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SOLUBILITY DATA SERIES

Volume 11

ALKALI METAL, ALKALINE-EARTH METAL AND AMMONIUM HALIDES. AMIDE SOLVENTS

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Volume 11

ALKALI METAL, ALKALINE-EARTH METAL AND AMMONIUM HALIDES. AMIDE SOLVENTS

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Robert Maxwell Publisher at Pergamon Press

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 produces 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, thus obviously are superficial in treatment. Since they command a wide market, we believe that their service to advancement of science is at least questionable. Additionalyy, 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 1s 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 qualiry 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 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:

ting 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:

(1) 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 avialable, 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, developed during our four years of existence, 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 respon-sive than ever before to the needs of users. The self-regulatory message to scientists of 15 years ago to refrain from unnecessary publication has not achieved much. The literature is still, in 1979, cluttered with poor-quality articles. The Weinberg report (in "Reader in Science Information", Eds. 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 pro-It should help to dispel confusion in the minds of many authors of ject. 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.

August, 1979

A. S. Kertes

PREFACE TO THE VOLUME

This volume surveys the solubility data available in the literature of the halides and pseudohalides of the alkali, alkali earth and ammonium ions in the six simple amides, formamide, N-methylformamide, N,N-dimethylformamide, acetamide, N-methylacetamide and N,N-dimethylacetamide.

These solvents are of both theoretical and practical interest, primarily because of their ability to dissolve significant quantities of electrolytes. With the exception of acetamide, the amides are liquid at room temperature and have liquid ranges of over 175°. All the solvent molecules are characterised by large dipole moments and, apart from the two di-N-substituted forms, have exceptionally high dielectric constants. There have been many physicochemical studies of electrolytic solutions in the amides. These have concentrated mainly on ionic solvation and mobility, thermodynamics and electrochemistry. Despite such investigations few comprehensive studies of solubility have been reported. The number of reliable data available is further restricted by the fact that many of these measurements were not undertaken with sufficient care in the use of procedures for purifying salt and/or solvent, in controlling the temperature and in selecting the most suitable analytical methods. In evaluating the various systems, particular attention was directed (i) to the techniques and procedures used in the purification of solvents and salts, and (ii) to the length of time allowed to ensure saturation and to the methods used to detect that equilibrium had been established.

All the solvents, when pure, are fairly stable - the acetamide series being more so than the formamides. They are, however, generally sensitive to heat, light and water, and to obtain them in reasonably pure state requires elaborate procedures and particular care. Decomposition of the formamides is particularly susceptible to traces of water impurity and is both acid- and base- catalysed. It is well known that a number of the salts investigated, such as lithium and alkali earth halides also require special methods in order to obtain them in a pure, dry state. A further problem that may arise when traces of water are present is preferential ionic solvation by water which may in turn give rise to overestimates of the solubility.

The following points have governed the general nature of this volume:

(i) The majority of quantitative solubility studies have been of fairly soluble salts. Since activity coefficients were generally not available for such concentrated solutions, few thermodynamic solubility products have been reported.

(ii) Only rarely was an analysis of the solid phase carried out and therefore no attempt has been made to provide a general discussion of the solid-liquid equilibria in this volume. Indeed many of the solubilities reported may have been considerably affected by undetected solvate formation.

(iii) The absence of reliable density data has not allowed the conversion of results reported in molar units into molal units and vice versa (in accordance with the recommendations and guidelines of the Solubility Data Project directorate). This has had the effect of reducing the possibilities of intercomparison for the evaluation and recommendation of solubility values.

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(iv) So far as we are aware, the entire literature has been covered in this survey. In a few cases it has not been possible to obtain copies of original papers published in the U.S.S.R. and data from these sources have not been compiled nor evaluated.

(v) It was not generally possible to find replicate data within a single study so that an independent analysis of the error could be undertaken. In a number of cases the authors' estimated error is given. In others approximate methods have been used to estimate the error, such as by the scatter of values of the logarithm of solubility when plotted against the reciprocal of the absolute temperature. Where clearly no serious attempt was made to obtain satisfactory anhydrous conditions, we have considered that an error, generally estimated as of the order of a few per cent, was likely. An error of similar magnitude was also attributed to data obtained after equilibration times considered too short to have allowed a saturation equilibrium to have been achieved. (In general dissolution is a particularly slow process in the amide systems, especially at low temperature).

(vi) All the above considerations were taken into account in the selection of the most reliable data for the solubility values. In a number of systems, however, no data could be recommended with confidence and only tentative values, very approximate estimates or even no values are given. In reporting the experimental methods and in the compilations, the units used by the original investigators are usually retained. In the critical evaluations the values have been converted to S.I. units.

Finally we would like to acknowledge all those who have contributed to the final version of this volume. In particular we thank C.M. Criss, J.W. Lorimer, G.H. Nancollas, M.Salomon and P.G. Sears for their valuable and helpful comments and suggestions.

August, 1979

B. ScrosatiC. A. Vincent

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:

 $x_{B} = n_{B} / \sum_{i=1}^{D} n_{i}$

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:

$$w_{\rm B} = m'_{\rm B} / \sum_{i=1}^{\rm C} m'_{i}$$
⁽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):

$$x_{S,B} = n_{B} \sum_{i=1}^{C} n_{i} = x_{B} \sum_{i=1}^{C} x_{i}$$
(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.

(1)

$$m_B = n_B / n_A M_A$$

SI base units: mol kg^{-1} (4)

where M_A is the molar mass of the solvent.

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

$$c_{B} = [B] = n_{B}^{V}$$
 SI base units: mol m⁻³

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)

(5)

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 $t^{\circ}C$, 1 atm divided by the density of water at $t^{\circ}C$, 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 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

 $RT \ln(f_B x_B) = \mu_B - \mu_B^*$ (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,

$$\lim_{B \to 1} f_{B} = 1$$
(8)

(b) Solutions.

by

(i) Solute substance, B. The molal activity coefficient
$$\gamma_{\rm B}$$
 is given

$$RT \ln(\gamma_B m_B) = \mu_B - (\mu_B - RT \ln m_B)^{\infty}$$
(9)

where the superscript $\stackrel{\infty}{}$ indicates an infinitely dilute solution. For any solute B,

$$\gamma_{\rm B}^{\infty} = 1 \tag{10}$$

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):

$$\gamma_{B} = x_{A} f_{x,B} = V_{A}^{*} (1 - \sum_{s} c_{s}) \gamma_{B}$$
(11)

or

$$f_{x,B} = (1 + M_A \sum_{s} m_s) \gamma_B = V_A^* Y_B / V_m$$
(12)

or

$$y_{B} = (V_{A} + M_{A_{S}} \Sigma_{S} V_{S}) \gamma_{B} / V_{A}^{*} = V_{m} f_{x,B} / V_{A}^{*}$$
 (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 Ξ $C_{\nu+}A_{\nