH solution for 3-5 h to obtain solid $Nd(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Nd determined several times by complexometric titration with potentiometric endpoint detection (1). The fluoride content in the filtrate was determined as described in (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $NdF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Neodymium fluoride; NdF_3; [13709-42-7]

(2) Dimethylsulfoxide; C_2H_6OS; [67-68-5]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, 2, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of NdF_3 in $(CH_3)_2SO$ at room temperature was given as

0.02 mass %

The corresponding molality calculated by the compiler is

$$1.0 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Nd:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg NdF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-2 hours to obtain quantitative separation of solid $Nd(OH)_3$ and a basic F^- solution. The $Nd(OH)_3$ was filtered, washed and dissolved with HCl. Nd determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

The reported solubility is a mean of "numerous parallel determinations," or at least two parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $NdF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Neodymium fluoride; NdF_3; [13709-42-7]

(2) Pyridine; C_6H_5N; [110-86-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of NdF_3 in pyridine at room temperature was reported to be

0.07 mass %

The corresponding molality calculated by the compiler is

$$3.5 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Nd:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg NdF_3 and 10-20 cm^3 of solvent mechanically agitated at room temperature for 100 hours. Samples of saturated solution for analyses were obtained by decantation or by centrifuging. 5-10 g of saturated solution were heated with about 10 cm^3 of 10 % KOH solution for 1-2 hours to obtain quantitative separation of solid $Nd(OH)_3$ and a basic F^- solution. The $Nd(OH)_3$ was filtered, washed and dissolved with HCl. Nd determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content of the basic filtrate was determined photometrically using Al-Eriochrome cyanine color lake (2).

The reported solubility is a mean of "numerous parallel determinations," or at least two parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $NdF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Hexachloro-1,3-butadiene; C_4Cl_6; [87-68-3]

ORIGINAL MEASUREMENTS:

Shevtsova, Z.N.; Korshunov, B.G.; Safonov, V.V.; Kogan, L.M.; Gudkova, V.I.

Zh. Neorg. Khim. 1968, *13*, 3096-9; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1968, *13*, 1596-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition, densities, viscosities and refractive indices of saturated solutions.

	solubility[a]					nature of the
t/°C	mass %	mol kg^{-1}	d/g cm^{-3}	η/p	n_D^{20}	solid phase
25	0.029	0.00116	1.679	0.0389	1.5553	$NdCl_3.3.5H_2O$
50	0.037	0.00148	1.648	0.0311	1.5553	"
75	0.055	0.00220	1.612	0.0249	1.5550	$NdCl_3.3H_2O$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Equilibrium attained after 12 d at 25°C, 10 d at 50°C, and 7 d at 75°C.

Initial salt, liquid phases and solid phases analysed for Nd by the oxalate method or by titration with Trilon B using Xylene Orange indicator, and for chloride by the Volhard method. Presumably water was found by difference. Solid phase compositions confirmed by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3.6H_2O$ prepd by dissolving 99.8% Nd_2O_3 in HCl, evaporating and cooling, recrystallizing, and drying in a desiccator. The oxide contained oxide impurities of other rare earths and Fe (0.01%), Ca((0.01-0.05%), and Cu (0.01%). The product was analysed for metal and halide (mass %): Nd 40.25%, Cl 26.30%, H_2O 33.45%.

Purified solvent (method not specified) had the following properties:

d_4^{20} = 1.6807 g cm^{-3}, and n_D^{20} = 1.5543.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: accuracy ± 0.1 K (authors).

REFERENCES:

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Methanol; CH_4O; [67-56-1]

EVALUATOR:
Mark Salomon
USA ET & DL
Ft. Monmouth, NJ, U.S.A.
and
Tomasz Mioduski
Institute of Nuclear Research
Warsaw, Poland

CRITICAL EVALUATION:

The solubility of $NdCl_3$ in methanol has been reported in three publications (1-3). West (1) studied the solubility from 283-313 K and reported his results in volume units. The results in (2) for 273-323 K and in (3) for 298.15 K are both in mass units, but there is serious disagreement between these results. For example at 298.2 K Grigorovich (2) reports a solubility of 4.466 mol kg^{-1} (solid phase is $NdCl_3 \cdot 3CH_3OH$) while Merbach et al. (3) report a solubility of 2.75 mol kg^{-1} (solid phase is $NdCl_3 \cdot 4CH_3OH$).

While we cannot directly compare West's solubility result of ~ 2.25 mol dm^{-3} at 298.2 K with those in (2,3), it certainly is more consistent with the result of Merbach et al. This does not necessarily imply that there is some systematic error in the results of Grigorovich, but rather that a metastable phase is involved.

Merbach et al. report two values for the solubility of $NdCl_3$ in methanol at 298.15 K. The first value of 2.75 mol kg^{-1} was obtained when the solution was equilibrated with the tetrasolvate. Starting with the anhydrous salt, Merbach et al. found a solubility of 2.84 mol kg^{-1}, and the difference in these two results (3 %) is much greater than the experimental precision of 0.5 % estimated by the compilers. It would thus appear that the higher solubility result of Merbach et al. represents a solution in "quasi" equilibrium with both the stable $NdCl_3 \cdot 4CH_3OH$ and metastable $NdCl_3 \cdot 3CH_3OH$ solid phases.

At this time we designate both results of Grigorovich (2) and Merbach et al. (3) as *tentative* solubility data. For 298.15 K the tentative solubility in the stable tetrasolvate system is 2.75 mol kg^{-1} (3), and at 273.2 K, 298.2 K and 323.2 K the tentative solubilities for the metastable trisolvate system are 3.349 mol kg^{-1}, 4.466 mol kg^{-1} and 5.133 mol kg^{-1}, respectively (2).

REFERENCES

1. West, D.H. *Masters Thesis*. University of Illinois. Urbana, IL. 1932. Some of West's data have also been published in reference 4 below.

2. Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986.

3. (a) Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44.
(b) Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.

4. Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Methanol; CH_4O; [67-56-1]

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283 - 313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of saturated sln			solubility of $NdCl_3$[a,b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	3.5696	3.5737	3.5717	2.1230
20	3.7587	3.7268	3.7428	2.2247
30	3.838	3.8162	3.8271[c]	2.2748
40	3.9727	3.9955	3.9841	2.3681

[a] Calculated by compilers.

[b] Calculated by compilers based on average mass of Nd_2O_3.

[c] Author gives average value of 3.8221 g/10 cc.

The solid phase was not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 cc of alcohol and excess salt placed in 250 cc glass stoppered bottle, and rubber tubing placed over the stopper and neck of the bottle and a rubber stopper fitted into the open end of the tubing to prevent leakage of water into the bottle. The bottle was immersed in a thermostat and mechanically agitated for at least 12 h. The saturated solutions were then permitted to settle for a minimum of 12 h, and duplicate 10 cc aliquots removed with a pipet. Water was added to the aliquots and the sln heated and oxalic acid added to precipitate the rare earth oxalate. The precipitate was filtered, washed with distilled water, and ignited to the oxide and weighed.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_2O_3, and evaporating the sln to a paste which crystallized upon cooling. The hydrate was dried in a stream of dry HCl by slowly increasing the temp. The anhyd salt was stored in cork-stoppered bottles in a desiccator over P_2O_5. Analysis by conversion to the oxalate and ignition to the oxide showed the salt to be anhydr. Commercial alcohol placed over CaO for 1 week and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was negative.

ESTIMATED ERROR:

Soly: precision probably within ± 3% (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. Some data from West's Thesis was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Neodymium chloride; $NdCl_3$; [10024-93-8] (2) Methanol; CH_4O; [67-56-1]	Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986-9.
VARIABLES:	**PREPARED BY:**
Temperature	T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
t/°C	mass %	mol kg^{-1}
0	45.63	3.349
25	52.81	4.466
50	56.26	5.133

[a] Molalities calculated by the compiler. At 25°C the solid phase is $NdCl_3.3CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05 %.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Methanol; CH_4O; [67-56-1]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P.
Helv. Chim. Acta 1972, 55, 44-52.

Pitteloud, M.N.
These. Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

	mean solubilities/mol kg^{-1}	
t/°C	a	b
25	2.75	2.84

a. Initial salt is the adduct $NdCl_3.4CH_3OH$. Equilibrated solid phase analyzed and found to be $NdCl_3.4CH_3OH$.

b. Solutions equilibrated with anhydrous $NdCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $NdCl_3.4CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Neodymium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $NdCl_3.4CH_3OH$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, 7, 224 (this is the reference to a paper by J.H. Freeman and M.L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as described in (3). The adduct $NdCl_3.4CH_3OH$ prepared by dissolving the hydrate in a small excess of o-methlyformate followed by distillation and crystallization from methanol.

Methanol was purified and dried by the Vogel method.

ESTIMATED ERROR:

Soly: precision ±0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:	EVALUATOR:
(1) Neodymium chloride; $NdCl_3$; [10024-93-8] (2) Ethanol; C_2H_6O; [64-17-5]	Mark Salomon USA ET & DL Ft. Monmouth, NJ, U.S.A. and T. Mioduski Institute of Nuclear Research Warsaw, Poland

CRITICAL EVALUATION:

The solubility of $NdCl_3$ in ethanol has been reported in three publications (1-3). King (1) studied the solubility from 273-323 K and reported his results in volume units. The results in (2) for 273-323 K and in (3) for for 298.15 K are both in mass units, but there is serious disagreement between these results. For example at 298.2 K Grigorovich (2) reports a solubility of 3.080 mol kg^{-1} (solid phase is $NdCl_3 \cdot 2C_2H_5OH$) while Merbach et al. (3) report a solubility of 1.35 mol kg^{-1} (solid phase is $NdCl_3 \cdot 3C_2H_5OH$).

While we cannot directly compare King's solubility result of 1.2858 mol dm^{-3} at 298 K with those in (2,3), it certainly is more consistent with the result of Merbach et al. This does not necessarily imply that there is some systematic error in the results of Grigorovich, but rather that a metastable phase is involved.

Merbach et al. report two values for the solubility of $NdCl_3$ in ethanol at 298.15 K. The first value of 1.35 mol kg^{-1} was obtained when the solution was equilibrated with the trisolvate. Starting with the anhydrous salt, Merbach et al. found a solubility of 1.52 mol kg^{-1}, and the difference in these two results (11 %) is much greater than the experimental precision of 0.5 % estimated by the compilers. It would thus appear that the higher solubility result of Merbach et al. represents a solution in "quasi" equilibrium with both the stable $NdCl_3 \cdot 3C_2H_5OH$ and $NdCl_3 \cdot 2C_2H_5OH$ solid phases.

At this time we designate both results of Grigorovich (2) and Merbach et al. (3) as *tentative* solubility data. For 298.15 K the tentative solubility in the stable trisolvate system is 1.35 mol kg^{-1}, and at 273.2 K, 298.2 K and 323.2 K, the tentative solubilities for the metastable disolvate system are 2.420 mol kg^{-1}, 3.080 mol kg^{-1} and 2.333 mol kg^{-1}, respectively. At this time we cannot explain the decrease in the solubility at 323.K in the metastable system: either an error exists or the system has reverted to the stable trisolvate system.

REFERENCES

1. King, F.E. *Masters Thesis*. University of Illinois. Urbana, IL. 1932. Some of King's data have also been published in reference 4 below.

2. Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986.

3. (a) Merbach, A.; Pitteloud, M.N.: Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44.
   (b) Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.

4. Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

King, F.E.

Masters Thesis. University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 273 - 323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc satd sln		density/g cm^{-3}		solubility of $NdCl_3$ [a,b]	
t/°C	experimental	average[a]	exptl	av[a]	mol dm^{-3}	mol kg^{-1}
0	1.5598	1.5673	1.0102	1.0105	0.9316	0.9219
0	1.5748		1.0108			
10	1.6053	1.6122	1.0370	1.0374	0.9582	0.9237
10	1.6190		1.0378			
15	1.6338	1.6330	1.0502	1.0505	0.9706	0.9240
15	1.6322		1.0507			
20	2.0129	2.0082	1.0613	1.0614	1.1937	1.1246
20	2.0035		1.0615			
25	2.4274	2.4194	1.1183	1.1184	1.4380	1.2858
25	2.4113		1.1185			
30	1.9223	1.9242	1.0524	1.0524	1.1437	1.0868
30	1.9260		1.0524			
40	2.2178	2.2256	1.0832	1.0832	1.3228	1.2213
40	2.2333		1.0831			
40[c]	3.0182	3.0173	1.207	-----	1.7935	1.486
40[c]	3.0164		-----			
50	2.9742	2.9705	1.1740	1.1745	1.7656	1.5033
50	2.9667		1.1750			

[a] Calculated by compilers.

[b] Calculated by compilers using average values for mass Nd_2O_3 and density of satd slns.

[c] These points detd after two points at 50°C detd: i.e. after cooling from 50°C to 40°C. These data points probably represent metastable equilibria.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Salt and alcohol placed in 250 cc stoppered bottle and mechanically agitated in a thermostat for at least 24 h. Bottle sealed by placing rubber tubing over stopper and neck of bottle. Slns allowed to settle for at least 12 h and duplicate 10 cc aliquots removed with pipet previously rinsed with the sln. Analyses performed by evapn of alcohol, addn of water, and pptn of the rare earth with oxalic acid. The oxalate was filtered and ignited to const weight as the oxide. Densities measured with a pycnometer, but author states loss by evapn resulted in slightly low values. Soly detns using single bottle by (1) starting at 0°C and raising the temp for the next detn, and (2) by cooling the bottle to a lower temp for a second analysis. Salt and solvent added to the bottle as needed. The results of the second duplicate analysis (i.e. by cooling) resulted in higher soly values (see table). Several samples of the solid were taken for analyses, but *temperature not given*. These samples were dried in vac over P_2O_5, weighed, converted to the oxalate and ignited to the oxide. Two analyses gave 2.59 and 1.65 molecules of crystallization.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd from spectro-pure Nd_2O_3 by treatment with aq HCl, and evapn to the point of crystn. Crystals dried in atm of dry HCl for 24 h followed by slow heating in dry HCl until the anhydr salt was obtained. The salt was stored in a vac desiccator over P_2O_5. The salt was analyzed for presence of H_2O by gravimetric analysis (oxlalate-oxide method), but results not given: presumably little or no water was found. Ethanol obtained from the stock room (i.e. source and purity unknown) dried with anhydr Na_2SO_4.

ESTIMATED ERROR:

Soly: precision no better than ± 5% (compilers).

Temp: precision ± 1 K.

REFERENCES:

1. Some of the data from King's Thesis was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS: (1) Neodymium chloride; $NdCl_3$; [10024-93-0] (2) Ethanol; C_2H_6O; [64-17-5]	**ORIGINAL MEASUREMENTS:** Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986-9.
VARIABLES: Temperature	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

t/°C	solubility[a] mass %	solubility[a] mol kg^{-1}
0	37.75	2.420
25	43.56	3.080
50	36.89	2.333

[a] Molalities calculated by the compiler. At 25°C the solid phase is $NdCl_3.2C_2H_5OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05%.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44-52.

Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$t/^{\circ}C$	mean solubilities/mol kg^{-1}	
	a	b
25	1.35	1.52

a. Initial salt is the adduct $NdCl_3.3C_2H_5OH$. Equilibrated solid phase analyzed and found to be $NdCl_3.3C_2H_5OH$.

b. Solutions equilibrated with anhydrous $NdCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $NdCl_3.3C_2H_5OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Neodymium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $NdCl_3.3C_2H_5O$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, *7*, 224 (this is the reference to a paper by J.H. Freeman and M.L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $NdCl_3.3C_2H_6O$ prepared by dissolving the hydrate in a small excess of o-ethylformate followed by distillation and crystallization from ethanol.

Ethanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, N.N.; Sakharova, Yu.Gl; Ezhova, T.A.; Izmailova, A.A.

Zh. Neorg. Khim. 1975, *20*, 1479-83; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 830-2.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $NdCl_3.6H_2O$ in 96.8% C_2H_5OH[a]

t/°C	sample 1 g/100 g[b]	sample 2 g/100 g	sample 3 g/100 g	sample 4 g/100 g	mean solubilities g/100 g	mol kg^{-1}[c]
20	35.38	35.54	35.44	35.50	35.47	0.989
30	36.75	36.80	36.87	36.70	36.80	1.026
40	38.92	38.86	39.39	39.03	39.05	1.089
50	42.98	43.19	48.64[d]	42.78	42.89	1.196
60	47.89	47.60	47.68	48.18	47.84	1.334

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

[d] This value appears to be a typographical error in the English translation. To obtain the reported mean solubility of 42.89 g/100 g, the correct value for this data point should be 42.64 g/100 g (compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table were obtained after 3 h of equilibration, and the remaining two data points were obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

The hexahydrate melted at 126.9-128.1°C.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analysed for the metal by titrn with Trilon B, and for Cl by the Volhard method. Found (%) for Nd:40.30, 40.10 (calcd 40.20). Found (%) for Cl; 29.59, 29.45 (calcd 29.69). 98.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn determined refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.8%. (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1,2-Ethanediol (ethylene glycol); $C_2H_6O_2$; [107-21-1]

ORIGINAL MEASUREMENTS:

Racster, L.V.

Masters Thesis. University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283 - 333

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of satd sln		density/g cm^{-3}		solubility $NdCl_3$[a,b]	
t/°C	experimental	average[a]	exptl	av[a]	mol dm^{-3}	mol kg^{-1}
10	1.5788	1.5996	1.3123	-----	0.9508	0.7245
10	1.6205		-----			
15	2.2830	2.2824	1.4029	-----	1.3566	0.9670
15	2.2818		-----			
20	2.7850	2.8059	1.4087	1.4087	1.6678	1.1839
20	2.8268		1.4087			
25	2.7790	2.7843	1.4550	1.4555	1.6549	1.1370
25	2.7895		1.4560			
30	2.3184	2.3214[c]	1.3490	1.3490	1.3798	1.0229
30	2.3244		1.3489			
40	2.9200	2.9539[d]	1.4666	1.4666	1.7554	1.1972
40	2.9878		1.4666			
50	3.3283	3.3517	1.5050	1.5060	1.9922	1.3228
50	3.3750		1.5070			
60	3.0925	3.0814	-----	-----	1.8316	-----
60	3.0703		-----			

[a] Calculated by compilers.

[b] Calculated by compilers from average mass Nd_2O_3 and average density of satd sln.

[c] Author gives av value of 2.3218 g Nd_2O_3: this appears to be a typographical error.

[d] Author gives av value of 2.9589 g Nd_2O_3: this appears to be a typographical error.

The solid phase was not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

$NdCl_3$ and solvent placed in 250 cc glass stoppered bottle and mechanically agitated for 24 h. Rubber tubing placed over stopper and neck of bottle, and end of tubing sealed with a rubber stopper to prevent water from entering the bottle. Slns allowed to settle for 12-18 h, but slight turbidity persisted, particularly at the lowest and highest temps. At 60°C turbidity was significant and appeared different leading author to speculate possible reaction between solute and solvent. Results at 50° and 60°C said to be approximate. Duplicate 10 cc aliquots pipetted from the bottle for each temp. Each aliquot diluted with 25 cc H_2O and the rare earth pptd as the oxalate with oxalic acid. The oxalate was filtered, ignited, and weighed as the oxide. Densities of satd slns detd pycnometrically using pycnometer calibrated at each temp. Densities at 10°C and 15°C may be high due to the condensation of atm water on the surface of the pycnometer.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_2O_3, and evapn of solvent until crystn. Crystals dehydrated by method of Kremers (2). Salt analyzed for presence of H_2O gravimetrically by conversion to oxalate and ignition to the oxide. No water of crystn was found. Ethylene glycol (source and purity not specified) was distilled and initial 5% of distillate discarded. The distilled solvent was stored in a flask sealed with paraffin.

ESTIMATED ERROR:

Soly: precision no better than ± 3% (compilers).

Temp: not specified.

REFERENCES:

1. Some data from Racster's Thesis was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

2. Kremers, H.C. *J. Am. Chem. Soc.* 1925, *17*, 298.

COMPONENTS:	EVALUATOR:
(1) Neodymium chloride; $NdCl_3$; [10024-93-8] (2) 1-Propanol; C_3H_8O; [71-23-8]	Mark Salomon USA ET & DL Ft. Monmouth, NJ, U.S.A. and Tomasz Mioduski Institute of Nuclear Research Warsaw, Poland

CRITICAL EVALUATION:

The solubility of $NdCl_3$ in 1-propanol has been reported in three publications (1-3). West (1) reported solubility data over the temperature range of 283-313 K, and his data are in volume units. The results for 273-323 K (2) and 298 K (3) are both in mass units, but there is serious disagreement between these two publications. For example at 298.2 K Grigorovich (2) reports a solubility of 1.587 mol kg^{-1} (solid phase is $NdCl_3 \cdot 2C_3H_7OH$), and Kirmse (3) reports a solubility of 1.295 % (the nature of the solid phase was not specified).

Since the difference in the results of (2,3) is much greater than the experimental precision in either study, a probable explaination is that the results of Grigorovich are for metastable equilibria (see the critical evaluations for the $NdCl_3$ - CH_3OH and $NdCl_3$ - C_2H_5OH systems).

Kirmse's data probably represent the stable system at 298 K, and for which we assign a *tentative* solubility of 1.295 mol kg^{-1} and a solid phase of $NdCl_3 \cdot nC_3H_7OH$ where $n \geq 3$.

Grigorovich's data probably represent metastable equilibria involving the solid phase $NdCl_3 \cdot 2C_3H_7OH$, and for which the *tentative* solubilities at 273.2 K, 298.2 K and 323.2 K are 1.474 mol kg^{-1}, 1.547 mol kg^{-1} and 1.483 mol kg^{-1}, respectively. Again the low solubility at 323.2 K either represents experimental error or (partial) reversion to the stable higher solvate system.

REFERENCES

1. West, D.H. *Thesis*. University of Illinois. Urbana, IL. 1932. Some data from West's thesis were published in graphical form in reference 4 below.
2. Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986.
3. Kirmse, E.M. *Tr. II Vses Konf. po Teor. Rastvorov* 1971, 200.
4. Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1-Propanol; C_3H_8O; [71-23-8]

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283-313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of saturated sln			solubility of $NdCl_3$[b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	1.4392	1.4694	1.4543	0.8644
20	1.8311	1.8353	1.8332	1.0896
30	2.0328	2.0293	2.0311	1.2072
40	2.5593	2.5753	2.5673	1.5260

[a] Calculated by compilers.

[b] Calculated by compilers using average mass of Nd_2O_3.

The solid phase was not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 cc of alcohol and excess salt placed in 250 cc glass stoppered bottle, and rubber tubing placed over the stopper and neck of the bottle and a rubber stopper fitted into the open end of the tubing to prevent leakage of water into the bottle. The bottle was immersed in a thermostat and mechanically agitated for at least 12 h. The saturated solutions were then permitted to settle for a minimum of 12 h, and duplicate 10 cc aliquots removed with a pipet. Water was added to the aliquots and the sln heated and oxalic acid added to precipitate the rare earth oxalate. The precipitate was filtered, washed with distilled water, and ignited and weighed as the oxide.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_2O_3, and evaporating the sln to a paste which crystallized upon cooling. The hydrate was dried in a stream of dry HCl by slowly increasing the temp. The anhyd salt was stored in cork-stoppered bottles in a desiccator over P_2O_5. Analysis by conversion to the oxalate and ignition to the oxide showed the salt to be anhydr. Commercial alcohol placed over CaO for 1 wk and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was neg.

ESTIMATED ERROR:

Soly: precision probably within ± 3% (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. Some data from West's Thesis was published in graphical form by Hopkins, B.S. Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1-Propanol; C_3H_8O; [71-23-8]

ORIGINAL MEASUREMENTS:

Grigorovich, Z.I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
t/°C	mass %	mol kg^{-1}
0	26.98	1.474
25	28.46	1.587
50	27.10	1.483

[a] Molalities calculated by the compiler. At 25°C the solid phase is $NdCl_3.2C_3H_7OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05 %.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Alcohols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$NdCl_3$ solubility[a,b] mass %	mol kg^{-1}
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	16.8	0.806
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	18.3	0.894
1,2-ethanediol;	$C_2H_6O_2$;	[107-21-1]	31.0	1.793
1-propanol;	C_3H_8O;	[71-23-8]	24.5	1.295

[a] Molalities calculated by the compilers.

[b] Nature of the solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the authors the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 2-Propanol; C_3H_8O; [67-63-0]

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932[1]

VARIABLES:

T/K = 283 - 313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of saturated sln			solubility of $NdCl_3$ [b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	0.0152	0.0149	0.0151	0.0089_5
20	0.0175	0.0187	0.0181	0.0108
30	0.0300	0.0295	0.0298	0.0177
40	0.0743	0.0710	0.0727	0.0432

[a] Calculated by compilers.

[b] Calculated by compilers using average mass of Nd_2O_3.

The solid phase was not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 cc of alcohol and excess salt placed in 250 cc glass stoppered bottle, and rubber tubing placed over the stopper and neck of the bottle and a rubber stopper fitted into the open end of the tubing to prevent leakage of water into the bottle. The bottle was immersed in a thermostat and mechanically agitated for at least 12 h. The saturated solutions were then permitted to settle for a minimum of 12 h, and duplicate 10 cc aliquots removed with a pipet. Water was added to the aliquots and the sln heated and oxalic acid added to precipitate the rare earth oxalate. The precipitate was filtered, washed with distilled water, and ignited and weighed as the oxide.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_2O_3, and evaporating the sln to a paste which crystallized upon cooling. The hydrate was dried in a stream of dry HCl by slowly increasing the temp. The anhyd salt was stored in cork-stoppered bottles in a desiccator over P_2O_5. Analysis by conversion to the oxalate and ignition to the oxide showed the salt to be anhydr. Commercial alcohol placed over CaO for 1 week and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was negative.

ESTIMATED ERROR:

Soly: precision probably within ± 3% (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. Some data from West's Thesis was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 2-Propanol; C_3H_8O; [67-63-0]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P.
Helv. Chim. Acta 1972, 55, 44-52.

Pitteloud, M.N.
These. Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubilities/mol kg^{-1}	
t/°C	a	b
25	0.04	0.06

a. Initial salt is the adduct $NdCl_3.3C_3H_7OH$. Equilibrated solid phase analyzed and found to be $NdCl_3.3C_3H_7OH$.

b. Solutions equilibrated with anhydrous $NdCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $NdCl_3.3C_3H_7OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Neodymium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $NdCl_3.3C_3H_8O$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, *7*, 224 (this is the reference to a paper by J.H. Freeman and M.L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $NdCl_3.3C_3H_8O$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and trans-solvation of the methanol complex with 2-propanol.

Iso-propanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1,2,3-Propanetriol (glycerol); $C_3H_8O_3$; [56-81-5]

ORIGINAL MEASUREMENTS:

Dawson, L.R.

Masters Thesis. University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283 - 333

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 25 cc satd sln		density/g cm^{-3}		soly $NdCl_3$[a,b]	
t/°C	experimental	average[a]	exptl	ave[c]	mol dm^{-3}	mol kg^{-1}
10	0.4982	0.4971	1.2690	1.2737	0.1182	0.0928
10	0.4959		1.2784			
20	2.7237	2.7264	1.3593	1.3595	0.6482	0.4768
20	2.7290		1.3597			
25	2.1932	2.1892	1.3386	1.3395	0.5205	0.3886
25	2.1851		1.3403			
30	1.3407	1.3418	1.3135	1.3141	0.3190	0.2428
30	1.3429		1.3147			
40	2.2771	2.2782	1.330	1.330	0.5417	0.407
40	2.2793		1.329			
50	3.0694	3.0785	1.3727	1.3723	0.7319	0.5334
50	3.0875		1.3718			
60	2.8782	2.8824	-----	-----	0.6853	-----
60	2.8865					

[a] Calculated by compilers.

[b] Based on average mass of Nd_2O_3.

[c] Recalculated by compilers.

The solid phase was not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

About 175 cc glycerol placed in 250 cc glass stoppered bottles and "liberal amounts" of salt added. Mixtures mechanically agitated in thermostat for 24 h, then permitted to stand for 15 h after which slns were still turbid. For analyses, duplicate 25 cc aliquots of turbid slns were taken from each bottle and the rare earth pptd as the oxalate. The ppt was filtered, ignited, and weighed as the oxide. Author states the presence of turbidity had a small effect on the overall accuracy of the soly determinations. Densities of satd slns determined by withdrawing samples from the bottles, placing them into a pycnometer, and weighing "as quickly as possible." The pycnometer was calibrated for each temp.

COMMENTS AND/OR ADDITIONAL DATA:

Since there is a sharp rise in soly from 10°C to 20°C followed by a sharp decrease to 30°C at which point the soly begins to rise again, it is evident that the solid phase in equil with the satd slns is changing. Unsuccessful attempts were made to isolate and identify the solid phases.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd by adding HCl to spectro-pure Nd_2O_3, and evaporating the solvent to the point of crystallization. Dehydration was carried out in a stream of dry HCl first at room temp for 24 h, then at 100°C for ~ 12 h 110°C for ~ 6 h, and 200°C for 3-4 h. HCl prepd from NaCl + H_2SO_4 and passed through H_2SO_4 drying towers. Glycerol (presumably c.p. or A.R. grade: compilers) distilled at reduced pressure and the "first portion" rejected (no other details given).

ESTIMATED ERROR:

Soly: based upon precision in analyses and temp control, overall precision in soly around ± 3% (compilers). Error in accuracy due to turbidity is unknown.

Temp: precision ± 0.5 K except for the 10°C run where precision was ± 1.5 K.

REFERENCES:

1. Some of the data from Dawson's Thesis was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, 19, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1-Butanol; $C_4H_{10}O$; [71-36-3]

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283 - 313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of saturated sln			Solubility of $NdCl_3$[b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	1.9230	1.9047	1.9139	1.1376
20	2.1378	2.1323	2.1351	1.2691
30	2.2974	2.3028	2.3001	1.3672
40	2.9250	2.8954	2.9102	1.7298

[a] Calculated by compilers

[b] Calculated by compilers using average mass of Nd_2O_3.

The solid phase was not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 cc of alcohol and excess salt placed in 250 cc glass stoppered bottle, and rubber tubing placed over the stopper and neck of the bottle and a rubber stopper fitted into the open end of the tubing to prevent leakage of water into the bottle. The bottle was immersed in a thermostat and mechanically agitated for at least 12 h. The saturated solutions were then permitted to settle for a minimum of 12 h, and duplicate 10 cc aliquots removed with a pipet. Water was added to the aliquots and the sln heated and oxalic acid added to precipitate the rare earth oxalate. The precipitate was filtered, washed with distilled water, and ignited and weighed as the oxide.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_2O_3, and evaporating the sln to a paste which crystallized upon cooling. The hydrate was dried in a stream of dry HCl by slowly increasing the temp. The anhyd salt was stored in cork-stoppered bottles in a desiccator over P_2O_5. Analysis by conversion to the oxalate and ignition to the oxide showed the salt to be anhydr. Commercial alcohol placed over CaO for 1 week and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was negative.

ESTIMATED ERROR:

Soly: precision probably within ± 3% (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. Some data from West's Thesis was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1-Butanol; $C_4H_{10}O$; [71-36-3]

ORIGINAL MEASUREMENTS:

Grigorovich, Z.I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

t/°C	solubility[a] mass %	 mol kg^{-1}
0	16.01	0.761
25	17.64	0.855
50	25.00	1.330

[a] Molalities calculated by the compiler. At 25°C the solid phase is $2NdCl_3.3C_4H_9OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05%.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1-Pentanol (amyl alcohol); $C_5H_{12}O$; [71-41-0]

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283 - 313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of saturated sln			solubility of $NdCl_3$[a,b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	2.5211	2.4910	2.5061	1.4896
20	2.5312	2.5403	2.5358	1.5072
30	2.5545	2.5825	2.5685	1.5267
40	2.8731	2.8873	2.8802	1.7120

[a] Calculated by compilers.

[b] Calculated by compilers from average mass Nd_2O_3.

The solid phase was not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 cc of alcohol and excess salt placed in 250 cc glass stoppered bottle, and rubber tubing placed over the stopper and neck of the bottle and a rubber stopper fitted into the open end of the tubing to prevent leakage of water into the bottle. The bottle was immersed in a thermostat and mechanically agitated for at least 12 h. The saturated solutions were then permitted to settle for a minimum of 12, and duplicate 10 cc aliquots removed with a pipet. Water was added to the aliquots and the sln heated and oxalic acid added to precipitate the rare earth oxalate. The precipitate was filtered, washed with distilled water, and ignited and weighed as the oxide.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_2O_3, and evaporating the sln to a paste which crystallized upon cooling. The hydrate was dried in a stream of dry HCl by slowly increasing the temp. The anhyd salt was stored in cork-stoppered bottles in a desiccator over P_2O_5. Analysis by conversion to the oxalate and ignition to the oxide showed the salt to be anhydr. Commercial alcohol placed over CaO for 1 wk and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was neg.

ESTIMATED ERROR:

Soly: precision probably within ± 3% (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. Some data from West's Thesis was published in graphical form by Hopkins, B.S. Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1-Pentanol; $C_5H_{12}O$; [71-41-0]

ORIGINAL MEASUREMENTS:

Grigorovich, Z.I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
t/°C	mass %	mol kg^{-1}
0	15.96	0.758
25	15.30	0.721
50	14.23	0.662

[a]Molalities calculated by the compiler. At 25°C the solid phase is $NdCl_3 \cdot 1.0C_5H_{12}O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermo= graphically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05%.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 2-Methoxyethanol (methyl cellosolve); $C_3H_8O_2$; [109-86-4]

ORIGINAL MEASUREMENTS:

McCarty, C.N.

Master of Science Thesis. The University of Illinois. Urbana, IL, USA. 1933.

VARIABLES:

T/K = 273 - 323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Nd_2O_3 [a] g/25 cc	$NdCl_3$ [b] g/dm^3	$NdCl_3$ [b] mol/dm^3
0	0.3470	20.67	0.0825
10	0.5709	34.02	0.1357
20	0.7465	44.48	0.1775
30	0.9132	54.41	0.2171
40	1.1691	69.66	0.2780
50	1.2654	75.39	0.3009

[a] Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $NdCl_3$ from preparations 1 and 2.

[b] Recalculated by the compilers using 1977 IUPAC recommended atomic masses.

Equilibrated solid phase not analyzed

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in bottles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and were sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber stopper was fitted into the open end of the tubing. After equilibration, the solutions were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet, two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

ESTIMATED ERROR:

Soly: precision probably within 3% (compilers).

Temp: precision ± 0.2 K (author).

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 wk and then distilled. A middle portion (fraction not specified) was retained and stored in a stoppered flask: b.p. 123°C. Nd salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide (no details) and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystallized salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extraction of the chloride was carried out with HCl satd ether, and the resulting chloride heated at 60°C first in a stream of dry HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 and by passing the resulting HCl through H_2SO_4 drying towers.

COMPONENTS: (1) Neodymium chloride; $NdCl_3$; [10024-93-8] (2) 2-Ethoxyethanol (ethyl cellosolve); $C_4H_{10}O_2$; [110-80-5]	**ORIGINAL MEASUREMENTS:** McCarty, C.N. *Master of Science Thesis.* The University of Illinois. Urbana, IL, USA. 1933.
VARIABLES: T/K = 273 - 323	**PREPARED BY:** M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Nd_2O_3 [a] g/25 cc	$NdCl_3$ [b] g/dm^3	$NdCl_3$ [b] mol/dm^3
0	0.2656	15.82	0.0631
10	0.4642	27.66	0.1104
20	0.6396	37.95	0.1514
30	0.7627	45.44	0.1813
40	0.9410	56.07	0.2237
50	1.1581	69.00	0.2753

[a] Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $NdCl_3$ from preparations 1 and 2.

[b] Recalculated by the compilers using 1977 IUPAC recommended atomic masses.

Equilibrated solid phase not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in bottles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and were sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber stopper was fitted into the open end of the tubing. After equilibration, the solutions were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet, two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 week and then distilled. A middle portion (fraction not specified) was retained and stored in a stoppered flask: b.p. 134°C. Nd salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide (no details) and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystallized salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extraction of the chloride was carried out with HCl satd ether, and the resulting chloride heated at 60°C first in a stream of dry HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 and by passing the resulting HCl through H_2SO_4 drying towers.

ESTIMATED ERROR:

Soly: precision probably within 3% (compilers).

Temp: precision ± 0.2 K (author).

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Diethyl ether (ethyl ether); $C_4H_{10}O$; [60-29-7]

ORIGINAL MEASUREMENTS:

Dzhuraev, Kh. Sh.; Mirsaidov, U.; Kurbanbekov, A.; Rakhimova, A.

Dokl. Akad. Nauk Tadzh. SSR 1976, *19*, 32-4.

VARIABLES:

$T/K = 293$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $NdCl_3$ in diethyl ether at 20°C was reported to be

$$5.8 \times 10^{-3} \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$2.31 \times 10^{-4} \text{ mol kg}^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrium was attained within 24 h and it was verified by constancy in the Nd concentration. Both the saturated solution and the equilibrated solid phase were analyzed. Nd determined by complexometric titration in the presence of urotropine buffer and methyl-thymol blue indicator. Chloride determined by titration with $AgNO_3$. The solid phase corresponded to $NdCl_3.0.3Et_2O$ (the etherate was dried under vacuum at 40°C prior to analysis).

SOURCE AND PURITY OF MATERIALS:

Anhydrous $NdCl_3$ prepared by the ethanol solvate method (no details given).

Ethyl ether was dried with Na and distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. *Z. Chem.* 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility^{a,b} mass %	solubility^{a,b} mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.04	1.6×10^{-3}
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	0.07	2.8×10^{-3}
1,3-dioxolane;	$C_3H_6O_2$;	[646-06-0]	0.8	3.2×10^{-2}
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.1	4.0×10^{-2}

[a] Molalities calculated by the compilers.

[b] Nature of the solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Nd was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Dressler, H.

Z. Chem. 1975, *15*, 239-40.

VARIABLES:

Room Temperature (293-298 K)

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	mol kg^{-1}
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	0.4[b]	0.016
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	0.02[c]	8×10^{-4}

[a] Molalities calculated by the compilers.

[b] Solid phase $NdCl_3$:$C_8H_{18}O$ found to be 1: > 2.

[c] Solid phase not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated (at room temperature) until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}. Nd was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.; Auer-Welsbach, C.

Monatsh. Chem. 1965, *96*, 602-5.

VARIABLES:

Room temperature: T/K about 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $NdCl_3$ in tetrahydrofuran at room temperature (about 20°C) was reported as

1.16 g/100 ml solution (0.046 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor for 60-80 hours at room temperature. Neodymium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. For the solid phase analysis, the solvent was determined by difference.

Anhydrous substances were handled in a dry box through which was passed a current of dry and CO_2-free nitrogen.

Analysis of the solid phase yielded a $NdCl_3$:C_4H_8O ratio of 1:1.99.

SOURCE AND PURITY OF MATERIALS:

Sources and purities not specified.

$NdCl_3$ prepared by reaction of the oxide at high temperatures with an excess of NH_4Cl followed by heating the product in a current of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.

Zh. Neorg. Khim. 1975, *20*, 908-14; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 508-11.

VARIABLES:

One temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solution

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg^{-1}(compiler)	density/g cm^{-3}
31.8	1.27	408.0	1.63	1.86	1.28

The solid phase is $NdCl_3$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd slns prepared isothermally with magnetic stirring. Equil was attained after 25-30 d. The sln was centrifuged and an aliquot for analysis taken and added to methanol and pptd with aq NH_3. The pptd $Pr(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analysed (no details given) for phosphorous and only anhydr $PrCl_3$ was found. All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complextion between TBP and $PrCl_3$ in solution. Additional studies with unsaturated solutions (IR spectra, viscosity, molar conductivities) are discussed in the source paper.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $NdCl_3$ prepd by chlorination of Nd_2O_3 with CCl_4 vapor (1,2). Source and purity of materials not given. Nd was analyzed gravimetrically, and Cl by Volhard's method. Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* 1964, *9*, 1427.
2. Novikov, G.I., Tolmacheva, V.D. *Zh. Prikl Khim.* 1965, *38*, 1160.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse. E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	mol kg^{-1}
1-propanamine;	C_3H_9N;	[107-10-8]	25.7	1.380
2-propanamine;	iso-C_3H_9N;	[75-31-0]	0.1	0.004
2-propen-1-amine[b];	C_3H_7N;	[107-11-9]	0.05	0.002

[a] Molalities calculated by the compilers.

[b] The original paper simply specifies the solvent as $C_3H_5NH_2$, and upon request the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1761-6;
Russ, J. Inorg. Chem. (*Engl. Transl.*) 1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of the anhydrous salt at 25 ± 3°C was given as

$$0.119 \pm 0.005 \text{ mol dm}^{-3}$$

Starting with the solvate $NdCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.125 \pm 0.001 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at room temperature (25 ± 3°C).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature (25 ± 3°C) until equilibrium was reached. Aliquots were withdrawn periodically and analysed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analysed and found to be $NdCl_3.3C_6H_{18}N_3OP$.

The solvate was analysed for metal content by complexometric titrn, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

Anhyd $NdCl_3$ prepd similarly to that in (1) by subliming NH_4Cl from a mixt of $NdCl_3$ and 6 moles of NN_4Cl in a stream of inert gas at 200-400°C (NdOCl content less than 3%). The solvent was purified as in (2).

$NdCl_3.3C_6H_{18}N_3OP$ prepd by dissolving the hydrate in $C_3H_{18}N_3OP$ and heating to 140-145° C for 5 m. The solvate was pptd by addition of abs ether, washing 7 times with ether, and drying over P_2O_5 in a stream of dry nitrogen. Yield was about 90 %.

ESTIMATED ERROR:

Soly: precision ± 0.001 mol dm^{-3} at a 95 % level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.
2. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Gallium chloride; $GaCl_3$; [13450-90-3]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Zh. Neorg. Khim. 1976, *21*, 2556-7; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1976, *21*, 1406.
Zh. Fiz. Khim. 1979, *53*, 1588-90; *Russ. J. Phys. Chem. (Engl. Transl.)* 1979, *53*, 897-9.

VARIABLES:

Concentration of $GaCl_3$ at 298 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$GaCl_3/NdCl_3$ molar ratio	$GaCl_3$ concentration mmol dm^{-3}	$NdCl_3$ solubility mmol dm^{-3}	equiv conductivity S cm^2 $equiv^{-1}$
pure $POCl_3$	——	0.01	----
1:22.4	1.0	23	4.6
1:17.8	2.3	39	3.9
1:11.9	3.0	36	5.6
1:10.0	4.1	41	5.2
1:7.6	5.5	42	5.1
1:7.5	6.1	46	4.8
1:5.9	7.1	42	5.4
1:6.3	8.1	51	4.6
1:5.6	9.1	51	5.7
1:5.3	10.1	54	5.8

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Anhydrous $GaCl_3$ and $NdCl_3$ were added to $POCl_3$ in an argon filled dry box. The specified amounts of $GaCl_3$ were dissolved first in $POCl_3$. Solubilities were determined at 25°C.

No other information given.

COMMENTS AND/OR ADDITIONAL DATA:

The authors state that the increase in the solubility of $NdCl_3$ upon increasing $GaCl_3$ concentration is due to complex formation.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $NdCl_3$ and $GaCl_3$ were prepared as described in (1).

$POCl_3$ was dehydrated with P_2O_5 and distilled twice under vacuum.

Solutions were prepared in an argon filled dry box.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Puzankova, N.L.; Slastenova, N.M.; Solov'ev, M.A.; Batyaev, I.M. *Sintez i Issledovanie Nizkotemperaturnykh Khloridnykh Matrits.* XXVI Herzen Lecture. Scientific Papers. 1973.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Gallium chloride; $GaCl_3$; [13450-89-0]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Zh. Fiz. Khim. 1979, *53*, 1588-90; *Russ. J. Phys. Chem.* (*Engl. Transl.*) 1979, *53*, 897-9. *Zh. Neorg. Khim.* 1976, *21*, 2556-7; *Russ. J. Inorg. Chem.* (*Engl. Transl*) 1976, *21*, 1406.

VARIABLES:

Concentration of $GaCl_3$

Temp not specified, but probably 298 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$GaCl_3$ concentration mol dm^{-3}	$NdCl_3$ solubility mol dm^{-3}
0	0.01
1.0	22
2.3	39
3.0	36
4.1	41
5.5	42
6.1	46
7.1	42
9.1	51
9.1	51
10.1	54

Authors state that the increase in the solubility of $NdCl_3$ with increasing concentration is due to the formation of $NdCl_3(POCl_3)_x(GaCl_3)_y$ complexes. Stepwise formation constants for these complexes are given below.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The $NdCl_3$-$POCl_3$-$GaCl_3$ systems were synthesized in sealed tubes by the solvolthermal method at 120°C as described in (1). No other details given. The compilers assume that after treating the solutions at 120°C for about 2 h, they were then isothermally equilibrated at 25°C (see the compilations of references 1 and 2 in this volume.)

COMMENTS AND/OR ADDITIONAL DATA:

For the equilibria

$$NdCl_3 + yGaCl_3 = NdCl_3.yGaCl_3$$

the following stepwise formation constants constants, βy, were given:

$$\beta_1 = (2.7 \pm 0.1) \times 10^6$$

$$\beta_2 = (3.6 \pm 0.2) \times 10^9$$

$$\beta_3 = (3.8 \pm 0.2) \times 10^8$$

$$\beta_4 = (3.6 \pm 0.2) \times 10^{14}$$

SOURCE AND PURITY OF MATERIALS:

Anhydrous metal chlorides were prepared as described in (3).

$POCl_3$ was purified by the usual method.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, *45*, 1176.
2. Batyaev, I.M.; Solov'ev, M.A. *Izv. Akad. Nauk SSSR, Neorg. Mater.* 1977, *13*, 104.
3. Slastenova, N.M.; Batyaev, I.M.; Bel'kova, N.L.; Kuz'menko, A.S.; Ryabov, E.N. *Zh. Prikl. Khim.* 1975, *48*, 1953.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

$SnCl_4$ concentration

T/K = 293 and 333

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4:POCl_3$ ratio (by volume)	$SnCl_4$ concn mol dm^{-3}	solubility of Nd_2O_3/mol dm^{-3} [a]		
		20°C [b]	20°C [c]	60°C [d]
0	0	0.007	-----	0.003
1:250	0.035	0.11	0.093	0.092
1:100	0.085	0.27	0.23	0.20
1:50	0.17	0.30	0.30	0.26
1:25	0.33	0.26	0.20	0.31
1:15	0.59	0.15	0.13	0.22
1:10	0.78	0.11	0.10	0.22
1:5	1.4	-----	-----	0.20
1:1.5	3.0	-----	-----	0.19
1:1	4.6	-----	-----	0.090
4:1	6.8	-----	0.010	0.010
pure $SnCl_4$	8.5	2×10^{-4}	-----	-----

[a] This is also the solubility of $NdCl_3$ since the oxide is quantitatively converted to the chloride according to

$$Nd_2O_3 + 6POCl_3 = 2NdCl_3 + 3P_2O_3Cl_4$$

Assuming $P_2O_3Cl_4$ to be soluble, the equilibrated solutions would then constitute a four component mixture.

[b] Preheated at 120°C for 2 hours.

[d] No pretreatment.

[c] Preheated at 60°C (time not specified).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Nd_2O_3 were placed in sealed ampoules, heated to 120°C for 2 hours to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2 hours. Without preheating equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20°C to 2 hours.

Nd was determined by colorimetric analysis, and in some cases by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

The solubility of $NdCl_3$ in pure $POCl_3$ is small, but in the presence of $SnCl_4$ the solubility increases due to complexation:

$$2NdCl_3 + 3SnCl_4 = Nd_2(SnCl_6)_3$$

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorous oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Izv. Akad. Nauk SSSR, Neorg. Mater. 1977, *13*, 104-8.

VARIABLES:

$SnCl_4$ concentration

T/K = 293

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$SnCl_4/POCl_3$ volume ratio	$SnCl_4$ concn 10^2c_2/mol dm^{-3}	$SnCl_4/NdCl_3$ mol ratio	solubility of $NdCl_3$ 10^3c_1/mol dm^{-3}
0.25:29.7	5.0	10:1	5.0
0.50:29.5	10.0	19.2:1	5.2
1.0:29.0	15.0	27.7:1	5.4
1.25:28.7	20.0	35.7:1	5.6
1.50:28.5	25.0	43.9:1	5.7
1.75:28.2	30.0	50.91:1	5.9
2.0:28.0	35.0	57.4:1	6.1
2.25:27.7	40.0	63.5:1	6.3
2.50:27.5	45.0	69.2:1	6.5
2.75:27.2	50.0	74.6:1	6.7
3.0:27.0	55.0	79.7:1	6.9

The solubility of $NdCl_3$ in pure $POCl_3$ was reported to be 10^{-5} mol dm^{-3} (solubility product = 2.7×10^{-19} mol^4 dm^{-12}). The increasing solubility of $NdCl_3$ observed upon increasing the $SnCl_4$ concentration from 0.05-0.55 mol dm^{-3} is attributed to outer sphere coordination of $SnCl_4$ by $POCl_3$. Stability constants, β_i, for the reactions

$$NdCl_3 + nPOCl_3 = NdCl_3.nPOCl_3$$

were calculated for i = 1-4.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solvent-solute mixtures were sealed in glass ampoules and rotated in an air thermostat at 120°C for 24 h. The ampoules were then rotated in a thermostat at 20°C for 24 h. After equilibrium was reached, aliquots were withdrawn in a dry argon atmosphere and evaporated in vacuum. The dry residues were hydrolyzed, the hydrolyzed products filtered, and Nd determined spectrophotometrically. The solutions were analyzed at pH 3 (phormic buffer) using 0.05% arsenazo III solution. A calibration curve was used for these analyses.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

COMMENTS AND/OR ADDITIONAL DATA:

Conflicting data exist between the source paper and earlier work (1). In the source paper the solubility of $NdCl_3$ in pure $POCl_3$ is given as 10^{-5} mol dm^{-3} compared to 7×10^{-3} mol dm^{-3} reported in (1). In (1), the enhancement of the solubility of $NdCl_3$ is attributed to the formation of $Nd_2(SnCl_6)_3$, and in the present work the enhancement is attributed to coordination of $SnCl_4$ by $POCl_3$.

REFERENCES:

1. Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, *45*, 1176. (see previous page for the compilation of this paper).

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Zh. Fiz. Khim. 1979, *53*, 1588-90;
Russ. J. Phys. Chem. (Engl. Transl.) 1979, *53*, 897-9.

VARIABLES:

Concentration of $SnCl_4$

Temp not specified, but probably 298 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$SnCl_4$ concentration mol dm^{-3}	$NdCl_3$ solubility mol dm^{-3}
0	0.01
50	5.0
100	5.2
150	5.4
200	5.6
250	5.7
300	5.9
350	6.1
400	6.3
450	6.5
500	6.7

Authors state that the increase in the solubility of $NdCl_3$ with increasing concentration is due to the formation of $NdCl_3(POCl_3)_x(SnCl_4)_y$ complexes. Stepwise formation constants for these complexes are given below.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The $NdCl_3$-$POCl_3$-$SnCl_4$ systems were synthesized in sealed tubes by the solvolthermal method at 120°C as described in (1). No other details given. The compilers assume that after treating the solutions at 120°C for about 2 h, they were then isothermally equilibrated at 25°C as in (2): see the compilations of references 1 and 2 in this volume

COMMENTS AND/OR ADDITIONAL DATA:

For the equilibria

$$NdCl_3 + ySnCl_4 = NdCl_3.ySnCl_4$$

the following stepwise formation constants constants, βy, were given:

$\beta_1 = (1.5 \pm 0.1) \times 10^4$

$\beta_2 = (3.8 \pm 0.2) \times 10^6$

$\beta_3 = (3.6 \pm 0.2) \times 10^5$

$\beta_4 = (1.2 \pm 0.1) \times 10^8$

SOURCE AND PURITY OF MATERIALS:

Anhydrous metal chlorides were prepared as described in (3).

$POCl_3$ and $SnCl_4$ were purified by the usual methods.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, *45*, 1176.
2. Batyaev, I.M.; Solov'ev, M.A. *Zh. Neorg. Khim.* 1976, *21*, 2556.
3. Slastenova, N.M.; Batyaev, I.M.; Bel'kova, N.L.; Kuz'menko, A.S.; Ryabov, E.N. *Zh. Prikl. Khim.* 1975, *48*, 1953.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Zinc chloride; $ZnCl_2$; [7846-85-7]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Zh. Fiz. Khim. 1979, 53, 1588-90; *Russ. J. Phys. Chem.* (*Engl. Transl.*) 1979, 53, 897-9.

VARIABLES:

Concentration of $ZnCl_2$

Temp not specified, but probably 298 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$ZnCl_2$ concentration mol dm^{-3}	$NdCl_3$ solubility mol dm^{-3}
0	0.01
17	2.0
16	1.9
14	1.8
13	1.7
12	1.6
12	1.5
11	1.4
10	1.3
9	1.2
9	1.1

Authors state that the increase in the solubility of $NdCl_3$ with increasing concentration is due to the formation of $NdCl_3(POCl_3)_x(ZnCl_2)_y$ complexes. Stepwise formation constants for these complexes are given below.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The $NdCl_3$-$POCl_3$-$ZnCl_2$ systems were synthesized in sealed tubes by the solvothermal method at 120°C as described in (1). No other details given. The compilers assume that after treating the solutions at 120°C for about 2 h, they were then isothermally equilibrated at 25°C as in (2): see the compilations of references 1 and 2 in this volume.

COMMENTS and/OR ADDITIONAL DATA:

For the equilibria

$$NdCl_3 + yZnCl_2 = NdCl_3.yZnCl_2$$

the following stepwise formation constants constants, β_y, were given:

$$\beta_1 = (6.7 \pm 0.3) \times 10^4$$
$$\beta_2 = (2.5 \pm 0.1) \times 10^5$$
$$\beta_3 = (7.4 \pm 0.3) \times 10^7$$
$$\beta_4 = (5.4 \pm 0.2) \times 10^9$$

SOURCE AND PURITY OF MATERIALS:

Anhydrous metal chlorides were prepared as described in (3).

$POCl_3$ was purified by the usual method.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, 45, 1176.
2. Batyaev, I.M.; Solov'ev, M.A. *Zh. Neorg. Khim.* 1976, 21, 2556.
3. Slastenova, N.M.; Batyaev, I.M.; Bel'kova, N.L.; Kuz'menko, A.S.; Ryabov, E.N. *Zh. Prikl. Khim.* 1975, 48, 1953.

COMPONENTS:

(1) Neodymium bromide; $NdBr_3$; [13536-80-6]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

The solubility of $NdBr_3$ in 1,2-diethoxyethane at 25°C was reported as

0.5 mass %

The corresponding molality calculated by the compiler is

$0.013 \text{ mol kg}^{-1}$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Neodymium bromide; $NdBr_3$; [13536-80-6]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Dressler, H.

Z. Chem. 1975, *15*, 239-40.

VARIABLES:

Room Temperature: (293-298 K)

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	mol kg^{-1}
1-methoxybutane;	$C_5H_{12}O$;	[628-28-4]	1.6	0.042
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	6.1	0.169
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	7.1[b]	0.199
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	6.9[c]	0.193
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	2.1	0.056
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	3.6	0.097

[a] Molalities calculated by the compilers. Composition of most solid phases were not specified.

[b] Solid phase is $NdBr_3.2C_8H_{18}O$.

[c] Solid phase is $NdBr_3.2C_9H_{20}O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated (at room temperature) until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}. Nd was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS: (1) Neodymium bromide; $NdBr_3$; [13536-80-6] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	**ORIGINAL MEASUREMENTS:** Rossmanith, K. *Monatsh. Chem.* 1966, 97, 1357-64.
VARIABLES: Room Temperature: T/K = 294-296	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $NdBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.71 g per 100 ml of solution (0.018 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Neodymium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $NdBr_3.3.5C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $NdBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Neodymium bromide; $NdBr_3$; [13536-80-6] (2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]	Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. *Z. Chem.* 1968, *8*, 472-3. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
Room temperature: T/K around 298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $NdBr_3$ in p-dioxane at about 25°C was given as

$$0.9_5 \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$0.025 \text{ mol kg}^{-1}$$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Nd was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Neodymium bromide; $NdBr_3$; [13536-80-6]

(2) 1,2-Ethanediamine; $C_2H_8N_2$; [107-15-3]

ORIGINAL MEASUREMENTS:

Moeller, T.; Zimmerman, P.A.

J. Am. Chem. Soc. 1953, *75*, 3940-5.

VARIABLES:

T/K = 303.15

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $NdBr_3$ in $H_2NCH_2CH_2NH_2$ at 30°C was given as

0.936 g/100g solvent

The corresponding molality calculated by the compiler is

$0.0244 \text{ mol kg}^{-1}$

Solid phase composition not determined.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The isothermal method was used. Reaction mixtures were sealed in 25 x 200 mm test tubes and thermostated for one week at 30 ± 0.05°C with frequent agitation. The density of the supernatant liquid was determined pycnometrically (but not reported in the source paper), and the neodymium content determined by precipitating the hydrous hydroxide from an aliquot by adding excess water, igniting to the oxide and weighing.

All anhydrous substances were handled in a dry box through which a current of nitrogen was passed. The nitrogen was freed of CO_2 and moisture by passage through concentrated sulfuric acid, soda lime, and Drierite. All solutions were prepared in the dry box, and were sealed before being removed.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 was converted to the bromide by high temperature reaction with NH_4Br. Unreacted NH_4Br was removed by heating in N_2 and then in vacuo. The oxide labeled Nd-28 from University stocks contained traces of other rare earth metals. Ethylenediamine (Carbide and Carbon Chemicals) purified as in (1). The product had an electrolytic resistivity of 2.65×10^5 ohm cm^{-1}. The solvent was stored under nitrogen in glass stoppered flasks sealed with wax.

ESTIMATED ERROR:

Soly: precision probably 1% at best (compiler).

Temp: precision ± 0.05K (authors).

REFERENCES:

1. Putnam, G.L.; Kobe, K.A. *Trans. Electrochem. Soc.* 1938, *74*, 609.

COMPONENTS:

(1) Neodymium bromide; $NdBr_3$; [13536-80-6]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] mol kg^{-1}
2-propanamine;	iso-C_3H_9N;	[75-31-0]	15.9	0.492
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	10.8	0.315
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	18.1	0.576
di-2-butylamine;	(sec-$C_4H_9)_2NH$;	[626-23-3]	0.02	5×10^{-4}

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Neodymium bromide; $NdBr_3$; [13536-80-6]

(2) Ethanolamine; C_2H_7NO; [75-39-8]

ORIGINAL MEASUREMENTS:

Moeller, T.; Zimmerman, P.A.

J. Am. Chem. Soc. 1953, *75*, 3940-5.

VARIABLES:

T/K = 303.15

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $NdBr_3$ in $H_2NCH_2CH_2OH$ at 30°C was given as

3.20 g/100g solvent

The corresponding molality calculated by the compiler is

$0.0833 \text{ mol kg}^{-1}$

Solid phase composition not determined.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The isothermal method was used. Reaction mixtures were sealed in 25 x 200 mm test tubes and thermostated for one week at 30 ± 0.05°C with frequent agitation. The density of the supernatant liquid was determined pycnometrically (but not reported in the source paper), and the neodymium content determined by precipitating the hydrous hydroxide from an aliquot by adding excess water, igniting to the oxide and weighing.

All anhydrous substances were handled in a dry box through which a current of nitrogen was passed. The nitrogen was freed of CO_2 and moisture by passage through concentrated sulfuric acid, soda lime, and Drierite. All solutions were prepared in the dry box, and were sealed before being removed.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 was converted to the bromide by high temperature reaction with NH_4Br. Unreacted NH_4Br was removed by heating in N_2 and then in vacuo. The oxide labeled Nd-28 from University stocks contained traces of other rare earth metals. Ethanolamine (Carbide and Carbon Chemicals) purified as in (1). The product boiled at 168°C (uncor), had a density of 1.0108 g/ml at 26.5°C and had an electrolytic conductivity of 1.93×10^{-5} S cm^{-1} at 20°C. It was stored under N_2 in flasks sealed with wax.

ESTIMATED ERROR:

Soly: precision probably 1% at best (compiler).

Temp: precision ± 0.05 K (authors).

REFERENCES:

1. Dirkse, T.P.; Briscoe, H.T. *Metal Ind.* 1938, *36*, 284.

COMPONENTS:

(1) Neodymium bromide; $NdBr_3$; [13536-80-6]

(2) Morpholine; C_4H_9NO; [110-91-8]

ORIGINAL MEASUREMENTS:

Moeller, T.; Zimmerman, P.A.

J. Am. Chem. Soc. 1953, 75, 3940-5.

VARIABLES:

$T/K = 303.15$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $NdBr_3$ in morpholine at 30°C was given as

0.099 g/100g solvent

The corresponding molality calculated by the compiler is

2.58×10^{-3} mol kg^{-1}

Solid phase composition not determined.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The isothermal method was used. Reaction mixtures were sealed in 25 x 200 mm test tubes and thermostated for one week at 30 ± 0.05°C with frequent agitation. The density of the supernatant liquid was determined pycnometrically (but not reported in the source paper), and the neodymium content determined by precipitating the hydrous hydroxide from an aliquot by adding excess water, igniting to the oxide and weighing.

All anhydrous substances were handled in a dry box through which a current of nitrogen was passed. The nitrogen was freed of CO_2 and moisture by passage through concentrated sulfuric acid, soda lime, and Drierite. All solutions were prepared in the dry box, and were sealed before being removed.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 was converted to the bromide by high temperature reaction with NH_4Br. Unreacted NH_4Br was removed by heating in N_2 and then in vacuo. The oxide labeled Nd-28 from University stocks contained traces of other rare earth metals. Morpholine was purified as in (1), and had a density of 0.9863 g/ml at 27°C, and an electrolytic conductivity of 3.368×10^{-8} S cm^{-1}. The solvent was stored under nitrogen in glass stoppered flasks sealed with wax.

ESTIMATED ERROR:

Soly: precision probably 1% at best (compiler).

Temp: precision ± 0.05 K (authors).

REFERENCES:

1. Dermer, V.H.; Dermer, O.C. *J. Am. Chem. Soc.* 1937, 59, 1148.

COMPONENTS:

(1) Neodymium iodide; NdI_3; [13813-24-6]

(2) 1-Butanol; $C_4H_{10}O$; [71-36-3]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Yastrebova, L.F.; Grigor, T.I.; Kuznetsova, G.P.; Stepin, B.D.

Zh. Neorg. Khim. 1981, *26*, 2238-9; *Russ. J. Inorg. Chem. (Engl. Transl.)*, 1981, *26*, 1203-4.

VARIABLES:

Composition at 273 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility at 0°C

solvent[b]	$NdI_3.9H_2O$ mass %	NdI_3[a] mass %	NdI_3[a] mol kg^{-1}	solid phase
$n\text{-}C_4H_9OH$	57.97	44.29	1.514	$NdI_3.9H_2O$
H_2O	87.21	66.63	3.804	"

[a] Results for the anhydrous salt calculated by the compilers.

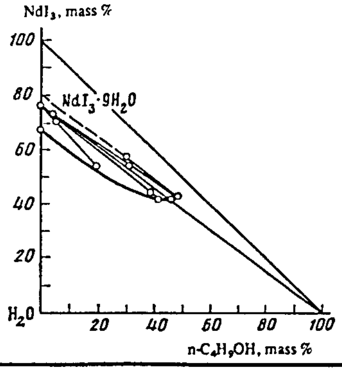
[b] Authors' original results reported in terms of the solubility of the nonohydrate in the pure alcohol. Accounting for the waters of hydration, the compilers calculate that at equilibrium the solvent contains 75.44 mass % alcohol and 24.56 mass % water.

The solubility isotherm for the ternary system is reproduced at the right. Numerical data for the various compositions represented in this diagram were not given.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. No information was given on how equilibrium was ascertained. Aliquots of saturated solution were withdrawn and analyzed for the metal complexometrically, for iodide by a potentiometric volumetric argentometric method, and for water by the Karl Fischer method. The alcohol and water contents in the mixtures were found by quantitative gas chromatography. Solid phase compositions were determined by Schreinemakers' method of resudues.



SOURCE AND PURITY OF MATERIALS:

The nonohydrate, $NdI_3.9H_2O$, was synthesized according to (1,2).

The alcohol was dried and purified by "recommended" methods.

The source and purity of water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Yakimova, Z.P.; Kuznetsova, G.P.; Yastrebova, L.F.; Stepin, B.D. *Zh. Neorg. Khim.* 1977, *22*, 251.
2. Belousova, A.P.; Kuznetsova, G.P.; Rukk, N.S.; Stepin, B.D. *Zh. Neorg. Khim.* 1979, *24*, 1410.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Neodymium iodide; NdI_3; [13813-24-6] (2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]	Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. *Z. Chem.* 1968, *8*, 472-3. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
Room temperature: T/K around 298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of NdI_3 in p-dioxane at about 25°C was given as

1.6 mass %.

The corresponding molality calculated by the compiler is

0.031 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Nd was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Neodymium iodide; NdI_3; [13813-24-6]

(2) 1,2-Ethanediamine; $C_2H_8N_2$; [107-15-3]

ORIGINAL MEASUREMENTS:

Moeller, T.; Zimmerman, P.A.

J. Am. Chem. Soc. 1953, *75*, 3940-3.

VARIABLES:

T/K = 303.15

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of NdI_3 in $H_2NCH_2CH_2NH_2$ at 30°C was given as

2.53 g/100 solvent

The corresponding molality calculated by the compiler is

$0.0482 \text{ mol kg}^{-1}$

Solid phase composition not determined.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The isothermal method was used. Reaction mixtures were sealed in 25 x 200 mm test tubes and thermostated for one week at 30 ± 0.05°C with frequent agitation. The density of the supernatant liquid was determined pycnometrically (but not reported in the source paper), and the neodymium content determined by precipitating the hydrous hydroxide from a measured aliquot by adding excess water, igniting to the oxide and weighing.

All anhydrous substances were handled in a dry box through which a current of nitrogen was passed. The nitrogen was freed of CO_2 and moisture by passage through concentrated sulfuric acid, soda lime, and Drierite. All solutions were prepared in the dry box, and were sealed before being removed.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 was converted to the iodide by high temperature reaction with NH_4I. Unreacted NH_4I was removed by heating in N_2 followed in vacuo. The oxide labeled Nd-28 from University stocks contained traces of other rare earth metals. Ethylenediamine (Carbide and Carbon Chemicals) purified as in (1). The product had an electrolytic resistivity of 2.65×10^5 ohm cm^{-1}. The solvent was stored under nitrogen in glass stoppered flasks sealed with wax.

ESTIMATED ERROR:

Soly: precision probably 1% at best (compiler).

Temp: precision ± 0.05 K (authors).

REFERENCES:

1. Putnam, G.L.; Kobe, K.A. *Trans. Electrochem. Soc.* 1938, *74*, 609.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Neodymium iodide; NdI_3; [13813-24-6] (2) Alkyl amines	Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	16.6	0.379
2-propanamine;	iso-C_3H_9N;	[75-31-0]	6.2	0.126
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	8.3	0.172

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, 24, 387.

COMPONENTS:

(1) Neodymium iodide; NdI_3; [13813-24-6]

(2) Ethanolamine; C_2H_7NO; [75-39-8]

ORIGINAL MEASUREMENTS:

Moeller, T.; Zimmerman, P.A.

J. Am. Chem. Soc. 1953, *75*, 3940-3.

VARIABLES:

T/K = 303.15

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of NdI_3 in $H_2NCH_2CH_2OH$ at 30°C was given as

1.93 g/100 solvent

The corresponding molality calculated by the compiler is

$0.0368 \text{ mol kg}^{-1}$

Solid phase composition not determined.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The isothermal method was used. Reaction mixtures were sealed in 25 x 200 mm test tubes and thermostated for one week at 30 ± 0.05°C with frequent agitation. The density of the supernatant liquid was determined pycnometrically (but not reported in the source paper), and the neodymium content determined by precipitating the hydrous hydroxide from a measured aliquot by adding excess water, igniting to the oxide and weighing.

All anhydrous substances were handled in a dry box through which a current of nitrogen was passed. The nitrogen was freed of CO_2 and moisture by passage through concentrated sulfuric acid, soda lime, and Drierite. All solutions were prepared in the dry box, and were sealed before being removed.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 was converted to the iodide by high temperature reaction with NH_4I. Unreacted NH_4I removed by heating in N_2 and in vacuo. The oxide labeled Nd-28 from University stocks contained traces of other rare earth metals. Ethanolamine (Carbide and Carbon Chemicals) purified as in (1). The product boiled at 168°C (uncor), had a density of 1.0108 g/ml at 26.5°C, and had an electrolytic conductivity of 1.93×10^{-5} S cm^{-1} at 20°C. It was stored under N_2 in flasks sealed with wax.

ESTIMATED ERROR:

Soly: precision probably 1% at best (compilers).

Temp: precision ± 0.05 K (authors).

REFERENCES:

1. Dirkse, T.P.; Briscoe, H.T. *Metal Ind.* 1938, *36*, 284.

COMPONENTS:

(1) Neodymium iodide; NdI_3; [13813-24-6]

(2) Morpholine; C_4H_9NO; [110-91-8]

ORIGINAL MEASUREMENTS:

Moeller, T.; Zimmerman, P.A.

J. Am. Chem. Soc. 1953, 75, 3940-3.

VARIABLES:

T/K = 303.15

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of NdI_3 in morpholine at 30°C was given as

0.500 g/100 solvent

The corresponding molality calculated by the compiler is

9.52×10^{-3} mol kg^{-1}

Solid phase composition not determined.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The isothermal method was used. Reaction mixtures were sealed in 25 x 200 mm test tubes and thermostated for one week at 30 ± 0.05°C with frequent agitation. The density of the supernatant liquid was determined pycnometrically (but not reported in the source paper), and the neodymium content determined by precipitating the hydrous hydroxide from a measured aliquot by adding excess water, igniting to the oxide and weighing.

All anhydrous substances were handled in a dry box through which a current of nitrogen was passed. The nitrogen was freed of CO_2 and moisture by passage through concentrated sulfuric acid, soda lime, and Drierite. All solutions were prepared in the dry box and were sealed before being removed.

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 was converted to the iodide by high temperature reaction with NH_4I. Unreacted NH_4I was removed by heating in N_2 and then in vacuo. The oxide labeled Nd-28 from University stocks contained traces of other rare earth metals. Morpholine was purified as in (1), and had a density of 0.9863 g/ml at 27°C, and an electrolytic conductivity of 3.368×10^{-8} S cm^{-1}. The solvent was stored under nitrogen in glass stoppered flasks sealed with wax.

ESTIMATED ERROR:

Soly: precision probably 1% at best (compilers).

Temp: precision $\pm$ 0.05 K (authors).

REFERENCES:

1. Dermer, V.H.; Dermer, O.C. *J. Am. Chem. Soc.* 1937, 59, 1148.

COMPONENTS:

(1) Neodymium iodide; NdI_3; [13813-24-6]

(2) N,N-Dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Moeller, T.; Galasyn, V.

J. Inorg. Nucl. Chem. 1960, *12*, 259-65.

VARIABLES:

T/K = 298.15

PREPARED BY:

M. Salomon

EXPERIMENTAL VALUES:

The solubility of NdI_3 in $HCON(CH_3)_2$ at 25°C was reported as

$$657.1 \text{ g dm}^{-3}$$

and as

$$0.5921 \text{ mol dm}^{-3}$$

The solid phase is the solvate $NdI_3.8HCON(CH_3)_2$. The melting point (sealed tube method) of this solvate given as 91.5 - 94.5°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Authors state that solubilities were determined by analysis of aliquots after equilibration at 25 ± 0.025°C, and that techniques were generally similar to those described in (1).

The rare earth content was determined by complexometric titration with EDTA at 60°C. Iodide was determined by the Volhard method, and carbon, hydrogen, and nitrogen by usual microanalytical techniques.

REFERENCES:

1. Moeller, T.; Cullen, G.W. *J. Inorg. Nucl. Chem.* 1959, *10*, 148.
2. Watt, G.W.; Gentile, P.S.; Helvenston, E.P. *J. Am. Chem. Soc.* 1955, *77*, 2752.
3. Biltz, H.; Biltz, W. *Laboratory Methods of Inorganic Chemistry (2nd Edition)*. John Wiley. N.Y. 1928.
4. Leader, G.R.; Gormley, J.F. *J. Am. Chem. Soc.* 1951, *73*, 5731.
5. Thomas, A.B.; Rochow, E.G. *J. Am. Chem. Soc.* 1957, *79*, 1843.

SOURCE AND PURITY OF MATERIALS:

The initial material was the rare earth oxide of 99.9+% purity. Iodides were prepd by two methods. 1. Acetyl iodide method (2) where the hydrated acetate is treated with acetyl iodide in benzene. Acetyl iodide prepd as in (3). 2. The iodide was prepd by metathesis by reaction of the hydrated $NdCl_3$ with KI in DMF followed by addition of benzene and distillation of the benzene-water azeotrope.

For both preparations the solvate $NdI_3.8DMF$ was recrystallized from DMF by addition of ether.

The solvent, DMF, was prepared as in (4,5), and its electrolytic conductance was $\geq 3.7 \times 10^{-7}$ S cm^{-1} at 25°C.

ESTIMATED ERROR:

Soly: precision around ± 0.1% (compiler).

Temp: precision ± 0.025 K (authors).

COMPONENTS:

(1) Samarium fluoride; SmF_3; [13765-26-7]

(2) Alcohols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			SmF_3 solubility[a,b] mass %	mol kg^{-1}
methanol	CH_4O;	[67-56-1]	0.01_5	7.2×10^{-4}
ethanol	C_2H_6O;	[64-17-5]	0.02	1×10^{-3}

[a] Molalities calculated by the compilers.

[b] Solid phases were dried in a desiccator over P_4O_{10} and the Sm:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of SmF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Sm(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Sm determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator.

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $SmF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310° for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Samarium fluoride; SmF_3; [13765-24-7]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen. GDR. 1980.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			SmF_3 solubility mass %	SmF_3 solubility mol/100g sln	solid phase Sm:F:solvent ratio
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	0.02	9.7×10^{-5}	1:3.02:0.06
1-(chloromethoxy) butane;	$C_5H_{11}ClO$;	[2351-69-1]	0.02	9.7×10^{-5}	1:3.00:0.16

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the Sm:F:H_2O ratio was 1:3.04:0.46.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen.* 1978, *2*, 85.

COMPONENTS:

(1) Samarium fluoride; SmF_3; [13765-24-7]

(2) Dimethylsulfoxide; C_2H_6OS; [67-68-5]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of SmF_3 in $(CH_3)_2SO$ at room temperature was given as

0.04 mass %

The corresponding molality calculated by the compiler is

$$1.9 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Sm:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of SmF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Sm(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Sm determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $SmF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Samarium fluoride; SmF_3; [13765-24-7] (2) Pyridine; C_6H_5N; [110-86-1]	Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES: Room Temperature	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of SmF_3 in pyridine at room temperature was reported to be

0.10 mass %

The corresponding molality calculated by the compiler is

4.8×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{11} and the Sm:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of SmF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Sm(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Sm determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $SmF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Samarium fluoride; SmF_3; [13765-24-7]

(2) Acidic nitrosyl fluoride; NOF.3HF; [14947-17-2]

ORIGINAL MEASUREMENTS:

Galkin, N.P.; Shishkov, Yu.D.
Khomyakov, V.I.

Radiokhimiya 1978, *20*, 136-41;
Soviet Radiochem. (*Engl. Transl.*).
1978, *20*, 109-13.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of SmF_3 in acidic nitrosyl fluoride at room temperature was reported to be

0.05 mass %

The molality calculated by the compiler is

2.4×10^{-3} mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solute-solvent mixture was placed in a Teflon vessel and mechanically agitated at room temperature for 10 h. The reaction mixture was allowed to settle for 24 h and the supernatant saturated solution was analyzed for the Sm content. An aliquot was evaporated to dryness under vacuum at 100-150°C, and the dry residue dissolved and analyzed (the method of analysis not specified).

Presumably the solid phase is the anhydrous salt (compiler).

SOURCE AND PURITY OF MATERIALS:

SmF_3 was at least 99% pure.

NOF.3HF prepared by saturation of liquid HF with NOF, and was distilled twice at 95°C before use. The melting point of acidic nitrosyl fluoride was 3.8°C.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Samarium chloride; $SmCl_3$; [10361-82-7] (2) Hexachloro-1,3-butadiene; C_4Cl_6; [87-68-3]	Shevtsova, Z.N.; Korshunov, B.G.; Safonov, V.V.; Kogan, L.M.; Gudkova, V.I. *Zh. Neorg. Khim.* 1968, *13*, 3096-9: *Russ. J. Inorg. Chem. (Engl. Transl.)* 1968, *13*, 1596-8.
VARIABLES:	**PREPARED BY:**
Temperature	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition, densities, viscosities and refractive indices of saturated solutions.

	solubility[a]					
t/°C	mass %	mol kg^{-1}	d/g cm^{-3}	η/P	n_D^{20}	nature of the solid phase
25	0.024	0.0093_5	1.6875	0.0386	1.5552	$SmCl_3.3.5H_2O$
50	0.035	0.00136	1.649	0.0311	1.5557	$SmCl_3.3.5H_2O$
75	0.054	0.00210	1.613	0.0249	1.5548	$SmCl_3.3.5H_2O$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Depending on temp, equil was established after 12 d at 25°C, 10 d at 50°C, and 7 d at 75°C.

Chloride was detd by the Volhard method, and samarium detd gravimetrically by precipitating as the oxalate and igniting to the oxide. Samarium was also detd by titration with Trilon B with Xylene Orange indicator.

The composition of the solid phase was established by chemical analysis, and confirmed by X-ray analysis.

Samples of the solid phases were also studied thermographically after removal of excess solvent by washing with absolute ethyl ether which is claimed not to change the composition of hydrate.

At 120°C, partial hydrolysis takes place with formation of SmOCl.

SOURCE AND PURITY OF MATERIALS:

$SmCl_3.6H_2O$ prepd by dissolving Sm_2O_3 in HCl, evaporating and cooling, and then recrystallized and dried in a desiccator. Sm_2O_3, 99.6% pure, contained oxide impurities of other rare earth metals, Fe (0.01%), Ca (0.01-0.05%), and Cu (0.01%). Analysis of the hexahydrate gave the following (in mass % units): Sm 41.51; Cl 28.90; H_2O 29.59.

Purified solvent (method not specified) had the following properties: d_4^{20} = 1.6807 g/ml, and n_D^{20} = 1.5543.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: accuracy ± 0.1 K (authors).

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Samarium chloride; $SmCl_3$; [10361-82-7] (2) Methanol; CH_4O; [67-56-1]	Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44-52. Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.
VARIABLES:	**PREPARED BY:**
T/K = 298.2	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubilities/mol kg^{-1} a	 b
25	3.33	3.37

a. Initial salt is the adduct $SmCl_3.4CH_3OH$. Equilibrated solid phase analyzed and found to be $SmCl_3.4CH_3OH$.

b. Solutions equilibrated with anhydrous $SmCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $SmCl_3.4CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Samarium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $SmCl_3.4CH_3OH$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, *7*, 224 (this is the reference to a paper by J. H. Freeman and M. L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as described in (3). The adduct $SmCl_3.4CH_3OH$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and crystallization from methanol.

Methanol was purified and dried by the Vogel method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, *55*, 44-52.

Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

	mean solubilities/mol kg^{-1}	
t/°C	a	b
25	1.97	2.03

a. Initial salt is the adduct $SmCl_3.3C_2H_5OH$. Equilibrated solid phase analyzed and found to be $SmCl_3.3C_2H_5OH$.

b. Solutions equilibrated with anhydrous $SmCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $SmCl_3.3C_2H_5OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Samarium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $SmCl_3.3C_2H_5O$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, 7, 224 (this is the reference to a paper by J. H. Freeman and M. L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $SmCl_3.3C_2H_6O$ prepared by dissolving the hydrate in a small excess of o-ethylformate followed by distillation and crystallization from ethanol.

Ethanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, N.N.; Sakharova, Yu.G.; Ezhova, T.A.; Izmailova, A.A.

Zh. Neorg. Khim. 1975, *20*, 1479-83; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 830-2.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $SmCl_3.6H_2O$ in 96.8 % C_2H_5OH[a]

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	28.97	28.89	29.29	28.87	29.00	1.195
30	29.49	29.66	29.59	29.63	29.59	1.152
40	30.42	30.47	30.61	30.75	30.56	1.206
50	31.93	31.96	31.83	31.88	31.90	1.284
60	34.82	34.58	34.79	34.65	34.73	1.458

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table were obtained after 3 h of equilibration, and the remaining two data points were obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

The hexahydrate melted at 145.5 - 146°C.

SOURCE AND PURITY OF MATERIALS:

$SmCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5, and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. Found (%) for Sm: 41.38, 41.25 (calcd 41.21). Found (%) for Cl: 29.10, 29.15 (calcd 29.18). 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn determined refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.8% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) 2-Propanol; C_3H_8O; [67-63-0]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, *55*, 44-52.

Pitteloud, M.N. *These.* Faculte des Sciences de l'Universite de Lausanne. 1971,

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubilities/mol kg^{-1}	
t/°C	a	b
25	0.23	0.23

a. Initial salt is the adduct $SmCl_3.3C_3H_7OH$. Equilibrated solid phase analyzed and found to be $SmCl_3.3C_3H_7OH$.

b. Solutions equilibrated with anhydrous $SmCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $SmCl_3.3C_3H_7OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Samarium determined by titration with $(NH_4)_3(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $SmCl_3.3C_3H_8O$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $SmCl_3.3C_3H_8O$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and trans-solvation of the methanol complex with 2-propanol.

Iso-propanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05K as in (1) (compilers).

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, *7*, 224 (this is the reference to a paper by J.H. Freeman and M.L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24* , 387 (see COMMENTS at left).

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Alcohols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$SmCl_3$ solubility[a] mass %	mol kg^{-1}	nature of the solid phase
1,2-ethanediol;	$C_2H_6O_2$;	[107-21-1]	32.9	1.91	$SmCl_3.3C_2H_6O_2$
1-propanol;	C_3H_8O;	[71-23-8]	30.5	1.71	$SmCl_3.C_3H_8O$
2-propen-1-ol[b];	C_3H_6O;	[107-18-6]	38.0	2.39	$SmCl_3.C_3H_6O$

[a] Molalities calculated by the compilers.

[b] In the original paper the solvent was specified simply as C_3H_5OH. Upon request, the author kindly identified the solvent as allyl alcohol.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Only the nature of the solid phase was reported. Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) 2-Methoxyethanol (methyl cellosolve); $C_3H_8O_2$; [109-86-4]

ORIGINAL MEASUREMENTS:

McCarty, C.N.
Master of Science Thesis. The University of Illinois. Urbana, IL, USA. 1933: this work was also cited by

Hopkins, B.S.; Audrieth, L.F.
Trans. Electrochem. Soc. 1934, *66*, 134-42.

VARIABLES:

Temperature: T/K = 273 - 323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Sm_2O_3 [a] g/25 cc	$SmCl_3$ [b] g/dm^3	$SmCl_3$ [b] mol/dm^3
0	0.2873	16.92	0.0659
10	0.5210	30.68	0.1195
20	0.7238	42.62	0.1660
30	1.0236	60.28	0.2348
40	1.2827	75.54	0.2942
50	1.4415	84.89	0.3306

[a] Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $SmCl_3$ from preparations 1 and 2.

[b] Recalculated by the compilers using 1977 IUPAC recommended atomic weights.

Equilibrated solid phase not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in bottles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and were sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber bung was fitted into the upper end of the tubing. After equilibration, the solutions were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet, two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

ESTIMATED ERROR:

Soly: precision probably within 3 % (compilers).

Temp: precision ± 0.2 K (author).

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 week and then distilled. A middle portion (fraction not specified) was retained and stored in a stoppered flask: b.p. 123°C. Sm salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide (no details) and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystallized salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extraction of the chloride was carried out with HCl satd ether, and the resulting chloride heated at 60°C first in a stream of dry HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 and by passing the resulting HCl through H_2SO_4 drying towers.

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) 2-Ethoxyethanol (ethyl cellosolve); $C_4H_{10}O_2$; [110-80-5]

ORIGINAL MEASUREMENTS:

McCarty, C.N.
Master of Science Thesis. The University of Illinois. Urbana, IL, USA. 1933: this work is also cited by

Hopkins, B.S.; Audrieth, L.F.
Trans. Electrochem. Soc. 1934, *66*, 135-42.

VARIABLES:

Temperature: T/K = 273 - 323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Sm_2O_3 [a] g/25 cc	$SmCl_3$ [b] g/dm^3	$SmCl_3$ [b] mol/dm^3
0	0.1556	9.16	0.0357
10	0.3307	19.47	0.0758
20	0.5666	33.37	0.1300
30	0.7720	45.46	0.1771
40	0.9873	58.14	0.2264
50	1.3540	79.74	0.3106

[a] Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $SmCl_3$ from preparations 1 and 2.

[b] Recalculated by the compilers using 1977 IUPAC recommended atomic weights.

Equilibrated solid phase not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in bottles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and were sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber bung was fitted into the upper end of the tubing. After equilibration, the solutions were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet, two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

ESTIMATED ERROR:

Soly: precision probably within 3 % (compilers).

Temp: precision ± 0.2 K (author).

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 week and then distilled. A middle portion (fraction not specified) was retained and stored in a stoppered flask: b.p. 134°C. Sm salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide (no details) and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystallized salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extracting the chloride was carried out with HCl satd ether, and the resulting chloride heated at 60°C first in a stream of dry HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 and by passing the resulting HCl through H_2SO_4 drying towers.

COMPONENTS: (1) Samarium chloride; $SmCl_3$; [10361-82-7] (2) Alkoxy-ethanols	**ORIGINAL MEASUREMENTS:** Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: T/K = 298	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] mol kg^{-1}	nature of the solid phase
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	5.7	0.24	$SmCl_3.nC_3H_8O_2$ (n=2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	26.3	1.39	$SmCl_3.2C_4H_{10}O_2$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the authors the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Diethyl ether (ethyl ether); $C_4H_{10}O$; [60-29-7]

ORIGINAL MEASUREMENTS:

Dzhuraev, Kh. Sh.; Mirsaidov, U.; Kurbanbekov, A.; Rakhimova, A.

Dokl. Akad. Nauk Tadzh. SSR 1976, *19*, 32-4.

VARIABLES:

T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $SmCl_3$ in diethyl ether at 20°C was reported to be

$$7.4 \times 10^{-3} \text{ mass } \%.$$

The corresponding molality calculated by the compiler is

$$2.84 \times 10^{-4} \text{ mol kg}^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrum was attained within 24 h and it was verified by constancy in the Sm concentration. The saturated solution and the equilibrated solid phase were analyzed. Sm determined by complexometric titration using urotropine buffer and methyl-thymol blue indicator. Chloride determined by titration with $AgNO_3$. The solid phase corresponded to $SmCl_3 \cdot 0.5Et_2O$ (the etherate was dried under vacuum at 40°C prior to analysis).

SOURCE AND PURITY OF MATERIALS:

Anhydrous $SmCl_3$ prepared by the ethanol solvate method (no details given).

Ethyl ether was dried with Na and distilled from $LiAlH_4$ before use.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U.
Z. Chem. 1968, *8*, 472-3.

Kirmse, E.M.
Tr. II Vses. Konf. po Teor. Rastvorov. 1971, 200-6.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$SmCl_3$ solubility[a,b] mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.45	0.018
1,3-dioxolane;	$C_3H_6O_2$;	[646-06-0]	2.6	0.104
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.07	0.0027

[a] Molalities calculated by the compilers.

[b] Nature of solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Sm was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:	**ORIGINAL MEASUREMENTS:**
(1) Samarium chloride; $SmCl_3$; [10361-82-7] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	Rossmanith, K.; Auer-Welsbach, C. *Monatsh. Chem.* 1965, *96*, 602-5.
VARIABLES:	**PREPARED BY:**
Room temperature: T/K about 293	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $SmCl_3$ in tetrahydrofuran at room temperature (about 20°C) was reported as

1.49 g/100 ml solution

The solid phase is

$SmCl_3 \cdot 1.98C_4H_8O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor for 60-80 hours at room temperature. Samarium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. For the solid phase analysis, the solvent was determined by difference.

Anhydrous substances were handled in a dry box through which was passed a current of dry and CO_2-free nitrogen.

SOURCE AND PURITY OF MATERIALS:

Sources and purities not specified. $SmCl_3$ prepared by reaction of the oxide at high temperatures with an excess of NH_4Cl followed by heating the product in a current of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.

Zh. Neorg. Khim. 1975, *20*, 908-14; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 508-11.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solution

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg^{-1} (compiler)	density/g cm^{-3}
33.4	1.30	436.5	1.70	1.95	1.30

The solid phase is $NdCl_3$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd slns prepared isothermally with magnetic stirring. Equil was attained after 25-30 d. The sln was centrifuged and an aliquot for analysis taken and added to methanol and pptd with aq NH_3. The pptd $Sm(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous and only anhydr $SmCl_3$ was found. All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complextion between TBP and $SmCl_3$ in solution. Additional studies with unsaturated solutions (IR spectra, viscosity, molar conductivities) are discussed in the source paper.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $SmCl_3$ prepd by chlorination of Sm_2O_3 with CCl_4 vapor (1,2). Source and purity of materials not given. Sm was analyzed gravimetrically, and Cl by Volhard's method. Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* 1964, *9*, 1427.
2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* 1965, *38*, 1160.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Samarium chloride; $SmCl_3$; [10361-82-7] (2) Amines	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$SmCl_3$ solubility[a] mass %	mol kg^{-1}
2-propanamine;	iso-C_3H_9N;	[75-31-0]	15.2	0.698
2-propen-1-amine[b];	C_3H_7N;	[107-11-9]	5.6	0.225

[a] Molalities calculated by the compilers.

[b] The original paper simply specifies the solvent as $C_3H_5NH_2$, and upon request the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Samarium chloride; $SmCl_3$; [10361-82-7] (2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]	Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A. *Zh. Neorg. Khim.* 1977, *22*, 1761-6; *Russ. J. Inorg. Chem. Engl. Transl.* 1977, *22*, 955-8.
VARIABLES:	**PREPARED BY:**
Room Temperature: T/K = 298 ± 3	T. Mioduski

EXPERIMENTAL VALUES:

At room temperature, the solubility was reported as

$$0.126 \text{ mol dm}^{-3}$$

The solid phase is the solvate $SmCl_3.3HMPT$

It is apparent that the authors determined the solubility of $SmCl_3$ several times, but only the above single value was reported.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Anhydrous $SmCl_3$ placed in a test-tube with the solvent in a dry box and shaken at room temperature. Aliquots removed periodically to test for equilibrium. The analysis was carried out by complexometric titration (no details) and by radioassay using ^{170}Tm. "The results obtained by the two methods agreed." Analysis showed the solid phase to be $SmCl_3.3HMPT$. Analysis of the solid phase performed by complexometric titration for Sm and by the Volhard method for Cl: HMPT was obtained by difference.

SOURCE AND PURITY OF MATERIALS:

$SmCl_3$ prepared similar to (1): a 6:1 molar mixture of NH_4Cl + Sm_2O_3 was heated to 200 to 400°C in a stream of inert gas. Excess NH_4Cl sublimed, and the remaining $SmCl_3$ contained less than 3% oxide impurity.

Hexamethylphosphorotriamide (HMPT) was purified as in (2).

ESTIMATED ERROR:

Soly: ± 0.001 mol dm^{-3} at the 95% confidence level.

Temp: 25 ± 3°C.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.
2. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

T/K = 293

Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	Sm_2O_3 solubility[a] moles Sm dm^{-3}
1:250	0.035	0.10
1:100	0.085	0.18
1:50	0.17	0.28
1:25	0.33	0.25
1:15	0.59	0.11
1:10	0.78	0.10

[a] This is also the solubility of $SmCl_3$ in the $SnCl_4$-$POCl_3$ mixtures because the oxide is quantitatively converted to the chloride according to

$$Sm_2O_3 + 6POCl_3 = 2SmCl_3 + 3P_2O_3Cl_4$$

Thus the equilibrated solutions should actually be considered to be a four component system containing $SnCl_4$, $SmCl_3$, $P_2O_3Cl_4$ and $POCl_3$ (the compiler assumes $P_2O_3Cl_4$ is soluble).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Sm_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20° to 2 hours.

Sm was determined by colorimetric analysis, and in some cases by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 of "the first sort" was heated at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS:

(1) Samarium bromide; $SmBr_3$; [13759-87-0]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

The solubility of $SmBr_3$ in 1,2-dimethoxyethane at 25°C was reported as

0.8 mass %

The corresponding molality calculated by the compiler is

0.021 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phase not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Samarium bromide; $SmBr_3$; [13759-87-0]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Dressler, H.

Z. Chem. 1975, *15*, 239-40.

VARIABLES:

Room Temperature (293-298 K)

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$SmBr_3$ solubility[a] mass %	 mol kg^{-1}
1-methoxybutane;	$C_5H_{12}O$;	[628-28-4]	3.0	0.079
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	2.2	0.058
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	7.3	0.202
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	13.5	0.400
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	7.6	0.211
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	4.6	0.124

[a] Molalities calculated by the compilers. Compositions of the solid phases were not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated (at room temperature) until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}. Pr was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Samarium bromide; $SmBr_3$; [13759-87-0]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. 1966, *97*, 1357-64.

VARIABLES:

Room temperature: T/K about 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $SmBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.55 g/100 ml solution

The solid phase is

$SmBr_3.3.5C_4H_8O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor for 60-80 hours at room temperature. Samarium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. For the solid phase analysis, the solvent was determined by difference.

Anhydrous substances were handled in a dry box through which was passed a current of dry and CO_2-free nitrogen.

SOURCE AND PURITY OF MATERIALS:

Sources and purities not specified. $SmBr_3$ prepared by reaction of the oxide at high temperatures with an excess of NH_4Br followed by heating the product in a current of dry nitrogen, and then in vacuum to removed unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Samarium bromide; $SmBr_3$; [13759-87-0]

(2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U.
Z. Chem. <u>1968</u>, *8*, 472-3;

Kirmse, E.M.
Tr. II Vses. Konf. po Teor. Rastvorov. <u>1971</u>, 200-6.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $SmBr_3$ in p-dioxane at about 25°C was given as

1.3 mass %

The corresponding molality calculated by the compiler is

$0.034 \text{ mol kg}^{-1}$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Sm was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. <u>1962</u>, *24*, 387.

COMPONENTS:

(1) Samarium bromide; $SmBr_3$; [13759-87-0]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E. M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$SmBr_3$ solubility[a] mass %	mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	23.8	0.801
2-propanamine;	iso-C_3H_9N;	[75-31-0]	33.1	1.26_8
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	38.5	1.60_5
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	23.8	0.801
di-2-butylamine;	(sec-$C_4H_9)_2NH$;	[626-23-3]	0.03	8×10^{-4}

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Samarium iodide; SmI_3; [13813-25-7] (2) N,N-Dimethylformamide; C_3H_7NO; [68-12-2]	Moeller, T.; Galasyn, V. *J. Inorg. Nucl. Chem.* 1960, *12*, 259-65.
VARIABLES:	**PREPARED BY:**
T/K = 298.15	M. Salomon

EXPERIMENTAL VALUES:

The solubility of SmI_3 in $HCON(CH_3)_2$ at 25°C was reported as

$$520.7 \text{ g dm}^{-3}$$

and as

$$0.4666 \text{ mol dm}^{-3}$$

The solid phase is the solvate $SmI_3.8HCON(CH_3)_2$. The melting point (sealed tube method) of this solvate given as 97.5 - 100.0°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Authors state that solubilities were determined by analysis of aliquots after equilibration at 25 ± 0.025°C, and that techniques were generally similar to those described in (1).

The rare earth content was determined by complexometric titration with EDTA at 60°C. Iodide was determined by the Volhard method, and carbon, hydrogen, and nitrogen by usual microanalytical techniques.

SOURCE AND PURITY OF MATERIALS:

The initial material was the rare earth oxide of 99.9+% purity. Iodides were prepd by two methods. 1. Acetyl iodide method (2) where the hydrated acetate is treated with acetyl iodide in benzene. Acetyl iodide prepd as in (3). 2. The iodide was prepd by metathesis by reaction of the hydrated $SmCl_3$ with KI in DMF followed by addition of benzene and distillation of the benzene-water azeotrope.

For both preparations the solvate $SmI_3.8DMF$ was recrystallized from DMF by addition of ether.

The solvent, DMF , was prepared as in (4,5), and its electrolytic conductance was $\geq 3.7 \times 10^{-7}$ S cm^{-1} at 25°C.

ESTIMATED ERROR:

Soly: precision around ± 0.1% (compiler).

Temp: precision ± 0.025 K (authors).

REFERENCES:

1. Moeller, T.; Cullen, G.W. *J. Inorg. Nucl. Chem.* 1959, *10*, 148.
2. Watt, G.W.; Gentile, P.S.; Helvenston, E.P. *J. Am. Chem. Soc.* 1955, *77*, 2752.
3. Biltz, H.; Biltz, W. *Laboratory Methods of Inorganic Chemistry (2nd Edition)*. John Wiley. N.Y. 1928.
4. Leader, G.R.; Gormley, J.F. *J. Am. Chem. Soc.* 1951, *73*, 5731.
5. Thomas, A.B.; Rochow, E.G. *J. Am. Chem. Soc.* 1957, *79*, 1843.

COMPONENTS:

(1) Europium fluoride; EuF_3; [13765-25-8]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen. GDR. 1980.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			EuF_3 solubility mass %	EuF_3 solubility mol/100g sln	solid phase Eu:F:solvent ratio
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	0.03	1.44×10^{-4}	1:2.83:0.06
1-(chloromethoxy)butane;	$C_5H_{11}ClO$;	[2351-69-1]	0.02	9.6×10^{-5}	1:3.15:0.16

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the Eu:F:H_2O ratio was 1:3.01:0.23.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen.* 1978, *2*, 85.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Europium fluoride; EuF_3; [13765-25-8] (2) Dimethylsulfoxide; C_2H_6OS; [67-68-5]	Kirmse, E.M. *Wiss, Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES:	PREPARED BY:
Room Temperature	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of EuF_3 in $(CH_3)_2SO$ at room temperature was given as

0.03 mass %

The corresponding molality calculated by the compiler is

1.4×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and the Eu:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of EuF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Eu(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Eu determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Eu_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $EuF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Europium fluoride; EuF_3; [13765-25-8]

(2) Pyridine; C_6H_5N; [110-86-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of EuF_3 in pyridine at room temperature was reported to be

0.15 mass %

The corresponding molality calculated by the compiler is

7.2×10^{-3} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{11} and the Eu:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of EuF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Eu(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Eu determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Eu_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $EuF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS: (1) Europium chloride; $EuCl_3$; [10025-76-0] (2) Alcohols	**ORIGINAL MEASUREMENTS:** Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: T/K - 298	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$EuCl_3$ solubility[a] mass %	mole kg^{-1}
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	4.4	0.18[b]
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	20.8	1.02[c]
1-propanol;	C_3H_8O;	[71-23-8]	33.5	1.95[d]

[a] Molalities calculated by the compilers.

[b] Solid phase is $EuCl_3.2.95C_3H_8O_2$.

[c] Solid phase is $EuCl_3.2C_4H_{10}O_2$.

[d] Solid phase is $EuCl_3.C_3H_8O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Europium chloride; $EuCl_3$; [10025-76-0]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, N.N.; Sakharova, Yu.G.; Ezhova, T.A.; Izmailova, A.A.

Zh. Neorg. Khim. 1975, *20*, 1479-83; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 830-2.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $EuCl_3.6H_2O$ in 96.8 % $C_2H_5OH^a$

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	29.23	29.18	29.20	29.30	29.23	1.127
30	29.39	29.32	29.22	29.17	29.27	1.129
40	29.47	29.58	29.86	29.94	29.71	1.154
50	30.95	31.13	30.99	31.16	31.05	1.229
60	33.33	33.14	32.87	32.70	33.01	1.345

[a] It is not clearly stated whether the mixture is 96.8 mass % of 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table were obtained after 3 h of equilibration, and the remaining two data points were obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

The hexahydrate melted at 151.2 - 151.7°C.

SOURCE AND PURITY OF MATERIALS:

$EuCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method.
Found (%) for Eu: 41.53, 41.40 (calcd 41.48).
Found (%) for Cl; 29.27, 22.10 (calcd 29.07).
96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn determined refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.8% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Europium chloride; $EuCl_3$; [10025-76-0]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.

Z. Chem. 1967, 7, 281.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $EuCl_3$ in 1,2-diethoxyethane at 25°C was reported to be

0.25 mass %

The corresponding molality calculated by the compiler is

$0.0097 \text{ mol kg}^{-1}$

The composition of the solid phase was given in terms of the Eu:Cl:ether ratio as

1:2.91:1.10

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over P_2O_5.

Eu was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, 24, 387.

COMPONENTS:

(1) Europium chloride; $EuCl_3$; [10025-76-0]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Dressler, H.

Z. Chem. 1975, *15*, 239-40.

VARIABLES:

Room Temperature: (293-298 K)

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] mol kg^{-1}
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	0.4	0.016
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	0.5	0.019
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	0.13	0.0050
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	0.5	0.019
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	1.1	0.043

[a] Molalities calculated by the compilers. Composition of solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated (at room temperature) until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}. Eu was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Europium chloride; $EuCl_3$; [10025-76-0]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. *Z. Chem.* 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room Temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a,b]	
			mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.6	0.023
1,3-dioxolane;	$C_3H_6O_2$;	[646-06-0]	3.5	0.14
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.07	0.0027

[a] Molalities calculated by the compilers.

[b] Nature of the solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Eu was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Europium chloride; $EuCl_3$; [10025-76-0]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.

Zh. Neorg. Khim. 1975, *20*, 908-14; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1975, *20*, 508-11.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solutions[a,b]

mass %	mol/kg sln[c]	g dm^{-3}[c]	mol dm^{-3}[c]	mol kg^{-1}	density/g cm^{-3}[c]
35.2	1.35	455	1.76	2.10	1.30

[a] Solid phase is $EuCl_3$.

[b] Molality calculated by the compilers from the experimental solubility of 35.2 mass %.

[c] It is implied that these data also correspond to the saturated solution. However the molality calculated from these data is 2.08 mol kg^{-1}.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and precipitated with aq NH_3. The pptd $Eu(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous, and only the anhydrous $EuCl_3$ was found.

All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complexation between TBP and $EuCl_3$ in solution.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $EuCl_3$ prepared by chlorination of Eu_2O_3 with CCl_4 vapor (1,2). Source and purity of materials not given. Eu was analyzed gravimetrically, and Cl by Volhard's method.

Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* 1964, *9*, 1427.
2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* 1965, *38*, 1160.

COMPONENTS:

(1) Europium chloride; $EuCl_3$; [10025-76-0]

(2) Amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	mol kg^{-1}
2-propanamine;	iso-C_3H_9N;	[75-31-0]	11.6	0.508
2-propen-1-amine[b];	C_3H_7N;	[107-11-9]	6.0	0.247
1-butanamine;	$C_4H_{11}N$;	[109-73-9]	25.5	1.325

[a] Molalities calculated by the compilers.

[b] The original paper simply specifies the solvent as $C_3H_5NH_2$, and upon request the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Europium chloride; $EuCl_3$; [10025-76-0]

(2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1761-6; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

Starting with the solvate $EuCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.121 \pm 0.001 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature (25 ± 3°C) until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $EuCl_3.3C_6H_{18}N_3OP$. The solvate was analyzed for metal content by Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$EuCl_3.3C_6H_{18}N_3OP$ prepd by dissolving the hydrate in $C_3H_{18}N_3OP$ and heating to 140-145° C for 5 m. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over P_2O_5 in a stream of dry nitrogen. Yield was about 90%.

The solvent was purified as described in (1).

ESTIMATED ERROR:

Soly: precision ± 0.001 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3K.

REFERENCES:

1. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Europium chloride; $EuCl_3$; [10025-76-0]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

$SnCl_4$ concentration

T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	solubility of Eu_2O_3[a] moles Eu dm^{-3}
0	0	0.005
1:250	0.035	0.11
1:100	0.085	0.22
1:50	0.17	0.28
1:25	0.33	0.25
1:15	0.59	0.048
1:10	0.78	0.11

[a] This is also the solubility of $EuCl_3$ since the oxide is quantitatively converted to the chloride according to

$$Eu_2O_3 + 6POCl_3 = 2EuCl_3 + 3P_2O_3Cl_4$$

Assuming $P_2O_3Cl_4$ to be soluble, the equilibrated solutions would then constitute a four component mixture.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Eu_2O_3 were placed in sealed ampoules and rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20°C to 2 hours.

Eu was determined by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

The solubility of $EuCl_3$ in pure $POCl_3$ is small, but in the presence of $SnCl_4$ the solubility increases due to complexation:

$$2EuCl_3 + 3SnCl_4 = Eu_2(SnCl_6)_3$$

SOURCE AND PURITY OF MATERIALS:

Eu_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS: (1) Europium (II) bromide; $EuBr_2$; [13780-48-8] (2) Alkyl ethers	**ORIGINAL MEASUREMENTS:** Kirmse, E.M.; Dressler, H. *Z. Chem.* 1975, *15*, 239-40.
VARIABLES: Room Temperature (293-298 K)	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$EuBr_2$ solubility[a] mass %	mol kg^{-1}
1-methoxybutane;	$C_5H_{12}O$;	[628-28-4]	0.15	0.0048
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	0.9	0.029
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	1.5	0.049
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	1.4	0.046
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	0.04	0.0013
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	0.7	0.023

[a] Molalities calculated by the compilers. Compositions of the solid phases were not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated (at room temperature) until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}. Eu was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Europium bromide; $EuBr_3$; [13759-88-1]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. 1966, 97, 1357-64.

VARIABLES:

Room Temperature: T/K = 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $EuBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.45 g per 100 ml of solution (0.011$_5$ mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Europium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $EuBr_3.3.5C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $EuBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Europium bromide; $EuBr_3$; [13759-88-1]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	11.0	0.316
2-propanamine;	iso-C_3H_9N;	[75-31-0]	0.08	0.0020
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	0.11	0.0028

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Gadolinium fluoride; GdF_3; [13765-26-9]

(2) Dimethylsulfoxide; C_2H_6OS; [67-68-5]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of GdF_3 in $(CH_3)_2SO$ at room temperature was given as

$$0.03_5 \text{ mass \%}$$

The corresponding molality calculated by the compiler is

$$0.0016 \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Gd:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of GdF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temp for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Gd(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Gd determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Gd_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $GdF_3 . 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gadolinium chloride; $GdCl_3$; [10138-52-0] (2) Methanol; CH_4O; [67-56-1]	Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44-52. Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.
VARIABLES:	**PREPARED BY:**
T/K = 298.2	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubility[a] mol kg^{-1}
25	4.21

[a] Initial salt was the adduct $GdCl_3.4CH_3OH$. Analysis of the equilibrated solid phase yielded $GdCl_3.4.1CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Gadolinium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $GdCl_3.4CH_3OH$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubility is a mean of 2-4 determinations.

SOURCE AND PURITY OF MATERIALS:

Gd_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The adduct $GdCl_3.4CH_3OH$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and crystallization from methanol.

Methanol was purified and dried by the Vogel method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, 47, 14.
2. Flatt, R. *Chimia* 1952, 6, 62.

COMPONENTS:	EVALUATOR:
(1) Gadolinium chloride; $GdCl_3$; [10138-52-0] (2) Ethanol; C_2H_6O; [64-17-5]	Tomasz Mioduski Institute of Nuclear Research Warsaw, Poland

CRITICAL EVALUATION:

The solubility of $GdCl_3$ in ethanol has been reported only at 298.2 K in two publications (1,2), and both publication report an identical solubility of 2.43 mol kg^{-1}. However Kirmse reported the solid phase to be the monosolvate $GdCl_3 \cdot C_2H_5OH$ whereas Merbach et al. reported $GdCl_3 \cdot 4C_2H_5OH$ as the equilibrated solid phase.

The initial salt used by Kirmse was anhydrous $GdCl_3$ prepared by the method of Taylor and Carter (3). Merbach et al. used $GdCl_3 \cdot 3C_2H_5OH$ as the initial solvate, and which was prepared by their transsolvation method. Since Merbach et al. confirmed the composition of the equilibrated solid phase as the tetrasolvate by ^1H NMR and X-ray diffraction, and since Kirmse does not report details on analysis of the solid phase, we conclude that the tetrasolvate is probably the stable solid phase at 298.2 K.

Merbach et al. state that their result is a mean of 2-4 determinations, and a precision of ± 0.5 % was estimated by the compilers. Although Kirmse did not report any experimental details, her reported solubility is probably the mean of at least two determinations. In consideration of the agreement in solubility and the precision estimated by the compilers, the *tentative* solubility of $GdCl_3$ in ethanol at 298.2 K is 2.43 mol kg^{-1} and the accuracy is estimated to be ± 0.04 mol kg^{-1}. The stable solid phase at 298.2 K appears to be the tetrasolvate $GdCl_3 \cdot 4C_2H_5OH$.

REFERENCES

1. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200.
2. (a) Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44.
   (b) Pitteloud, M.N. *These.* Faculte des Sciences de l'Universite de Lausanne. 1971.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44-52.

Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971,

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubility[a] mol kg^{-1}
25	2.43

[a] Initial salt was the adduct $GdCl_3.3C_2H_5OH$. Analysis of the equilibrated solid phase yielded $GdCl_3.4.1C_2H_5OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Gadolinium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $GdCl_3.4C_2H_5O$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubility is a means of 2-4 determinations.

SOURCE AND PURITY OF MATERIALS:

Gd_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The adduct $GdCl_3.3C_2H_6O$ prepared by dissolving the hydrate in a small excess of o-ethylformate followed by distillation and crystallization from ethanol.

Ethanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, 47, 14.
2. Flatt, R. *Chimia* 1952, 6, 62.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Alcohols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			mass %	mole kg^{-1}	nature of the solid phase
ethanol;	C_2H_6O;	[64-17-5]	39.0	2.43	$GdCl_3.C_2H_6O$
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	4.0	0.16	$GdCl_3.nC_3H_8O_2$ (n = 2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	15.2	0.680	$GdCl_3.C_4H_{10}O_2$
2-propen-1-ol;[b]	C_3H_6O;	[107-18-6]	30.0	1.63	$GdCl_3.C_3H_6O$

[a] Molalities calculated by the compilers.

[b] The source paper reports the solvent as C_3H_5OH. Upon request, the author kindly specified the solvent as allyl alcohol.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the authors, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, N.N.; Sakharova, Yu.G.; Ezhova, T.A.; Izmailova, A.A.

Zh. Inorg. Khim. 1975, *20*, 1479-83; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1975, *20*, 830-2.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $GdCl_3.6H_2O$ in 96.8 % C_2H_5OH[a]

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	30.48	30.67	30.67	30.45	30.56	1.184
30	29.86	29.70	29.82	29.78	29.79	1.142
40	29.88	29.99	29.87	30.05	29.94	1.150
50	30.46	30.43	30.58	30.35	30.45	1.178
60	31.80	31.63	31.63	31.73	31.69	1.248

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table were obtained after 3 h of equilibration, and the remaining two data points were obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

The hexahydrate melted at 156.8 - 157.8°C.

SOURCE AND PURITY OF MATERIALS:

$GdCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. Found (%) for Gd: 42.39, 42.30 (calcd 42.31). Found (%) for Cl: 28.69, 28.54 (calcd 28.65). 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn determined refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.8% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) 2-Propanol; C_3H_8O; [67-63-0]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P.
Helv. Chim. Acta 1972, 55, 44-52.

Pitteloud, M.N.
These. Faculte des Sciences de
l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubility[a] mol kg^{-1}
25	0.32

[a] Initial salt was the adduct $GdCl_3.3$iso-PrOH. Analysis of the equilibrated solid phase yielded $GdCl_3$.x-iso-PrOH where x = 3.0 - 3.1.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Gadolinium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $GdCl_3.3C_3H_8O$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubility is a mean of 2-4 determinations.

SOURCE AND PURITY OF MATERIALS:

Gd_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The adduct $GdCl_3.3C_3H_8O$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and trans-solvation of the methanol complex with 2-propanol.

Iso-propanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.

2. Flatt, R. *Chimia* 1952, *6*, 62.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) 2-Methoxyethanol (methyl cellosolve); $C_3H_8O_2$; [109-86-4]

ORIGINAL MEASUREMENTS:

McCarty, C.N.

Master of Science Thesis.
University of Illinois. Urbana, IL, USA.
<u>1933</u>[1].

VARIABLES:

T/K = 273-323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Gd_2O_3[a] g/25 cc	$GdCl_3$[b] g/dm^3	$GdCl_3$[b] mol/dm^3
0	0.6345	36.91	0.1400
10	0.8111	47.19	0.1790
20	1.0495	61.06	0.2316
30	1.1564	67.27	0.2552
40	1.3202	76.80	0.2914
50	1.4464	84.15	0.3192

[a] Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $GdCl_3$ from preparations 1 and 2.

[b] Recalculated by the compilers using 1977 IUPAC recommended atomic masses.

The equilibrated solid phase not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in botttles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and were sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles. A rubber stopper was fitted into the upper end of the tubing. After equilibration, the slns were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet, two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

ESTIMATED ERROR:

Soly: precision probably within 3% (compilers).

Temp: precision ± 0.2 K (author).

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 week and then distd. A middle portion (fraction not specified) was retained and stored in a stoppered flask: b.p. 123°C. Gd salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide, and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystd salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extraction of the chloride was carried out with HCl satd ether, and the resulting chloride heated at 60°C first in a stream of dry HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 followed by passage through H_2SO_4 drying towers.

REFERENCES:

1. Hopkins, B.S.; Audrieth, L.F. *Trans. Electrochem. Soc.* <u>1934</u>, *66*, 135.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) 2-Ethoxyethanol (ethyl cellosolve); $C_4H_{10}O_2$; [110-80-5]

ORIGINAL MEASUREMENTS:

McCarty, C.N.

Master of Science Thesis.
The University of Illinois. Urbana, IL, USA. 1933[1]

VARIABLES:

T/K = 273-323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Gd_2O_3[a] g/25 cc	$GdCl_3$[b] g/dm^3	$GdCl_3$[b] mol/dm^3
0	0.2282	13.28	0.0504
10	0.3714	21.61	0.0820
20	0.6029	35.07	0.1331
30	0.9456	55.01	0.2087
40	1.1362	66.10	0.2507
50	1.2442	72.38	0.2746

[a] Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $GdCl_3$ from preparations 1 and 2.

[b] Recalculated by the compilers using 1977 IUPAC recommended atomic masses.

The equilibrated solid phase not analyzed

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in bottles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and were sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber stopper was fitted into the upper end of the tubing. After equilibration, the solutions were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet, two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

ESTIMATED ERROR:

Soly: precision probably within 3% (compilers).

Temp: precision ± 0.2 K (author).

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 week and then dist. A middle portion (fraction not specified) was retained and stored in a stoppered flask: b.p. 134°C. Gd salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide, and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystd salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extraction of the chloride was carried out with HCl satd ether, and the resulting chloride heated at 60°C first in a stream of dry HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 followed by passage through H_2SO_4 drying towers.

REFERENCES:

1. Hopkins, B.S.; Audrieth, L.F. *Trans. Electrochem. Soc.* 1934, *66*, 135.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Diethyl ether (ethyl ether); $C_4H_{10}O$; [60-29-7]

ORIGINAL MEASUREMENTS:

Dzhuraev, Kh. Sh.; Mirsaidov, U.; Kurbanbekov, A.; Rakhimova, A.

Dokl. Akad. Nauk Tadzh. SSR 1976, *19*, 32-4.

VARIABLES:

T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $GdCl_3$ in diethyl ether at 20°C was reported to be

0.028 mass %

The corresponding molality calculated by the compiler is

1.06×10^{-3} mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrium was attained within 24 h and it was verified by constancy in the Gd concentration. The saturated solution and the equilibrated solid phase were analyzed. Gd determined by complexometric titration in presence of urotropine buffer and methyl-thymol blue indicator. Chloride determined by titration with $AgNO_3$. The solid phase corresponded to $GdCl_3.0.5Et_2O$ (the etherate was dried under vacuum at 40°C prior to analysis).

SOURCE AND PURITY OF MATERIALS:

Anhydrous $GdCl_3$ prepared by the ethanol solvate method (no details given). Ethyl ether was dried with Na and distilled from $LiAlH_4$ before use.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) 1,2-Diethyoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.

Z. Chem. 1967, *7*, 281.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $GdCl_3$ in 1,2-diethoxyethane at 25°C was reported to be

0.33 mass %

The corresponding molality calculated by the compiler is

0.0126 mol kg^{-1}

The composition of the solid phase was given in terms of the Gd:Cl:ether ratio as

1:2.91:0.99

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over P_2O_5.

Gd was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:

No estimate possible.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U.
Z. Chem. 1968, *8*, 472-3.

Kirmse, E.M.
Tr. II Vses. Konf. po Teor. Rastvorov. 1971, 200-6.

VARIABLES:

Room Temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$GdCl_3$ solubility[a,b] mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.55	0.021
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	0.08	0.0030
1,4-dioxane;	C_4H_8O;	[123-91-1]	0.1	0.0038

[a] Molalities calculated by the compilers.

[b] Nature of solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Gd was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.; Auer-Welsbach, C.

Monatsh. Chem. 1965, *96*, 602-5.

VARIABLES:

Room Temperature: T/K about 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $GdCl_3$ in tetrahydrofuran at 20°C (room temperature) was reported to be

1.91 g per 100 ml of solution

(0.0725 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Gadolinium was determined by the oxalate method, and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $GdCl_3.2.07C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $GdCl_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Cl followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin. S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.

Zh. Neorg. Khim. 1975, *20*, 908-14; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 508-11.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solution

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg^{-1} (compiler)	density/g cm^3
37.8	1.44	494.0	1.88	2.31	1.33

The solid phase is $GdCl_3$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and pptd with aq NH_3. The pptd $Gd(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous and only anhydrous $GdCl_3$ was found. All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complexation between TBP and $GdCl_3$ in solution. Additional IR spectra studies with unsaturated solutions are discussed in the source paper.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $GdCl_3$ prepared by chlorination of Gd_2O_3 with CCl_4 vapor (1,2). Source and purity of materials not given. Gd was analyzed gravimetrically and Cl by Volhard's method. Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* 1964, *9*, 1427.

2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* 1965, *38*, 1160.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$GdCl_3$ solubility[a] mass %	mol kg^{-1}
1-propanamine;	C_3H_9N;	[107-10-8]	32.4	1.82
2-propanamine;	iso-C_3H_9N;	[75-31-0]	21.0	1.01
2-propen-1-amine;[b]	C_3H_7N;	[107-11-9]	14.6	0.649
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	23.0	1.13
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	18.7	0.873

[a] Molalities calculated by the compilers.

[b] The original paper simply specifies the solvent as $C_3H_5NH_2$, and upon request, the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. <u>1977</u>, *22*, 1761-6; *Russ. J. Inorg. Chem. (Engl. Transl.)* <u>1977</u>, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

Starting with the solvate $GdCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.125 \pm 0.002 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at room temperature (25 ± 3°C).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature (25 ± 3°C) until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $GdCl_3.3C_6H_{18}N_3OP$.

The solvate was analyzed for metal content by complexometric titrn, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$GdCl_3.3C_6H_{18}N_3OP$ prepd by dissolving the hydrate in $C_3H_{18}N_3OP$ and heating to 140-150° C for 5 m. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over P_2O_5 in a stream of dry nitrogen. Yield was about 90%. The solvent was purified as described in (1).

ESTIMATED ERROR:

Soly: precision ± 0.002 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3K.

REFERENCES:

1. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* <u>1975</u>, *11*, 163.

COMPONENTS:

(1) Gadolinium chloride; $GdCl_3$; [10138-52-0]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, 45, 1176-8.

VARIABLES:

T/K = 293

Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	Gd_2O_3 solubility^{a,b} moles Gd dm^{-3}
0	0	0.015
1:250	0.035	0.11
1:100	0.085	0.27
1:50	0.17	0.29 (0.16)
1:25	0.33	0.079
1:15	0.59	0.042
1:10	0.78	0.11

[a] Solutions preheated to 220°C. Value in parenthesis corresponds to preheating at 120°C.

[b] This is also the solubility of $GdCl_3$ in the $SnCl_4$-$POCl_3$ mixture because the oxide is quantitatively converted to the chloride according to

$$Gd_2O_3 + 6POCl_3 = 2GdCl_3 + 3P_2O_3Cl_4$$

Thus the equilibrated solutions should actually be considered to be a four component system containing $SnCl_4$, $GdCl_3$, $P_2O_3Cl_4$ and $POCl_3$ (the compiler assumes $P_2O_3Cl_4$ is soluble).

Authors state that the solubility of $GdCl_3$ is enhanced by complex formation according to

$$2GdCl_3 + 3SnCl_4 = Gd_2(SnCl_6)_3$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Gd_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20°C to 2 hours.

Gd was determined by colorimetric analysis, and in some cases by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Gd_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS: (1) Gadolinium bromide; $GdBr_3$; [13818-75-2] (2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]	**ORIGINAL MEASUREMENTS:** Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: T/K = 298	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $GdBr_3$ in 1,2-diethoxyethane at 25°C was reported as

0.9 mass %.

The corresponding molality calculated by the compiler is

0.023 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phase not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS: (1) Gadolinium bromide; $GdBr_3$; [13818-75-2] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	**ORIGINAL MEASUREMENTS:** Rossmanith, K. *Monatsh. Chem.* 1966. 97, 1357-64.
VARIABLES: Room Temperature: T/K = 294-296	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $GdBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.38 g per 100 ml of solution (0.0096 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Gadolinium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $GdBr_3.3.5C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $GdBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS: (1) Gadolinium bromide; $GdBr_3$; [13818-75-2] (2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]	**ORIGINAL MEASUREMENTS:** Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472-3. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.
VARIABLES: Room Temperature: T/K around 298	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $GdBr_3$ in p-dioxane at around 25°C was given as

$$0.9_5 \text{ mass \%}$$

The corresponding molality calculated by the compiler is

$$0.024 \text{ mol kg}^{-1}$$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Gd was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Gadolinium bromide; $GdBr_3$; [13818-75-2]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$GdBr_3$ solubility[a] mass %	mol kg^{-1}
2-propanamine;	iso-C_3H_9N;	[75-31-0]	26.3	0.899
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	27.8	0.970
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	26.25	0.897
di-2-butylamine;	(sec-$C_4H_9)_2NH$;	[626-23-3]	0.25	0.0063

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS: (1) Gadolinium iodide; GdI_3; [13572-98-0] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	ORIGINAL MEASUREMENTS: Kachkimbaeva, S.A.; Chalova, E.P.; Bleshinskii, S.V. *Khim. Kompleks. Soedin. Redk. Soputstvuyushchikh Elem.* 1970, 122-6.
VARIABLES: T/K = 293	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

The solubility of GdI_3 in tetrahydrofuran at 20°C was reported to be

$$2.67 \text{ g dm}^{-3}$$

$$(0.00496 \text{ mol dm}^{-3}, \text{compiler})$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were equilibrated isothermally with constant agitation. Solid and liquid phases separated by decantation, and in some cases by centrifuging. Gd determined by the oxalate method. I determined by titration with an $AgNO_3$ solution (the Volhard method).

SOURCE AND PURITY OF MATERIALS:

GdI_3 prepared by heating cp grade iodine with an excess powdered metal (Gd-0-Sort) in an ampoule at 1200°C. The iodide formed sublimated from the hot to the cold part of the ampoule. The product was analyzed for Gd and I contents, and found to contain GdI_2. The I/Gd ratio was 2.67. Cp grade solvent (GDR) bp=65.6°C, dried with NaOH and Na and distilled from metallic sodium.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Gadolinium iodide; GdI_3; [13572-98-0]

(2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. *Z. Chem.* 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room Temperature: T/K around 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of GdI_3 in p-dioxane at about 25°C was given as

0.4 mass %

The corresponding molality calculated by the compiler is

0.0075 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Gd was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gadolinium iodide; GdI_3; [13572-98-0] (2) Alkyl amines	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	4.3	0.084
2-propanamine;	iso-C_3H_9N;	[75-31-0]	14.8	0.323
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	17.0	0.381
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	11.5	0.242

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, 24, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gadolinium iodide; GdI_3; [13572-98-0] (2) N,N-Dimethylformamide; C_3H_7NO; [68-12-2]	Moeller, T.; Galasyn, V. *J. Inorg. Nucl. Chem.* 1960, *12*, 259-65.
VARIABLES:	**PREPARED BY:**
T/K = 298.15	M. Salomon

EXPERIMENTAL VALUES:

The solubility of GdI_3 in $HCON(CH_3)_2$ at 25°C was reported as

$$451.0 \quad g\ dm^{-3}$$

and as

$$0.4018 \quad mol\ dm^{-3}$$

The solid phase is the solvate $GdI_3.8HCON(CH_3)_2$. The melting point (sealed tube method) of this solvate given as 102.0 - 104.0°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Authors state that solubilities were determined by analysis of aliquots after equilibration at 25 ± 0.025°C, and that techniques were generally similar to those described in (1).

The rare earth content was determined by complexometric titration with EDTA at 60°C. Iodide was determined by the Volhard method, and carbon, hydrogen, and nitrogen by usual microanalytical techniques.

SOURCE AND PURITY OF MATERIALS:

The initial material was the rare earth oxide of 99.9+% purity. Iodides were prepd by two methods. 1. Acetyl iodide method (2) where the hydrated acetate is treated with acetyl iodide in benzene. Acetyl iodide prepd as in (3). 2. The iodide was prepd by metathesis by reaction of the hydrated $GdCl_3$ with KI in DMF followed by addition of benzene and distillation of the benzene-water azeotrope.

For both preparations the solvate $GdI_3.8DMF$ and its electrolytic conductance was $\geq 3.7 \times 10^{-7}$ S cm^{-1} at 25°C.

ESTIMATED ERROR:

Soly: precision around ± 0.1% (compiler).

Temp: precision ± 0.025 K (authors).

REFERENCES:

1. Moeller, T.; Cullen, G.W. *J. Inorg. Nucl. Chem.* 1959, *10*, 148.
2. Watt, G.W.; Gentile, P.S.; Helvenston, E.P. *J. Am. Chem. Soc.* 1955, *77*, 2752.
3. Biltz, H.; Biltz, W. *Laboratory Methods of Inorganic Chemistry (2nd Edition)*. John Wiley. N.Y. 1928.
4. Leader, G.R.; Gormley, J.F. *J. Am. Chem. Soc.* 1951, *73*, 5731.
5. Thomas, A.B.; Rochow, E.G. *J. Am. Chem. Soc.* 1957, *79*, 1843.

COMPONENTS: (1) Terbium fluoride; TbF_3; [13708-63-9] (2) Ethers	**ORIGINAL MEASUREMENTS:** Dressler, H. *Dissertationschrift.* Paed. Inst. Koethen. GDR. 1980.
VARIABLES: Room Temperature	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] 10^4 mol/100 g sln
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	0.03	1.39[a]
1-(chloromethoxy)butane;	$C_5H_{11}ClO$;	[2351-69-1]	0.03	1.39[b]

[a] Solid phase. Tb:F:ether:H_2O ratio found to be 1:2.82:0.11:0.34.

[b] Solid phase. Tb:F:ether ratio found to be 1:2.99:0.26.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the Tb:F:H_2O ratio was 1:3.00:0.12.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen.* 1978, 2, 85.

COMPONENTS: (1) Terbium fluoride; TbF_3; [13708-63-9] (2) Dimethylsulfoxide; C_2H_6OS; [67-68-5]	**ORIGINAL MEASUREMENTS:** Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES: Room Temperature	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of TbF_3 in $(CH_3)_2SO$ at room temperature was given as

0.01 mass %.

The corresponding molality calculated by the compiler is

4.7×10^{-4} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and the Tb:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of TbF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Tb(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Tb determined several times by complexometric titration with potentiometric endpoint detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Tb_4O_7 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $TbF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Terbium chloride; $TbCl_3$; [10042-88-3]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, Yu.G.; Ezhova, T.A.

Zh. Neorg. Khim. 1976, *21*, 551-4; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1976, *21*, 296-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $TbCl_3.6H_2O$ in 96.8 % $C_2H_5OH^a$

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1c}
20	30.75	30.68	30.50	30.61	30.63	1.183
30	29.94	30.03	30.32	30.06	30.08	1.152
40	30.31	30.49	30.33	29.94	30.27	1.163
50	30.84	30.81	30.77	30.92	30.83	1.194
60	32.92	33.03	32.65	32.63	32.80	1.307

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$TbCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 158.0 - 158.8°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.9 % (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Terbium chloride; $TbCl_3$; [10042-88-3]

(2) Alkoxy-ethanols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$TbCl_3$ solubility[a] mass %	mol kg^{-1}	nature of the solid phase
2-methoxyethanol;	C_3H_8O;	[109-86-4]	3.8	0.15	$TbCl_3.nC_3H_8O_2$ (n = 2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	11.9	0.509	$TbCl_3.2C_4H_{10}O_2$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the authors the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS: (1) Terbium chloride; $TbCl_3$; [10042-88-3] (2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]	**ORIGINAL MEASUREMENTS:** Kirmse, E.M.; Zwietasch, K.J. *Z. Chem.* <u>1967</u>, *7*, 281.
VARIABLES: T/K = 298	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $TbCl_3$ in 1,2-diethoxyethane at 25°C was reported to be

0.22 mass %

The corresponding molality calculated by the compiler is

$0.0083 \text{ mol kg}^{-1}$

The composition of the solid phase was given in terms of the Tb:Cl:ether ratio as

1:2.93:1.37

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over P_2O_5.

Tb was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* <u>1962</u>, *24*, 387.

COMPONENTS:

(1) Terbium chloride; $TbCl_3$; [10042-88-3]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J. Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$TbCl_3$ solubility[a,b] mass %	 mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.6	0.023
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.3	0.011

[a] Molalities calculated by the compilers.

[b] Nature of solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Tb was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS: (1) Terbium chloride; $TbCl_3$; [10042-88-3] (2) Alkyl ethers	**ORIGINAL MEASUREMENTS:** Kirmse, E.M.; Dressler, H. *Z. Chem.* 1975, *15*, 239-40.
VARIABLES: Room Temperature (293-298 K)	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] mol kg^{-1}
1-methoxybutane;	$C_5H_{12}O$;	[628-28-4]	0.23	0.0087
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	0.7	0.027
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	3.3	0.129
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	1.5	0.057
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	2.75	0.107
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	1.9	0.073

[a] Molalities calculated by the compilers. Compositions of solid phases were not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated (at room temperature) until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}. Tb was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Terbium chloride; $TbCl_3$; [10042-88-3]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin. S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.

Zh. Neorg. Khim. 1975, *20*, 908-14; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 508-11.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solutions

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg-1 (compiler)	density/g cm^{-3}
38.5	1.45	525.0	1.98	2.36	1.36

The solid phase is $TbCl_3$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and precipitated with aq NH_3. The pptd $Tb(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous, and only the anhydrous $TbCl_3$ was found.

All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complexation between TBP and $TbCl_3$ in solution.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $TbCl_3$ prepared by chlorination of the oxide with CCl_4 vapor (1,2). Source and purity of materials not given. Tb was analyzed gravimetrically, and Cl by Volhard's method.

Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* 1964, *9*, 1427.
2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* 1965, *38*, 1160.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Terbium chloride; $TbCl_3$; [10042-88-3] (2) Alkyl amines	Kirmse, E. M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	PREPARED BY:
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$TbCl_3$ solubility[a] mass %	mol kg^{-1}
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	14.6	0.644
di-2-butylamine;	(sec-$C_4H_9)_2NH$;	[626-23-3]	0.15	0.0057

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Terbium chloride; $TbCl_3$; [10042-88-3]

(3) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1761-6; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with the solvate $TbCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.128 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $TbCl_3.3C_6H_{18}N_3OP$.

The solvate was analyzed for metal content by complexometric titration, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate were also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$TbCl_3.3C_6H_{18}N_3OP$ prepared by dissolving the hydrate in the solvent and heating to 140-145°C for 5 m. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over P_2O_5 in a stream of dry nitrogen. Yield was about 90%.

The solvent was purified as described in (1).

ESTIMATED ERROR:

Soly: precision ± 0.007 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3K.

REFERENCES:

1. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Terbium chloride; $TbCl_3$; [10042-88-3]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

T/K = 293

Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	solubility/moles Tb dm^{-3} [a,b]	
		Tb_2O_3	Tb_4O_7
1:250	0.035	0.069	----
1:100	0.085	0.30	0.056
1:50	0.17	0.15 (0.13)	0.18 (0.015)
1:25	0.33	0.067	0.059
1:15	0.59	0.051	0.040
1:10	0.78	0.018	0.047

[a] Solutions preheated to 220°C. Values in parenthesis correspond to preheating at 120°C.

[b] This is also the solubility of $TbCl_3$ in the $SnCl_4$-$POCl_3$ mixture because the oxide is quantitatively converted to the chloride according to

$$Tb_2O_3 + 6POCl_3 = 2TbCl_3 + 3P_2O_3Cl_4$$

Authors state that the solubility of $TbCl_3$ is enhanced by complex formation according to

$$2TbCl_3 + 3SnCl_4 = Tb_2(SnCl_6)_3$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Tb_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 220°C lowered the equilibration time at 20°C to 2 hours.

Tb was determined by colorimetric analysis or the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Tb_4O_7 of "the first sort" was reduced to Tb_2O_3 with hydrogen at 950°C.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2 K (compiler).

REFERENCES:

COMPONENTS:

(1) Terbium bromide; $TbBr_3$
[14456-47-4]

(2) Tetrahydrofuran; C_4H_8O;
[109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. 1966, 97, 1357-64.

VARIABLES:

Room Temperature: T/K = 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $TbBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.29 g per 100 ml of solution

(0.0073 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Terbium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $TbBr_3.4C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $TbBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Terbium bromide; $TbBr_3$;
[14456-47-4]

(2) 1,4-Dioxane; $C_4H_8O_2$;
[123-91-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U.
Z. Chem. 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room Temperature: T/K around 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $TbBr_3$ in p-dioxane at about 25°C was given as

0.9 mass %

The corresponding molality calculated by the compiler is

$0.023 \text{ mol kg}^{-1}$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Tb was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
   J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Terbium bromide; $TbBr_3$; [14456-47-4] (2) Alkyl amines	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	PREPARED BY:
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$TbBr_3$ solubility[a] mass %	mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	17.0	0.514
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	31.25	1.140
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	34.7	1.33_3

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Terbium iodide; TbI_3; [13813-40-6]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Kachkimbaeva, S.A.; Chalova, E.P.; Bleshinskii, S.V.

Khim. Kompleks. Soedin. Redk. Soputstvuyushchickh Elem. 1970, 122-6.

VARIABLES:

T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of TbI_3 in tetrahydrofuran at 20°C was reported to be

8.63 g dm^{-3} (0.0160 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were equilibrated isothermally by agitation. The phases were separated by decantation, and in some cases by centrifuging. Tb determined by the oxalate method. Iodide determined by titration with an $AgNO_3$ solution (the Volhard method).

SOURCE AND PURITY OF MATERIALS:

TbI_3 prepared by heating "cp" grade I_2 with excess metallic Tb (RETU-1101-68, Tb-1) in a sealed ampoule at 1200°C. TbI_3 collected by sublimation at the cold part of the ampoule. The product was analyzed for Tb and I, and presumably found to be sufficiently pure.

"C.p." grade tetrahydrofuran (GDR), b.p. = 65.6°C was treated with NaOH and Na, and then distilled from metallic sodium.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Dysprosium fluoride; DyF_3; [13569-80-7]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of DyF_3 in methanol at room temperature was reported to be

$$0.01_5 \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$6.8 \times 10^{-4} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Dy:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of DyF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Dy(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Dy determined several times by complexometric titration with potentiometric endpoint detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Dy_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $DyF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS: (1) Dysprosium fluoride; DyF_3; [13569-80-7] (2) Alkyl ethers	**ORIGINAL MEASUREMENTS:** Dressler, H. *Dissertationschrift.* Paed. Inst. Koethen. GDR. 1980.
VARIABLES: Room temperature	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			DyF_3 solubility mass %	 mol/100 g sln	solid phase Dy:F:solvent ratio
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	0.03	1.37×10^{-4}	1:2.93:0.10
1-(chloromethoxy)butane;	$C_5H_{11}ClO$;	[2351-69-1]	0.01	4.6×10^{-5}	1:2.81:0.11

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the Dy:F:H_2O ratio was 1:3.02:0.30.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen.* 1978, *2*, 85.

COMPONENTS:

(1) Dysprosium fluoride; DyF_3; [13569-80-7]

(2) Tributyl phosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of DyF_3 in $[CH_3(CH_2)_3]_3P(O)$ at room temperature was given as

$$0.01 \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$4.6 \times 10^{-4} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Dy:F ratio determined to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of DyF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Dy(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Dy determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Dy_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $DyF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS: (1) Dysprosium fluoride; DyF_3; [13569-80-7] (2) Dimethylsulfoxide; C_2H_6OS; [67-68-5]	**ORIGINAL MEASUREMENTS:** Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES: Room Temperature	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of DyF_3 in $(CH_3)_2SO$ at room temperature was given as

0.01 mass %

The corresponding molality calculated by the compiler is

4.6×10^{-4} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and the Dy:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of DyF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Dy(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Dy determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Dy_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $DyF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Dysprosium fluoride; DyF_3; [13569-80-7]

(2) Pyridine; C_5H_5N; [110-86-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of DyF_3 in pyridine at room temperature was given as

0.03 mass %

The corresponding molality calculated by the compiler is

$$1.4 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Dy:F ratio determined to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of DyF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Dy(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Dy determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Dy_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $DyF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, Yu.G.; Ezhova, T.A.

Zh. Neorg. Khim. <u>1976</u>, *21*, 551-4; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) <u>1976</u>, *21*, 296-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $DyCl_3.6H_2O$ in 96.8 % C_2H_5OH[a]

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	33.46	33.38	33.64	33.24	33.43	1.332
30	32.60	32.40	32.27	32.26	32.38	1.270
40	31.91	31.98	32.36	32.12	32.09	1.254
50	33.67	33.39	33.58	33.10	33.43	1.332
60	35.04	34.66	34.87	35.10	34.91	1.423

[a] It is not clearly stated whetehr the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$DyCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 161.8 - 163.1°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.9 % (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Dysprosium chloride; $DyCl_3$; [10025-74-8] (2) Alkoxy-ethanols	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

VARIABLES:	PREPARED BY:
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$DyCl_3$ solubility[a] mass %	mol kg^{-1}	nature of the solid phase
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	3.9	0.151	$DyCl_3.nC_3H_8O_2$ (n = 2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	9.4	0.386	$DyCl_3.2C_4H_{10}O_2$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, 24, 387.

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(2) Diethyl ether (ethyl ether); $C_4H_{10}O$; [60-29-7]

ORIGINAL MEASUREMENTS:

Dzhuraev, Kh. Sh.; Mirsaidov, U.; Kurbanbekov, A.; Rakhimova, A.

Dokl. Akad. Nauk Tadzh. SSR 1976, *19*, 32-4.

VARIABLES:

T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $DyCl_3$ in diethyl ether at 20°C was reported to be

$$3.2 \times 10^{-2} \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$1.19 \times 10^{-3} \text{ mol kg}^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrium was attained within 24 h and it was verified by constancy in the Dy concentration. The saturated solution and the equilibrated solid phase were analyzed. Dy determined by complexometric titration using urotropine buffuer and methyl-thymol blue indicator. Chloride determined by titration with $AgNO_3$ solution. The solid phase corresponded to $DyCl_3.Et_2O$ (the etherate was dried under vacuum at 40°C prior to analysis).

SOURCE AND PURITY OF MATERIALS:

Anhydrous $DyCl_3$ prepared by the ethanol solvate method (no details given).

Ethyl ether was dried with Na and distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.

Z. Chem. 1967, *7*, 281.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $DyCl_3$ in 1,2-diethoxyethane at 25°C was reported to be

0.25 mass %

The corresponding molality calculated by the compiler is

$0.0093 \text{ mol kg}^{-1}$

The composition of the solid phase was given in terms of the Dy:Cl:ether ratio as

1:2.91:1.70

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over P_2O_5.

Dy was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U.
Z. Chem. 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

VARIABLES:

Room Temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$DyCl_3$ solubility[a,b] mass %	 mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.6	0.022
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	0.08	0.003
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.4	0.015

[a] Molalities calculated by the compilers.

[b] Nature of solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Dy was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
   J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Dysprosium chloride; $DyCl_3$; [10025-74-8] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	Rossmanith, K.; Auer-Welsbach, C. *Monatsh. Chem.* 1965, *96*, 602-5.
VARIABLES:	**PREPARED BY:**
Room Temperature: T/K about 293	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $DyCl_3$ in tetrahydrofuran at 20°C (room temperature) was reported to be

0.645 g per 100 ml of solution

(0.00240 mol dm^{-3}, compiler)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Dysprosium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $DyCl_3.3.45C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $DyCl_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Cl followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.

Zh. Neorg. Khim. 1975, *20*, 908-14; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1975, *20*, 508-11.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solutions

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg^{-1} (compiler)	density/g cm^{-3}
39.2	1.45	534.4	1.98	2.40	1.36

The solid phase is $DyCl_3$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and precipitated with aq NH_3. The pptd $Dy(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous and only the anhydrous $DyCl_3$ was found.

All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complexation between TBP and $DyCl_3$ in solution.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $DyCl_3$ prepared by chlorination of the oxide with CCl_4 vapor (1,2). Source and purity of materials not given. Dy was analyzed gravimetrically, and Cl by Volhard's method.

Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* 1964, *9*, 1427.
2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* 1965, *38*, 1160.

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(2) Amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] mol kg^{-1}
2-propanamine;	iso-C_3H_9N;	[75-31-0]	24.5	1.207
2-propen-1-amine;[b]	C_3H_7N;	[107-11-9]	18.0	0.816
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	21.2	1.001
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	15.4	0.677

[a] Molalities calculated by the compilers.

[b] The original paper specifies the solvent as $C_3H_5NH_2$, and upon request the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1761-6; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with the solvate $DyCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.109 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $DyCl_3 \cdot 3C_6H_{18}N_3OP$.

The solvate was analyzed for metal content by complexometric titration, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate were also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$DyCl_3.3C_6H_{18}N_3OP$ prepared by dissolving the hydrate in the solvent and heating to 140-145°C for 5 m. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over P_2O_5 in a stream of dry nitrogen. Yield was about 90%.

The solvent was purified as described in (1).

ESTIMATED ERROR:

Soly: precision ± 0.002 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov. E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

T/K = 293

Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio	$SnCl_4$ concentration	Dy_2O_3 solubility[a,b]
(by volume)	mol dm^{-3}	moles Dy dm^{-3}
1:100	0.085	0.022
1:50	0.17	0.023 (0.018)
1:25	0.33	0.034
1:15	0.59	0.023
1:10	0.78	0.032

[a] Solutions preheated to 220°C. Value in parenthesis corresponds to preheating at 120°C.

[b] This is also the solubility of $DyCl_3$ in the $SnCl_4$-$POCl_3$ mixture because the oxide is quantitatively converted to the chloride according to

$$Dy_2O_3 + 6POCl_3 = 2DyCl_3 + 3P_2O_3Cl_4$$

Authors state that the solubility of $DyCl_3$ is enhanced by complex formation according to

$$2DyCl_3 + 3SnCl_4 = Dy_2(SnCl_6)_3$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Dy_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution and then rotated in an air thermostat at 20° C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 220°C lowered the equilibration time at 20°C to 2 hours.

Dy was determined by colorimetric analysis or by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Dy_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2 K (compiler).

REFERENCES:

COMPONENTS:

(1) Dysprosium bromide; $DyBr_3$; [14456-48-5]

(2) 1,2-Diethyoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $DyBr_3$ in 1,2-diethoxyethane at 25°C was given as

1.2 mass %

The corresponding value of the molality calculated by the compiler is

$0.0302 \text{ mol kg}^{-1}$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Dysprosium bromide; $DyBr_3$; [14456-48-5]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. 1966, *97*, 1357-64.

VARIABLES:

Room Temperature: T/K = 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $DyBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.26 g per 100 ml of solution (0.0065 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Dysprosium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $DyBr_3 \cdot 3.5C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $DyBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Dysprosium bromide; $DyBr_3$;
[14456-48-5]

(2) 1,4-Dioxane; $C_4H_8O_2$;
[123-91-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U.
Z. Chem. 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

VARIABLES:

Room Temperature: T/K around 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $DyBr_3$ in p-dioxane at about 25°C was given as

0.9_5 mass %

the corresponding molality calculated by the compiler is

0.0238 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Dy was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Dysprosium bromide; $DyBr_3$; [14456-48-5]

(2) 2-Propanamine; iso-C_3H_9N; [75-31-0]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $DyBr_3$ in 2-propanamine at 25°C was reported as

43.0 mass %

The corresponding molality calculated by the compiler is

$1.876 \text{ mol kg}^{-1}$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Dysprosium iodide; DyI_3; [15474-63-2]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water: H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Yastrebova, L.F.; Grigor, T.I.; Kuznetsova, C.P.; Stepin, B.D.

Zh. Neorg. Khim. 1981, *26*, 2238-9; *Russ. J. Inorg. Chem. (Engl. Transl.)*, 1981, *26*, 1203-4.

VARIABLES:

Composition at 273 K

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

solubility at 0°C

	$DyI_3.9H_2O$	DyI_3[a]		
solvent	mass %	mass %	mol kg^{-1}	solid phase
C_2H_5OH[b]	79.81	61.46	2.936	$DyI_3.9H_2O$
H_2O	88.86	68.43	3.991	"

[a] Results for the anhydrous salt calculated by the compilers.

[b] Authors' original results reported in terms of the solubility of the nonohydrate in the pure alcohol. Accounting for the waters of hydration, the compilers calculate that at equilibrium, the solvent contains 52.39 mass % alcohol and 47.61 mass % water.

COMMENTS AND/OR ADDITIONAL DATA:

In several instances in investigating the ternary systems, the initial nonohydrate was dehydrated in vacuum (3-4 mm Hg) at 30-40°C. Under these conditions the hexahydrate was produced after 40 hours. Howdver the authors state that in every instance the solid phase in the equilibrated solutions is the nonohydrate.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. No information was given on how equilibrium was ascertained. Aliquots of saturated solution were withdrawn and analyzed for the metal complexometrically, for iodide by a potentiometric volumetric argentometric method, and for water by the Karl Fischer method. The alcohol and water contents in the mixtures[c] were found by quantitative gas chromatography. Solid phase compositions were determined by Schreinemakers' method of residues.[c]

[c] These statements indicate that the authors studied the ternary system over a wide range of compositions. However no phase diagram was given, and the only numerical results reported are those given in the data table above. The phase diagram is stated to be similar to that for the NdI_3-H_2O-C_4H_9OH system (see the compilation for this system).

SOURCE AND PURITY OF MATERIALS:

The nonohydrate, $DyI_3.9H_2O$, was synthesized according to (1,2).

The alcohol was dried and purified by "recommended" methods.

The source and purity of water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Yakimova, Z.P.; Kuznetsova, G.P.; Ya Yastrebova, L.F.; Stepin, B.D. *Zh. Neorg. Khim.* 1977, *22*, 251.
2. Belousova, A.P.; Kuznetsova, G.P.; Rukk, N.S.; Stepin, B.D. *Zh. Neorg. Khim.* 1979, *24*, 1410.

COMPONENTS:

(1) Dysprosium iodide; DyI_3; [15474-63-2]

(2) 1-Butanol; $C_4H_{10}O$; [71-36-3]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Yastrebova, L.F.; Grigor, T.I.; Kuznetsova, G.P.; Stepin, B.D.

Zh. Neorg. Khim. <u>1981</u>, *26*, 2238-9; *Russ. J. Inorg. Chem.* (*Engl. Transl.*), <u>1981</u>, *26*, 1203-4.

VARIABLES:

Composition at 273 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility at 0°C

	$DyI_3.9H_2O$	DyI_3[a]		
solvent[b]	mass %	mass %	mol kg^{-1}	solid phase
n-C_4H_9OH	62.53	48.16	1.710	$DyI_3.9H_2O$
H_2O	88.86	68.43	3.991	"

[a] Results for the anhydrous salt calculated by the compilers.

[b] Authors' original results reported in terms of the solubility of the nonohydrate in the pure alcohol. Accounting for the waters of hydration, the compilers calculate that at equilibrium the solvent contains 72.27 mass % alcohol and 27.73 mass % water.

<u>COMMENTS AND/OR ADDITIONAL DATA</u>:

In several instances in investigating the tenary systems, the initial nonohydrate was dehydrated in vacuum (3-4 mm Hg) at 30-40°C. Under these conditions, the hexahydrate was produced after 40 hours. However the authors state that in every instance the solid phase in the equilibrated solutions is the nonohydrate.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. No information was given on how equilibrium was ascertained. Aliquots of saturated solution were withdrawn and analyzed for the metal complexometrically, for iodide by a potentiometric volumetric argentometric method, and for water by the Karl Fischer method. The alcohol and water contents in the mixtures[c] were found by quantitative gas chromatography. Solid phase compositions were determined by Schreinemakers' method of resudies.[c]

[c] These statements indicate that the authors studied the ternary system over a wide range of compositions. However no phase diagram was given, and the only numerical results reported are those given in the data table above. The phase diagram is stated to be similar to that for the $NdI_3 - H_2O - C_4H_9OH$ system (see the compilation for this system).

SOURCE AND PURITY OF MATERIALS:

The nonohydrate, $DyI_3.9H_2O$, was synthesized according to (1,2).

The alcohol was dried and purified by "recommended" methods.

The source and purity of water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Yakimova, Z.P.; Kuznetsova, G.P.; Yastrebova, L.F. Stepin, B.D. *Zh. Neorg. Khim.* <u>1977</u>, *22*, 251.
2. Belousova, A.P.; Kuznetsova, G.P.; Rukk, N.S.; Stepin, B.D. *Zh. Neorg. Khim.* <u>1979</u>, *24*, 1410.

COMPONENTS: (1) Dysprosium iodide; DyI_3; [15474-63-2] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	**ORIGINAL MEASUREMENTS:** Kachkimbaeva, S.A.; Chalova, E.P.; Bleshinskii, S.V. *Khim. Kompleks. Soedin. Redk. Soputstvuyushchikh Elem.* 1970, 122-6.
VARIABLES: T/K = 293	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of DyI_3 in tetrahydrofuran at 20°C was reported to be

$$3.553 \text{ g dm}^{-3}$$

$$(0.00654 \text{ mol dm}^{-3}, \text{ compiler})$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were equilibrated isothermally by agitation. The phases were separated by decantation, and in some cases by centrifuging. Dy determined by the oxalate method. I determined by titration with an $AgNO_3$ solution (the Volhard method).

SOURCE AND PURITY OF MATERIALS:

DyI_3 prepared by heating cp grade iodine with excess powered metal (Dy-0-Sort) in an ampoule at 1200°C. The iodide formed sublimated from the hot to the cold part of the ampoule. The product was analyzed for Dy and I contents. DyI_3 contained DyI_2 as found by titration with an iodine solution, and the I/Dy ratio was 2.78. C.p. grade tetrahydrofuran, (GDR) b.p. = 65.6°C, dried with NaOH and Na, and then distilled from metallic sodium.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Holmium fluoride; HoF_3; [13760-78-6]

(2) Alcohols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

			HoF_3 solubility[a,b]	
solvent			mass %	mol kg^{-1}
methanol	CH_4O;	[67-56-1]	0.01	4.5×10^{-4}
ethanol	C_2H_6O;	[64-17-5]	0.01	4.5×10^{-4}

[a] Molalities calculated by the compilers.

[b] Solid phases were dried in a desiccator over P_4O_{10} and the Ho:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of HoF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Ho(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Ho determined several times by complexometric titration with potentiometric endpoint detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator.

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Ho_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $HoF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvents were dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Holmium fluoride; HoF_3; [13760-78-6]

(2) Ethers

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen. GDR. 1980.

VARIABLES:

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility mass %	 mol/100 g sln
1-methoxydecane;	$n\text{-}C_{11}H_{24}O$;	[7289-52-3]	0.03[a]	1.35×10^{-4}
1-(chloromethoxy)butane;	$n\text{-}C_5H_{11}ClO$	[2351-69-1]	0.02[b]	9.0×10^{-5}

[a] In the solid phase the Ho:F:ether:H_2O ratio is 1:2.99:0.02:0.40

[b] In the solid phase the Ho:F:ether ratio is 1:2.89:0.06.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the Ho:F:H_2O ratio was 1:3.04:0.70.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen.* 1978, 2, 85.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Holmium fluoride; HoF_3; [13760-78-6] (2) Tributyl phosphate; $C_{12}H_{27}O_4P$; [126-73-8]	Kirmse, E.M. *Wiss. Hefte, Paed. Inst. Koethen* 1978, *2*, 85-90.
VARIABLES:	**PREPARED BY:**
Room temperature	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of HoF_3 in $[CH_3(CH_2)_3]_3P(O)$ at room temperature was given as

0.02 mass %

The corresponding molality calculated by the compiler is

$$9.0 \times 10^{-4} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Ho:F ratio determined to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of HoF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Ho(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Ho determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Ho_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $HoF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Holmium fluoride; HoF_3;
[13760-78-6]

(2) Dimethylsulfoxide; C_2H_6OS;
[67-68-5]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of HoF_3 in $(CH_3)_2SO$ at room temperature was given as

0.02 mass %

The corresponding molality calculated by the compiler is

9.0×10^{-4} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and the Ho:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of HoF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Ho(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Ho determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel detemrinations," or "at least two parallel determinations,"

SOURCE AND PURITY OF MATERIALS:

Ho_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $HoF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M.
Z. Chem. 1974, *14*, 484.

2. Schilbach, U.; Hetze, I.; Kirmse, E.M.
Chemia Analityczna 1975, *20*, 33.

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, Yu.G; Ezhova, T.A.

Zh. Neorg. Khim. 1976, *21*, 551-4; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1976, *21*, 296-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $HoCl_3.6H_2O$ in 96.8 % C_2H_5OH[a]

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	34.62	34.81	34.86	34.87	34.79	1.406
30	33.72	33.85	34.04	33.89	33.87	1.350
40	33.88	34.02	33.83	34.16	33.97	1.356
50	34.25	34.68	34.47	34.56	34.49	1.388
60	36.02	36.11	35.77	35.83	35.93	1.478

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$HoCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 163.4 - 164.5°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.9% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) Alkoxy-ethanols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$HoCl_3$ solubility[a] mass %	mol kg^{-1}	nature of the solid phase
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	3.2	0.122	$HoCl_3.nC_3H_8O_2$ (n = 2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	8.4	0.338	$HoCl_3.2C_4H_{10}O_2$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.

Z. Chem. 1967, *7*, 281.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $HoCl_3$ in 1,2-diethoxyethane at 25°C was reported to be

0.37 mass %

The corresponding molality calculated by the compiler is

$0.0137 \text{ mol kg}^{-1}$

The composition of the solid phase was given in terms of the Ho:Cl:ether ratio as

1:2.97:1.82

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over P_2O_5.

Ho was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) Alkyl ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Dressler, H.

Z. Chem. 1975, *15*, 239-40.

VARIABLES:

Room temperature: T/K = 293-298

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

solvent			$HoCl_3$ solubility[a] mass %	mol kg^{-1}
1-methoxypentane;[b]	$C_6H_{14}O$;	[628-80-8]	2.4[b]	0.091
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	1.0	0.037
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	1.55	0.058
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	1.8	0.068
1-methoxydecane;	$C_{11}H_{24}O$;	[7289-52-3]	2.5	0.095

[a] Molalities calculated by the compilers.

[b] Identical result for this system reported in (1)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were agitated at room temperature until the solutions were saturated. The anhydrous reagents were handled in a dry box containing P_2O_5.

Holmium was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations for each system.

SOURCE AND PURITY OF MATERIALS:

No information given.

ESTIMATED ERROR:

No information given.

REFERENCES:

1. Kirmse, E.M. *Tr. II. Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. *Z. Chem.* 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a,b] mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.6_5	0.024
di-n-propyl ether;	$C_6H_{14}O$;	[111-43-3]	0.1	0.004
1-ethoxybutane;	$C_6H_{14}O$;	[628-81-9]	0.01	0.0004
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	2.4	0.091
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.5_5	0.020

[a] Molalities calculated by the compilers.

[b] Nature of the solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Ho was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.; Auer-Welsbach, C.

Monatsh. Chem. 1965, *96*, 602-5.

VARIABLES:

Room Temperature: T/K about 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $HoCl_3$ in tetrahydrofuran at 20°C (room temperature) was reported to be

0.698 g per 100 ml of solution

(0.0257 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Holmium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $HoCl_3.3.33C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $HoCl_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Cl followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.

Zh. Neorg. Khim. <u>1975</u>, *20*, 908-14; *Russ. J. Inorg. Chem. (Engl. Transl.)* <u>1975</u>, *20*, 508-11.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solutions

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg^{-1} (compilers)	density/g cm^{-3}
41.0	1.52	573.1	2.12	2.56	1.39

The solid phase is $HoCl_3$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and precipitated with aq NH_3. The pptd $Ho(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous and only the anhydrous $HoCl_3$ was found.

All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complexation between TBP and $HoCl_3$ in solution.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $HoCl_3$ prepared by chlorination of the oxide with CCl_4 vapor (1,2). Source and purity of materials not given. Ho was analyzed gravimetrically, and Cl by Volhard's method.

Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimate possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* <u>1964</u>, *9*, 1427.
2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* <u>1965</u>, *38*, 1160

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

			$HoCl_3$ solubility[a]	
solvent			mass %	mol kg^{-1}
1-propanamine;	$n-C_3H_9N$;	[107-10-8]	33.4	1.849
di-2-butylamine;	$(sec-C_3H_9N)_2NH$;	[626-23-3]	1.2	0.045

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1761-6; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with the solvate $HoCl_3.3C((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.108 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $HoCl_3.3C_6H_{18}N_3OP$.

The solvate was analyzed for metal content by complexometric titration, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate were also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$HoCl_3.3C_6H_{18}N_3OP$ prepared by dissolving the hydrate in the solvent and heating to 140-145°C for 5 m. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over P_2O_5 in a stream of dry nitrogen. Yield was about 90%.

The solvent was purified as described in (1).

ESTIMATED ERROR:

Soly: precision ± 0.001 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Holmium chloride; $HoCl_3$; [10138-62-2]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

T/K = 293

Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	solubility^{a,b} moles Ho dm^{-3}
0	0	0.003
1:100	0.085	0.009
1:50	0.17	0.026 (0.015)
1:25	0.33	0.041
1:15	0.59	0.031
1:10	0.78	0.044

[a] Solutions preheated to 220°C. Value in parenthesis corresponds to preheating at 120°C.

[b] This is also the solubility of $HoCl_3$ in the $SnCl_4$-$POCl_3$ mixture because the oxide is quantitatively converted to the chloride according to

$$Ho_2O_3 + 6POCl_3 = 2HoCl_3 + 3P_2O_3Cl_4$$

Authors state that the solubility of $HoCl_3$ is enhanced by complex formation according to

$$2HoCl_3 + 3SnCl_4 = Ho_2(SnCl_6)_3$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Ho_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20° C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Prehating to 220°C lowered the equilibration time at 20°C to 2 hours.

Ho was determined by colorimetric analysis or by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Ho_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS:

(1) Holmium bromide; $HoBr_3$; [13825-76-8]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. 1966, 97, 1357-64.

VARIABLES:

Room Temperature: T/K = 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $HoBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.38 g per 100 ml of solution (0.0094 mol dm^{-3}, (compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Holmium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $HoBr_3.3.5C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $HoBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Erbium fluoride; ErF_3;
[13760-83-3]

(2) Alcohols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			ErF_3 solubility[a,b] mass %	mol kg^{-1}
methanol	CH_4O;	[67-56-1]	0.01	4.5×10^{-4}
ethanol	C_2H_6O	[64-17-5]	0.01	4.5×10^{-4}

[a] Molalities calculated by the compilers.

[b] Solid phases were dried in a desiccator over P_4O_{10} and the Er:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of ErF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Er(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Er determined several times by complexometric titration with potentiometric endpoint detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator.

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Er_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $ErF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvents were dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Erbium fluoride; ErF_3;
[13760-83-3]

(2) Ethers

ORIGINAL MEASUREMENTS:

Dressler, H.

Dissertationschrift. Paed. Inst. Koethen. GDR. 1980.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			ErF_3 solubility mass %	 mol/100 g sln	solid phase Er:F:solvent ratio
1-methoxydecane;	$n-C_{11}H_{24}O$;	[7289-52-3]	0.02	$9x10^{-5}$	1:2.95:0.15
1-(chloromethoxy)butane;	$n-C_5H_{11}ClO$;	[2351-69-1]	0.02	$9x10^{-5}$	1:2.88.0.33

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method analogous to that described in (1). No other information available.

SOURCE AND PURITY OF MATERIALS:

It appears that the fluoride was prepared as in (1). In spite of drying the fluoride by two methods at 573 K, the $Er:F:H_2O$ ratio was 1:3.01:0.50.

No other information available.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kirmse, E.M.
Wiss. Hefte, Paed. Inst. Koethen. 1978, *2*, 85.

COMPONENTS:

(1) Erbium fluoride; ErF_3; [13760-83-3]

(2) Tributyl phosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of ErF_3 in $[CH_3(CH_2)_3]_3P(O)$ at room temperature was given as

0.01 mass %

The corresponding molality calculated by the compiler is

4.5×10^{-4} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{10} and the Er:F ratio determined to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of ErF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Er(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Er determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Er_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $ErF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Erbium fluoride; ErF_3;
[13760-83-3]

(2) Dimethylsulfoxide; C_2H_6OS;
[67-68-5]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of ErF_3 in $(CH_3)_2SO$ at room temperature was given as

0.03 mass %

The corresponding molality calculated by the compiler is

$$1.3 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Er:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of ErF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Er(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Er determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous prallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Er_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $ErF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M.
Z. Chem. 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M.
Chemia Analityczna 1975, *20*, 33.

COMPONENTS:

(1) Erbium fluoride; ErF_3; [13760-83-3]

(2) Pyridine; C_6H_5N; [110-86-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of ErF_3 in pyridine at room temperature was reported to be

0.02 mass %

The corresponding molality calculated by the compiler is

8.9×10^{-4} mol kg^{-1}

The solid phase was dried in a desiccator over P_4O_{11} and the Er:F ratio found to equal almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of ErF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% $er(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Er determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Er_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $ErF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Erbium chloride; $ErCl_3$; [10138-41-7]

(2) Methanol; CH_4O; [67-56-1]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P.
Helv. Chim. Acta 1972, 55, 44-52.

Pitteloud, M.N.
These. Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubilities/mol kg^{-1} a	b
25	4.53	4.63

[a] Initial salt is the adduct $ErCl_3.4CH_3OH$. Equilibrated solid phase analyzed and found to be $ErCl_3.4CH_3OH$.

[b] Solutions equilibrated with anhydrous $ErCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $ErCl_3.4CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Erbium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $ErCl_3.4CH_3OH$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, 7, 224 (this is the reference to a paper by J.H. Freeman and M.L. Smith which described the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Er_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as described in (3). The adduct $ErCl_3.4CH_3OH$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and crystallization from methanol.

Methanol was purified and dried by the Vogel method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:	EVALUATOR:
(1) Erbium chloride; $ErCl_3$; [10138-41-7] (2) Ethanol; C_2H_6O; [64-17-5]	Tomasz Mioduski Institute of Nuclear Research Warsaw, Poland and Mark Salomon USA ET & DL Ft. Monmouth, NJ, USA

CRITICAL EVALUATION:

The solubility of erbium chloride in ethanol at 298.2 K has been reported in two publications (1,2), and the results are summarized in the following table.

initial solid	equilibrated solid	solubility/mol kg^{-1}	(ref)
$ErCl_3$	$ErCl_3 \cdot C_2H_5OH$	3.35	(1)
$ErCl_3$	not analyzed	3.48	(2)
$ErCl_3 \cdot 4C_2H_5OH$	$ErCl_3 \cdot 4.1C_2H_5OH$	3.41	(2)

The publication by Kirmse (1) does not contain experimental details or sources and purities of materials, but they probably were similar to those reported by Merbach et al. (2). For a full description of Kirmse's experimental methods, precision and sources of materials, see the INTRODUCTION to this volume. Kirmse prepared the anhydrous salt by the method of Taylor and Carter (3), and Merbach et al. propably used the method of Freeman and Smith (4). The experimental methods used by Merbach et al. were described in detail: high purity Er_2O_3 (>99.9 %) was used, the stoichiometry of the salts verified by both cation and anion chemical analysis and by X-ray diffraction, the absence of the oxychloride and the purity of the solvent were confirmed. Given these details the compilers have estimated an experimental precision of ±0.5 % for the final results of Merbach et al. While it is difficult to determine the precision of Kirmse's data, the accuracy of these data are probably 2-3 % at best.

Merbach et al. (2) consider the difference in solubilities of the anhydrous chloride and the tetrasolvate to be due to the formation of small quantities of chloroalcoholates (dissolution of the anhydrous salt is highly exothermal). However Kirmse reports the solid phase to be the monosolvate when starting with the anhydrous salt. If in this case it is true that the "equilibrated" solid phase is the monosolvate, then we would have to conclude that either the monosolvate or the tetrasolvate is metastable. It would appear to the evaluators that the tetrasolvate is the stable solid phase which means that the solubility should be higher in the metastable monosolvate system. The data of Merbach et al. suggest this, but the data of Kirmse is contradictory. It is interesting to note that the average solubility for the "monosolvate" systems is 3.42 mol kg^{-1} which is essentially identical (within experimental error) to the value of 3.41 mol kg^{-1} reported by Merbach et al. for the tetrasolvate system.

In summary it appears that at 298.2 K the stable solid phase is the tetrasolvate, and that we can designate the *tentative* solubility in this system as 3.41 mol kg^{-1}. When solutions are equilibrated with anhydrous $ErCl_3$, it is not clear whether the resulting saturated solution contains a metastable monosolvate solid phase, or whether the solid phase is actually the stable tetrasolvate.

REFERENCES

1. Kirmse, E. M. *Tr. II Vses Konf. po Teor. Rastvorov* 1971, 200.
2. Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44: Pitteloud, M.N. *These.* Faculté des Sciences de l'Université de Lausanne. 1971.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.
4. Freeman, J.H.; Smith, M.L. *J. Inorg. Nucl. Chem.* 1958, *7*, 224.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Erbium chloride; $ErCl_3$; [10138-41-7] (2) Alcohols	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.

VARIABLES:	PREPARED BY:
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			mass %	mol kg^{-1}	nature of the solid phase
ethanol;	C_2H_6O;	[64-17-5]	47.8	3.347	$ErCl_3 \cdot C_2H_6O$
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	3.5	0.133	$ErCl_3.nC_3H_8O_2$ (n = 2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	7.6	0.301	$ErCl_3.2C_4H_{10}O_2$

[a]Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the authors, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Erbium chloride; $ErCl_3$; [10138-41-7]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44-52.

Pitteloud, M.N. *These*. Faculte des Sciences de l'universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubilities/mol kg^{-1} a	b
25	3.41	3.48

[a] Initial salt is the adduct $ErCl_3.4C_2H_5OH$. Equilibrated solid phase analyzed and found to be $ErCl_3.4.1C_2H_5OH$.

[b] Solutions equilibrated with anhydrous $ErCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $ErCl_3.4C_2H_5OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Erbium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $ErCl_3.4C_2H_5O$ confirmed by ^1H NMR and X-ray diffraction

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, *7*, 224 (this is the reference to a paper by J.H. Freeman and M.L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Er_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $ErCl_3.4C_2H_6O$ prepared by dissolving the hydrate in a small excess of o-ethylformate followed by distillation and crystallization from ethanol.

Ethanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR method.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl Chem.* 1962, *24*, 387 (see COMMENTS at left).

COMPONENTS:

(1) Erbium chloride; $ErCl_3$; [10138-41-7]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, Yu.G; Ezhova, T.A.

Zh. Neorg. Khim. 1976, *21*, 551-4; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1976, *21*, 296-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $ErCl_3.6H_2O$ in 96.8 % $C_2H_5OH^a$

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g^b	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}
20	37.86	37.57	38.19	37.99	37.90	1.599
30	37.38	37.68	37.61	37.47	37.53	1.574
40	37.67	37.50	37.37	37.50	37.51	1.573
50	38.58	38.84	38.62	38.78	38.70	1.654
60	40.29	40.49	40.02	40.30	40.27	1.766

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$ErCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 163.8 - 165.0°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.9 % (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Erbium chloride; $ErCl_3$; [10138-41-7]

(2) 2-Propanol; C_3H_8O; [67-63-0]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44-52.

Pitteloud, M.N. *These.* Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

	mean solubilities/mol kg^{-1}	
t/°C	a	b
25	0.84	0.83

[a] Initial salt is the adduct $ErCl_3.3C_3H_7OH$. Equilibrated solid phase analyzed and found to be $ErCl_3.3.7C_3H_8O$.

[b] Solutions equilibrated with anhydrous $ErCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $ErCl_3.3C_3H_7OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Erbium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $ErCl_3.3C_3H_8O$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

COMMENTS AND/OR ADDITIONAL DATA:

Reference (3) was incorrectly cited in the source paper as: *J. Inorg. Nucl. Chem.* 1958, 7, 224 (this is the reference to a paper by J.H. Freeman and M.L. Smith which describes the preparation of anhydrous salts by treatment with thionyl chloride). Reference (3) was corrected by the compilers.

SOURCE AND PURITY OF MATERIALS:

Er_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $ErCl_3.3C_3H_8O$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and trans-solvation of the methanol complex with 2-propanol.

Iso-propanol (fluka) was used as received. Purity and absence of water was confirmed by NMR.

ESTIMATED ERROR:

Soly: precision ± 0.5% as in (1) (compilers).

Temp: precision probably at least ± 0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47* 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.
3. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387 (see COMMENTS at left.

COMPONENTS:

(1) Erbium chloride; $ErCl_3$; [10138-41-7]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.

Z. Chem. 1967, *7*, 281.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ErCl_3$ in 1,2-diethoxyethane at 25°C was reported to be

0.67 mass %

The corresponding molality calculated by the compiler is

$0.0247 \text{ mol kg}^{-1}$

The composition of the solid phase was given in terms of the Er:Cl:ether ratio as

1:2.93:1.86

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over P_2O_5.

Er was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:	EVALUATOR:
(1) Erbium chloride; $ErCl_3$; [10138-41-7] (2) 1,1'-Oxybis-propane (di-n-propyl ether); $C_6H_{14}O$; [111-43-3]	Tomasz Mioduski Institute of Nuclear Research Warsaw, Poland

CRITICAL EVALUATION:

The solubility of erbium chloride in di-n-propyl ether at 298.2 K has been reported in three publications by Kirmse et al. (1-3). In (1) the solubility was given as 0.1 mass % (0.004 mol kg^{-1}), and in (2,3) the solubility was given as 0.2 mass % (0.007 mol kg^{-1}). Reference (1) specifies the solid phase as $ErCl_3 \cdot 0.47C_6H_{14}O$.

Since we believe all the solubility data in (1) to contain a large systematic error, these results must be rejected. This also means that the composition of the equilibrated solid phase cannot be assumed to be correct.

The solubility of 0.007 mol kg^{-1} at 298.2 K reported in (2,3) is designated as a *tentative* solubility, and the precision of this value is probably 10 % at best.

REFERENCES

1. Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J. *Wiss. Hefte, Paed. Inst. Koethen,* 1968, *1*, 128.
2. Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472.
3. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200.

COMPONENTS:	EVALUATOR:
(1) Erbium chloride; $ErCl_3$; [10138-41-7] (2) 1-Ethoxybutane (ethyl n-butyl ether); $C_6H_{14}O$; [628-81-9]	Tomasz Mioduski Institute of Nuclear Research Warsaw, Poland

CRITICAL EVALUATION:

The solubility of erbium chloride in 1-ethoxybutane at 298.2 K has been reported in three publications by Kirmse et al. (1-3). In (1) the solubility was given as 0.3 mass % (0.011 mol kg^{-1}), and in (2,3) the solubility was given as 0.5 mass % (0.018 mol kg^{-1}). Reference (1) specifies the solid phase as $ErCl_3 \cdot 0.49C_6H_{14}O$.

Since we believe all the solubility data in (1) to contain a large systematic error, these results must be rejected. This also means that the composition of the equilibrated solid phase cannot be assumed to be correct.

The solubility of 0.018 mol kg^{-1} at 298.2 K reported in (2,3) is designated as a *tentative* solubility, and the precision of this value is probably 10 % at best.

REFERENCES

1. Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J. *Wiss. Hefte, Paed. Inst. Koethen,* 1968, *1*, 128.
2. Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472.
3. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200.

COMPONENTS:	EVALUATOR:
(1) Erbium chloride; $ErCl_3$; [10138-41-7] (2) 1-Methoxypentane (methyl n-amyl ether); $C_6H_{14}O$; [628-80-8]	Tomasz Mioduski Institute of Nuclear Research Warsaw, Poland

CRITICAL EVALUATION:

The solubility of erbium chloride in 1-methoxypentane at 298.2 K has been reported in three publications by Kirmse et al. (1-3). In (1) the solubility was given as 30.8 mass % (1.63 mol kg^{-1}), and in (2,3) the solubility was given as 33.5 mass % (1.84 mol kg^{-1}). Reference (1) reports the solid phase to be $ErCl_3 \cdot 0.49C_6H_{14}O$.

Since descriptions of experimental techniques and purities of materials were not given, it is not possible to determine the source of the differences in solubility values. However we can probably conclude that the data in (1) should be rejected.

The solubility data reported in (1) are in all cases significantly different than those subsequently reported by Kirmse (e.g. see the critical evaluations for $ErCl_3$ in 1-ethoxybutane and in di-n-propyl ether, and for YCl_3 in 1-methoxypentane). It is interesting to also note that the solubility data reported in (3) are generally in agreement with those data reported by Merbach et al. (4) (e.g. see the critical evaluation for the $ErCl_3$ - C_2H_5OH system where the difference between Kirmse's result and Merbach's result is around 2 %). On this basis the data in (1) are rejected which also means that the composition of the equilibrated solid phase cannot be assumed to be correct.

The solubility result of 1.84 mol kg^{-1} at 298.2 K reported in (2,3) is designated as a *tentative* value, and the precision of this value is probably around 2 %.

REFERENCES

1. Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J. *Wiss. Hefte, Paed. Inst. Koethen,* 1968, *1*, 128.
2. Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472.
3. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200.
4. Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44.

COMPONENTS:

(1) Erbium chloride; $ErCl_3$; [10138-41-7]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.

Wiss. Hefte, Paed. Inst. Koethen 1968, *1*, 128-30.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	mol kg^{-1}
di-n-propyl ether;	$C_6H_{14}O$;	[111-43-3]	0.1[b]	0.004
1-ethoxybutane;	$C_6H_{14}O$;	[628-81-9]	0.3[c]	0.011
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	30.8[d]	1.627

[a] Molalities calculated by the compilers.

[b] Solid phase is $ErCl_3.0.47C_6H_{14}O$.

[c] Solid phase is $ErCl_3.0.37C_6H_{14}O$.

[d] Solid phase is $ErCl_3.0.49C_6H_{14}O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Mixtures were agitated isothermally at 25°C until equilibrium was reached. Er was determined by complexometric titration using Xylenol Orange indicator.

No other information available.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not specified. The anhydrous rare earth chloride was prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Erbium chloride; $ErCl_3$; [10138-41-7]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U.
Z. Chem. 1968, *8*, 472-3.

Kirmse, E.M.
Tr. II Vses. Konf. po Teor. Rastvorov. 1971, 200-6.

VARIABLES:

Room Temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$ErCl_3$ solubility[a,b] mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.9	0.033
di-n-propyl ether;	$C_6H_{14}O_2$;	[111-43-3]	0.2	0.007
1-ethoxybutane;	$C_6H_{14}O$;	[628-81-9]	0.5	0.018
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	33.5	1.841
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.7	0.026

[a] Molalities calculated by the compilers.

[b] Nature of solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Er determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS: (1) Erbium chloride; $ErCl_3$; [10138-41-7] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	**ORIGINAL MEASUREMENTS:** Rossmanith, K.; Auer-Welsbach, C. *Monatsh. Chem.* 1965, *96*, 602-5.
VARIABLES: Room Temperature: T/K about 293	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ErCl_3$ in tetrahydrofuran at 20°C (room temperature) was reported to be

0.786 g per 100 ml of solution

(0.0287 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.
Erbium was determined by the oxalate method, and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $ErCl_3 \cdot 3.47C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $ErCl_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Cl followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Erbium chloride; $ErCl_3$; [10138-41-7] (2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]	Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N. *Zh. Neorg. Khim.* <u>1975</u>, *20*, 908-14; *Russ. J. Inorg. Chem. (Engl. Transl.)* <u>1975</u>, *20*, 508-11.
VARIABLES:	**PREPARED BY:**
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solutions[a,b]

mass %	mol/kg sln[c]	g dm^{-3c}	mol dm^{-3c}	mol kg^{-1}	density/g cm^{-3}
41.3	1.43	530.0	1.94	2.57	1.36

[a] Solid phase is $ErCl_3$.

[b] Molality calculated by the compilers from the experimental solubility of 41.3 mass %.

[c] It is <u>implied</u> that these data also correspond to the saturated solution. However the molality calculated from these data is 2.35 mol kg^{-1}. The compilers conclude that these data probably do not correspond to saturated solutions.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and precipitated with aq NH_3. The pptd $Er(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous and only the anhydrous $ErCl_3$ was found.

All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complexation between TBP and $ErCl_3$ in solution.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $ErCl_3$ prepared by chlorination of the oxide with CCl_4 vapor (1,2). Source and purity of materials not given. Er was analyzed gravimetrically, and Cl by Volhard's method.

Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimate possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* <u>1964</u>, *9*, 1427.
2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* <u>1965</u>, *38*, 1160.

COMPONENTS:

(1) Erbium chloride; $ErCl_3$;
[10138-41-7]

(2) Amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

			$ErCl_3$ solubility[a]	
solvent			mass %	mol kg^{-1}
diethylamine;	$(C_2H_5)_2NH$;	[109-89-7]	0.1	0.004
2-propanamine;	iso-C_3H_9N;	[75-31-0]	26.9	1.34_5
2-propen-1-amine;[b]	C_3H_7N;	[107-11-9]	29.9	1.559
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	27.9	1.414
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	18.6	0.835
di-2-butylamine;	(sec-$C_4H_9)_2N$;	[626-23-3]	1.0	0.037

[a] Molalities calculated by the compilers.

[b] The source paper specifies the solvent as $C_3H_5NH_2$. Upon request, the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of the solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Erbium chloride; $ErCl_3$; [10138-41-7]

(2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1961-6; *Russ. J. Inrog. Chem. (Engl. Transl.)* 1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with the solvate $ErCl_3.3C((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.098 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $ErCl_3.3C_6H_{18}N_3OP$.

The solvate was analyzed for metal content by complexometric titration, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate were also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$ErCl_3.3C_6H_{18}N_3OP$ prepared by dissolving the hydrate in the solvent and heating to 140-145°C for 5 m. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over P_2O_5 in a stream of dry nitrogen. Yield was about 90%.

The solvent was purified as described in (1).

ESTIMATED ERROR:

Soly: precision ± 0.002 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Erbium chloride; $ErCl_3$; [10138-41-7]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

$SnCl_4$ concentration

T/K = 293 and 333

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concn mol dm^{-3}	solubility of Er_2O_3/mol Er dm^{-3a} 20°C^b	20°C^c	60°C^d
0	0	0.002	----	0.0001
1:250	0.035	----	0.004	0.005
1:100	0.085	0.006	0.006	0.005
1:50	0.17	0.008 (0.007)	0.006	0.006
1:25	0.33	0.024	0.007	0.007
1:15	0.59	0.024	0.006	0.005
1:10	0.78	0.034	0.006	0.0055
1:5	1.4	----	----	0.011
1:1.5	3.0	----	----	0.011
1:1	4.6	----	----	0.011
4:1	6.8	----	0.010	0.001
pure $SnCl_4$	8.5	0.001	----	----

[a] This is also the solubility of $ErCl_3$ since the oxide is quantitatively converted to the chloride according to

$$Er_2O_3 + 6POCl_3 = 2ErCl_3 + 3P_2O_3Cl_4$$

Assuming $P_2O_3Cl_4$ to be soluble, the equilibrated solutions would then constitute a four component mixture.

[b] Preheated at 220°C for 2 hours. (120°C for the value in parenthesis).

[c] Preheated at 60°C (time not specified).

[d] No pretreatment.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3 + SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Er_2O_3 were placed in sealed ampoules, heated to 120°C for 2 hours to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20°C to 2 hours.

Er was determined by colorimetric analysis, and in some cases by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

The solubility of $ErCl_3$ in pure $POCl_3$ is small, but in the presence of $SnCl_4$ the solubility increases due to complexation:

$$2ErCl_3 + 3SnCl_4 = Er_2(SnCl_6)_3$$

SOURCE AND PURITY OF MATERIALS:

Er_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2 K (compiler).

REFERENCES:

COMPONENTS: (1) Erbium bromide; $ErBr_3$; [13536-73-7] (2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]	**ORIGINAL MEASUREMENTS:** Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: T/K = 298	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ErBr_3$ in 1,2-diethoxyethane at 25°C was reported as

1.25 mass %

The corresponding molality calculated by the compiler is

0.0311 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:
Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Erbium bromide; $ErBr_3$; [13536-73-7]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. 1966, 97, 1357-64.

VARIABLES:

Room Temperature: T/K = 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ErBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.41 g per 100 ml of solution

(0.0101 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Erbium determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $ErBr_3.3.5C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $ErBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS: (1) Erbium bromide; $ErBr_3$; [13536-73-7] (2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]	**ORIGINAL MEASUREMENTS:** Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. *Z. Chem.* 1968, *8*, 472-3. Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.
VARIABLES: Room Temperature: T/K around 298	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $ErBr_3$ in p-dioxane at about 25°C was given as

0.6 mass %

The corresponding molality calculated by the compiler is

$0.015 \text{ mol kg}^{-1}$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Er was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Erbium bromide; $ErBr_3$; [13536-73-7]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	24.5	0.797
2-propanamine;	iso-C_3H_9N;	[75-31-0]	38.9	1.564
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	21.4	0.669
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	37.0	1.443
di-2-butylamine;	(sec-$C_4H_9)_2NH$;	[626-23-3]	0.8	0.020

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Thulium chloride; $TmCl_3$; [13537-18-3]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, Yu.G.; Ezhova, T.A.

Zh. Neorg. Khim. 1976, *21*, 551-4; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1976, *21*, 296-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $TmCl_3.6H_2O$ in 96.8% C_2H_5OH[a]

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	42.09	42.20	42.26	41.91	42.11	1.897
30	41.06	41.16	41.12	41.14	41.11	1.821
40	41.70	41.73	42.09	41.83	41.83	1.876
50	41.17	44.21	44.44	44.50	44.33	2.077
60	47.08	47.26	47.46	47.35	47.29	2.340

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$TmCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 162.4 - 163.5°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.9 % (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Thulium chloride; $TmCl_3$; [13537-18-3]

(2) 2-Methoxyethanol; $C_3H_8O_2$; [109-86-4]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $TmCl_3$ in 1,2-dimethoxyethane at 25°C was reported as

6.3 mass %

The corresponding molality calculated by the compiler is

0.244 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Thulium chloride; $TmCl_3$; [13537-18-3]

(2) Diethyl ether; $C_4H_{10}O$; [60-29-7]

ORIGINAL MEASUREMENTS:

Dzhuraev, Kh. Sh.; Mirsaidov, U.; Kurbanbekov, A.; Rakhimova, A.

Dokl. Akad. Nauk Tadzh. SSR 1976, *19*, 32-4

VARIABLES:

T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $TmCl_3$ in diethyl ether at 20°C was reported as

0.053 mass %

The corresponding molality calculated by the compiler is

1.94×10^{-3} mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrium was attained within 24 h, and it was verified by constancy in the Tm concentration. The saturated solution and the equilibrated solid phase were analyzed. Tm determined by complexometric titration using urotropine buffer and methyl-thymol blue indicator. Chloride determined by titration with $AgNO_3$. The solid phase corresponded to $TmCl_3.Et_2O$ (the etherate was dried under vaccum at 40°C prior to analysis).

SOURCE AND PURITY OF MATERIALS:

Anhydrous $TmCl_3$ prepared by the ethanol solvate method (no details given).

Ethyl ether was dried with Na and distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thulium chloride; $TmCl_3$; [13537-18-3] (2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]	Kirmse, E.M.; Zwietasch, K.J. *Z. Chem.* 1967, *7*, 281.
VARIABLES:	**PREPARED BY:**
T/K = 298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $TmCl_3$ in 1,2-diethoxyethane at 25°C was reported to be

0.88 mass %

The corresponding molality calculated by the compiler is

0.0323 mol kg^{-1}

The composition of the solid phase was given in terms of the Eu:Cl:ether ratio as

1:2.97:2.00

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over P_2O_5.

Tm was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
   J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Thulium chloride; $TmCl_3$; [13537-18-3]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.

Zh. Neorg. Khim. 1975, *20*, 908-14; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 508-11.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solutions

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg^{-1} (compilers)	density/g cm^{-3}
41.4	1.49	580.2	2.03	2.57	1.40

The solid phase is $TmCl_3$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and precipitated with aq NH_3. The pptd $Tm(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous, and only the anhydrous $TmCl_3$ was found.

All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complexation between TBP and $TmCl_3$ in solution.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $TmCl_3$ prepared by chlorination of the oxide with CCl_4 vapor (1,2). Source and purity of materials not given. Tm was analyzed gravimetrically, and Cl by Volhard's method.

Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimate possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* 1964, *9*, 1427.
2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* 1965, *38*, 1160.

COMPONENTS: (1) Thulium chloride; $TmCl_3$; [13537-18-3] (2) Amines	**ORIGINAL MEASUREMENTS:** Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES: T/K = 298	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	mol kg^{-1}
2-propanamine;	iso-C_3H_9N;	[75-31-0]	13.7	0.577
2-propen-1-amine;[b]	C_3H_7N;	[107-11-9]	29.0	1.484

[a] Molalities calculated by the compilers.

[b] The original paper simply specifies the solvent as $C_3H_5NH_2$, and upon request, the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Thulium chloride; $TmCl_3$; [13537-18-3]

(2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. <u>1977</u>, *22*, 1761-6; *Russ. J. Inorg. Chem. (Engl. Transl.)* <u>1977</u>, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with the solvate $TmCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.0935 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm 170 ($t_{1/2}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $TmCl_3.3C_6H_{18}N_3OP$.

The solvate was analyzed for metal content by complexometric titration, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate were also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $TmCl_3$, prepared by modification of Taylor and Carter's method (1) by subliming NH_4Cl from a mixture of $TmCl_3$ with 6 moles of NH_4Cl in a stream of inert gas at 200-400°C. The product contained less than 3% of TmOCl.

The solvent was purified as in (2).

ESTIMATED ERROR:

Soly: precision ± 0.001 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3K.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* <u>1962</u>, *24*, 387.
2. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* <u>1975</u>, *11*, 163.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thulium chloride; $TmCl_3$; [13537-18-3] (2) Tetrachlorostannate; $SnCl_4$; [7646-78-8] (3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]	Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, *45*, 1176-8.
VARIABLES:	**PREPARED BY:**
T/K = 293 Concentration of $SnCl_4$	T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	Tm_2O_3 solubility[a,b] moles Tm dm^{-3}
1:100	0.085	0.8
1:50	0.17	0.8 (0.6)
1:25	0.33	1.2
1:15	0.59	1.3
1:10	0.78	2.1

[a] This is also the solubility of $TmCl_3$ in the $SnCl_4$-$POCl_3$ mixtures because the oxide is quantitatively converted to the chloride according to

$$Tm_2O_3 + 6POCl_3 = 2TmCl_3 + 3P_2O_3Cl_4$$

Thus the equilibrated solutions should actually be considered to be a four component system containing $SnCl_4$, $TmCl_3$, $P_2O_3Cl_4$ and $POCl_3$ (the compiler assumes $P_2O_3Cl_4$ is soluble).

[b] Mixtures preheated to 220°C for 2 hours prior to equilibration at 20°C (value in parenthesis indicates preheating at 120°C.)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Tm_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 220°C lowered the equilibration time at 20° to 2 hours.

Tm was determined by colorimetric analysis, and in some cases by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Tm_2O_3 of "the first sort" was heated at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS:

(1) Thulium bromide; $TmBr_3$;
[14456-51-0]

(2) Tetrahydrofuran; C_4H_8O;
[109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. <u>1966</u>, *97*, 1357-64.

VARIABLES:

Room Temperature: T/K = 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $TmBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.41 g per 100 ml of solution

(0.0100 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Thulium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $TmBr_3.3C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $TmBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Ytterbium fluoride; YbF_3; [13760-80-0]

(2) Dimethylsulfoxide; C_2H_6OS; [67-68-5]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YbF_3 in $(CH_3)_2SO$ at room temperature was given as

0.04 mass %.

The corresponding molality calculated by the compiler is

$0.0017 \text{ mol kg}^{-1}$

The solid phase was dried in a desiccator over P_4O_{10} and the Yb:F ratio found to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of YbF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Yb(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Yb determined several times by complexometric titration with potentiometric endpoint detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Yb_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $YbF_3 \cdot 0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: nothing specified.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Ytterbium fluoride; YbF_3; [13760-80-0]

(2) Pyridine; C_5H_5N; [110-86-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Wiss. Hefte, Paed. Inst. Koethen 1978, *2*, 85-90.

VARIABLES:

Room Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YbF_3 in pyridine at room temperature was given as

0.03 mass %

The corresponding molality calculated by the compiler is

$$1.3 \times 10^{-3} \text{ mol kg}^{-1}$$

The solid phase was dried in a desiccator over P_4O_{10} and the Yb:F ratio determined to be almost 1:3.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 mg of YbF_3 was added to 10-20 cm^3 of solvent, and the mixture mechanically agitated at room temperature for 100 h. 5-10 g of saturated solution were removed by decanting or by centrifuging, and the solution evaporated to dryness. The residue was heated with about 10 cm^3 of 10% KOH solution for 1-2 h to obtain solid $Yb(OH)_3$ and a basic F^- solution. The precipitate was washed, dissolved in aq HCl, and Yb determined several times by complexometric titration with potentiometric end-point detection (1). The fluoride content in the filtrate was determined photometrically using Al-Eriochrome cyanine color lake indicator (2).

The reported solubility is a mean of "numerous parallel determinations," or "at least two parallel determinations."

SOURCE AND PURITY OF MATERIALS:

Yb_2O_3 (source and purity not specified) was dissolved in HCl and the fluoride precipitated by addition of aq HF. The solid produced was $YbF_3.0.5H_2O$ and was dehydrated by washing with acetone followed by drying at 310°C for 120 hours.

The solvent was dried and purified by "standard methods."

ESTIMATED ERROR:

Soly: results with relative errors exceeding 50% were rejected.

Temp: unknown.

REFERENCES:

1. Schilbach, U.; Kirmse, E.M. *Z. Chem.* 1974, *14*, 484.
2. Schilbach, U.; Hetze, I.; Kirmse, E.M. *Chemia Analityczna* 1975, *20*, 33.

COMPONENTS:

(1) Ytterbium chloride; $YbCl_3$; [10361-91-8]

(2) Methanol; CH_4O; [67-56-1]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, *55*, 44-52.

Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubility[a] mol kg^{-1}
25	4.90

[a] Initial salt is the adduct $YbCl_3.4CH_3OH$. Equilibrated solid phase analyzed and found to be $YbCl_3.3.8CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Ytterbium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $YbCl_3.4CH_3OH$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

SOURCE AND PURITY OF MATERIALS:

Yb_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The adduct $YbCl_3.4CH_3OH$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and crystallization from methanol.

Methanol was purified and dried by the Vogel method.

ESTIMATED ERROR:

Soly: precision $\pm 0.5\%$ as in (1) (compilers).

Temp: precision probably at least ± 0.05 K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinch, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.

COMPONENTS:

(1) Ytterbium chloride; $YbCl_3$; [10361-91-8]

(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, *55*, 44-52.

Pitteloud, M.N. *These.* Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubility[a] mol kg^{-1}
25	4.26

[a] Initial salt is the adduct $YbCl_3.3C_2H_5OH$. Equilibrated solid phase analyzed and found to be $YbCl_3.3.6C_2H_5OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Ytterbium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $YbCl_3.3C_2H_5O$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

SOURCE AND PURITY OF MATERIALS:

Yb_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The adduct $YbCl_3.3C_2H_6O$ prepared by dissolving the hydrate in a small excess of o-ethylformate followed by distillation and crystallization from ethanol.

Ethanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR method.

ESTIMATED ERROR:

Soly: precision ±0.5% as in (1) (compilers).

Temp: precision probably at least ±0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.

COMPONENTS:

(1) Ytterbium chloride; $YbCl_3$; [10361-91-8]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, Yu.G.; Ezhova, T.A.

Zh. Neorg. Khim. 1976, *21*, 551-4; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1976, *21*, 296-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $YbCl_3.6H_2O$ in 96.8 % C_2H_5OH[a]

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	47.38	47.30	47.45	47.35	47.37	2.323
30	47.70	47.65	47.74	47.76	47.71	2.355
40	49.54	49.65	49.59	49.63	49.60	2.540
50	51.95	52.10	51.90	51.85	51.95	2.790
60	58.20	58.35	58.29	58.40	58.31	3.610

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$YbCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 153.0 - 155.2°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.9 % (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Ytterbium chloride; $YbCl_3$; [10361-91-8]

(2) 2-Propanol; C_3H_8O; [67-63-0]

ORIGINAL MEASUREMENTS:

Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, *55*, 44-52.

Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubility[a] mol kg^{-1}
25	3.6

[a] Initial salt is the adduct $YbCl_3.3C_3H_7OH$. Equilibrated solid phase analyzed and found to be $YbCl_3.nC_3H_7OH$ where n = 3.5 to 3.6.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Ytterbium determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $YbCl_3.3C_3H_8O$ confirmed by ^1H NMR and X-ray diffraction.

The reported solubilities are mean values of 2-4 determinations.

SOURCE AND PURITY OF MATERIALS:

Yb_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The adduct $YbCl_3.3C_3H_8O$ prepared by dissolving the hydrate in a small excess of o-methlformate followed by distillation and trans-solvation of the methanol complex with 2-propanol.

Iso-propanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR.

ESTIMATED ERROR:

Soly: precision ±0.5% as in (1) (compilers).

Temp: precision probably at least ±0.05K as in (1) (compilers).

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, R. *Chimia* 1952, *6*, 62.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ytterbium chloride; $YbCl_3$; [10361-91-8] (2) Alkoxy-ethanols	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	PREPARED BY:
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$YbCl_3$ solubility[a] mass %	mol kg^{-1}	nature of the solid phase
2-methoxyethanol;	C_3H_8O;	[109-86-4]	8.6	0.337	$YbCl_3.nC_3H_8O_2$ (n = 2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	9.1	0.358	$YbCl_3.2C_4H_{10}O_2$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the authors, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS: (1) Ytterbium chloride; $YbCl_3$; [10361-91-8] (2) Diethyl ether; $C_4H_{10}O$; [60-29-7]	**ORIGINAL MEASUREMENTS:** Dzhuraev, Kh. Sh.; Mirsaidov, U.; Kurbanbekov, A.; Rakhimova, A. *Dokl. Akad. Nauk Tadzh. SSR* 1976, *19*, 32-4.
VARIABLES: T/K = 293	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $YbCl_3$ in diethyl ether at 20°C was reported to be

0.085 mass %

The corresponding molality calculated by the compiler is

3.04×10^{-3} mol kg^{-1}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrium was attained within 24 h and was verified by constancy in the $YbCl_3$ concentration. The saturated solution and the equilibrated solid phase were analyzed. Yb determined by complexometric titration in presence of urotropine buffer and methyl-thymol indicator. Chloride determined by titration with $AgNO_3$. The solid phase corresponded to $YbCl_3.C_4H_{10}O$ (the etherate was dried under vacuum at 40°C prior to analysis).

SOURCE AND PURITY OF MATERIALS:

Anhydrous $YbCl_3$ prepared by the ethanol solvate method (no details given).

Ethyl ether was dried with Na and distilled from $LiAlH_4$ before use.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Ytterbium chloride; $YbCl_3$; [10361-91-8]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.

Z. Chem. 1967, *7*, 281.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $YbCl_3$ in 1,2-diethoxyethane at 25°C was reported to be

1.15 mass %

The corresponding molality calculated by the compiler is

$0.0416 \text{ mol kg}^{-1}$

The composition of the solid phase was given in terms of the Yb:Cl:ether ratio as

1:3.03:0.94

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over P_2O_5.

Yb was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials were not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethyleter of ethylene glycol.

ESTIMATED ERROR:

No estimate possible.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ytterbium chloride; $YbCl_3$; [10361-91-8] (2) 1-Methoxyhexane; $C_7H_{16}O$; [4747-07-3]	Baganz, E. *Staatsexamenarbeit*. Koethen Paedag. Institut. 1970. Original data cited by Kirmse, E.M.; Dressler, H. *Z. Chem.* 1975, *15*, 239-40.
VARIABLES:	**PREPARED BY:**
Room temperature; T/K = 293 - 298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $YbCl_3$ in 1-methoxyhexane at 20-25°C was reported as

18.3 mass %.

The corresponding molality calculated by the compiler is

0.802 mol kg^{-1}.

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated until equilibrium was attained.

The anhydrous reagents were handled in a dry box containing P_4O_{10}.

Ytterbium was determined by complexometric titration using Xylenol Orange indicator.

The reported solubility is a mean value based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Ytterbium chloride; $YbCl_3$; [10361-91-8]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. *Z. Chem.* 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a,b] mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	1.4_5	0.053
di-n-propyl ether;	$C_6H_{14}O$;	[111-43-3]	13.7	0.568
1-ethoxybutane;	$C_6H_{14}O$;	[628-81-9]	12.0	0.488
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	43.5	2.756
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.8	0.029

[a] Molalities calculated by the compilers.

[b] Nature of the solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Yb was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Ytterbium chloride; $YbCl_3$;
[10361-91-8]

(2) Tetrahydrofuran; C_4H_8O;
[109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.; Auer-Welsbach, C.

Monatsh. Chem. 1965, *96*, 602-5.

VARIABLES:

Room Temperature: T/K about 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $YbCl_3$ in tetrahydrofuran at 20°C (room temperature) was reported to be

1.98 g per 100 ml of solution

(0.0709 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Ytterbium was determined by the oxalate method, and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $YbCl_3.2.92C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $YbCl_3$ was prepared by conversion of the oxide by high temperature conversion with an excess of NH_4Cl followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ytterbium chloride; $YbCl_3$; [10361-91-8] (2) Amines	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$YbCl_3$ solubility[a] mass %	mol kg^{-1}
ethanamine;	C_2H_7N;	[75-04-7]	37.8	2.175
diethylamine;	$(C_2H_5)_2NH$;	[109-89-7]	2.0	0.073
1-propanamine;	C_3H_9N;	[107-10-8]	35.5	1.970
2-propanamine;	iso-C_3H_9N;	[75-31-0]	8.1	0.315
2-propen-1-amine;[b]	C_3H_7N;	[107-11-9]	34.2	1.860
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	38.7	2.260
2-butanamine;	iso-$C_4H_{11}N$;	[13952-84-6]	20.2	0.906
di-2-butylamine;	$(sec\text{-}C_4H_9)_2NH$;	[626-23-3]	3.9	0.145

[a] Molalities calculated by the compilers.

[b] The original paper simply specifies the solvent as $C_3H_5NH_2$, and upon request, the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Ytterbium chloride; $YbCl_3$; [10361-91-8]

(2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1961-6; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with the solvate $YBCl_3.3C((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.085 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{1/2}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $YbCl_3.3C_6H_{18}N_3OP$.

The solvate was analyzed for metal content by complexometric titration, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate were also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $YbCl_3$ prepared by modification of Taylor & Carter's method (1) by subliming NH_4Cl from a mixture of $YbCl_3$ with 6 moles of NH_4Cl in a stream of inert gas at 200-400°C. The product contained less than 3% of YbOCl.

The solvent was purified as in (2).

ESTIMATED ERROR:

Soly: precision ± 0.01 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.
2. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Ytterbium chloride; $YbCl_3$; [10361-91-8]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, 45, 1176-8.

VARIABLES:

T/K = 293
Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	Yb_2O_3 solubility^{a,b} moles Yb dm^{-3}
1:100	0.085	0.4
1:50	0.17	0.5 (0.1)
1:25	0.33	0.6
1:15	0.59	0.5
1:10	0.78	0.6

[a] Solutions preheated to 220°C. Value in parenthesis corresponds to preheating at 120°C.

[b] This is also the solubility of $YbCl_3$ in the $SnCl_4$-$POCl_3$ mixture because the oxide is quantitatively converted to the chloride according to

$$Yb_2O_3 + 6POCl_3 = 2YbCl_3 + 3P_2O_3Cl_4$$

Thus the equilibrated solutions should actually be considered to be a four component system containing $SnCl_4$, $YbCl_3$, $P_2O_3Cl_4$ and $POCl_3$ (the compiler assumes $P_2O_3Cl_4$ is soluble).

Authors state that the solubility of $YbCl_3$ is enhanced by complex formation according to

$$2YbCl_3 + 3SnCl_4 = Yb_2(SnCl_6)_3$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Yb_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20°C to 2 hours.

Yb was determined by colorimetric analysis, and in some cases by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Yb_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2 K (compiler).

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ytterbium bromide; $YbBr_3$; [13759-89-2] (2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
T/K = 298	T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $YbBr_3$ in 1,2-dimethoxyethane at 25°C was reported as

0.9 mass %.

The corresponding molality calculated by the compiler is

0.022 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phase not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D. Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Ytterbium bromide; $YbBr_3$;
[13759-89-2]

(2) Tetrahydrofuran; C_4H_8O;
[109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. 1966, 97, 1357-64.

VARIABLES:

Room Temperature: T/K = 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $YbBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.25 g per 100 ml of solution

or

6.05×10^{-3} mol dm^{-3} (compiler)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Ytterbium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $YbBr_3 \cdot 3C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $YbBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Ytterbium bromide; $YbBr_3$; [13759-89-2]

(2) 1,4-Dioxane; $C_4H_8O_2$; [123-81-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess. U. *Z. Chem.* 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II. Vses . Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room Temperature: T/K around 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $YbBr_3$ in p-dioxane at around 25°C was given as

1.7 mass %

The corresponding molality calculated by the compiler is

0.042 mol kg^{-1}

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Yb was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Ytterbium bromide; $YbBr_3$; [13759-89-2]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$YbBr_3$ solubility[a] mass %	mol kg^{-1}
1-propanamine;	n-C_3H_9N;	[107-10-8]	34.5	1.276
2-propanamine;	iso-C_3H_9N;	[75-31-0]	22.75	0.713
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	24.5	0.786
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	33.9	1.243
di-2-butylamine;	(sec-C_4H_9)$_2$NH;	[626-23-3]	0.4	0.0097

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.

COMPONENTS:

(1) Ytterbium iodide; YbI_3;
[13813-44-0]

(2) 1,4-Dioxane; $C_4H_8O_2$;
[123-81-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U.
Z. Chem. 1968, *8*, 472-3.

Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov.* 1971, 200-6.

VARIABLES:

Room Temperature: T/K around 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of YbI_3 in p-dioxane at around 25°C was given as

$$0.1_5 \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$0.0027 \text{ mol kg}^{-1}$$

The nature of the solid phase was not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Yb was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Ytterbium iodide; YbI_3; [13813-44-0]

(2) Alkyl amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			YbI_3 solubility[a] mass %	mol kg^{-1}
1-propanamine;	C_3H_9N;	[107-10-8]	1.7	0.031
2-propanamine;	iso-C_3H_9N;	[75-31-0]	1.2	0.022
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	0.4	0.007
2-butanamine;	iso-$C_4H_{11}N$;	[13952-84-6]	8.9	0.176

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Taylor, M.D.; Carter, C.P.
J. Inorg. Nucl. Chem. 1962, *24*, 387.

COMPONENTS:

(1) Lutetium chloride; $LuCl_3$; [10099-66-8]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sakharova, Yu.G.; Ezhova, T.A.

Zh. Neorg. Khim. 1976, *21*, 551-4; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1976, *21*, 296-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $LuCl_3.6H_2O$ in 96.8 % C_2H_5OH[a]

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	53.30	53.20	53.28	53.26	53.26	2.926
30	54.86	54.70	54.75	54.80	54.78	3.111
40	57.10	56.90	56.95	57.05	57.00	3.404
50	61.60	61.48	61.52	61.45	61.51	4.104
60	68.35	68.20	68.39	68.18	68.28	5.528

[a] It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

[b] Solubilities reported as grams of hexahydrate in 100 g of solvent.

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$LuCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 148.0 - 150.5°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.9% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS: (1) Lutetium chloride; $LuCl_3$; [10099-66-8] (2) Hexamethylphosphorotriamide; $C_6H_{18}N_3OP$; [680-31-9]	**ORIGINAL MEASUREMENTS:** Mikheev, N.B.; Kamenskaya, A.N. Konovalova, N.A.; Zhilina, T.A. *Zh. Neorg. Khim.* <u>1977</u>, *22*, 1761-6; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) <u>1977</u>, *22*, 955-8.
VARIABLES: Room temperature: T/K = 298 ± 3	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with the solvate $LuCl_3.3C((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.073 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{1/2}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $LuCl_3.3C_6H_{18}N_3OP$.

The solvate was analyzed for metal content by complexometric titration, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate were also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $LuCl_3$ prepared by modification of Taylor & Carter's method (1) by subliming NH_4Cl from a mixture of $LuCl_3$ with 6 moles of NH_4Cl in a stream of inert gas at 200-400°C. The product contained less than 3% of LuOCl.

The solvent was purified as in (2).

ESTIMATED ERROR:

Soly: precision ± 0.001 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* <u>1962</u>, *24*, 387.
2. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* <u>1975</u>, *11*, 163.

COMPONENTS:

(1) Lutetium chloride; $LuCl_3$; [10099-66-8]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

Lyubimov, E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

T/K = 293
Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4:POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	Lu_2O_3 solubility[a,b] moles Lu dm^{-3}
0	0	0.1
1:100	0.085	0.3
1:50	0.17	0.7 (0.1)
1:25	0.33	0.9
1:15	0.59	0.7
1:10	0.78	0.8

[a] Solutions preheated to 220°C. Value in parenthesis corresponds to preheating at 120°C.

[b] This is also the solubility of $LuCl_3$ in the $SnCl_4$-$POCl_3$ mixture because the oxide is quantitatively converted to the chloride according to

$$Lu_2O_3 + 6POCl_3 = 2LuCl_3 + 3P_2O_3Cl_4$$

Authors state the the solubility of $LuCl_3$ is enhanced by complex formation according to

$$2LuCl_3 + 3SnCl_4 = Lu_2(SnCl_6)_3$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Lu_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20° C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 220°C lowered the equilibration time at 20°C to 2 hours.

Lu was determined by colorimetric analysis or by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Lu_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES:

COMPONENTS:

(1) Lutetium bromide; $LuBr_3$; [14456-53-2]

(2) Tetrahydrofuran; C_4H_8O; [109-99-9]

ORIGINAL MEASUREMENTS:

Rossmanith, K.

Monatsh. Chem. 1966, 97, 1357-64.

VARIABLES:

Room Temperature: T/K = 294-296

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $LuBr_3$ in tetrahydrofuran at 21-23°C was reported to be

0.30 g per 100 ml of solution

or

7.2×10^{-3} mol dm^{-3} (compiler)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Lutetium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $LuBr_3.3C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $LuBr_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Br followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH_4Br.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

SYSTEM INDEX

Page numbers preceded by E refer to evaluation texts whereas those which are not refer to compiled tables.
Substances are listed by their Chemical Abstracts name, for example 2-ethoxyethanol appears as ethanol, 2-ethoxy- ; systems are indexed under the solid not under solvent.

REGISTRY NUMBER INDEX

Page numbers preceded by E refer to evaluation texts whereas those which are not refer to compiled tables.

AUTHOR INDEX

Page numbers preceded by E refer to evaluation texts whereas those which are not refer to compiled tables.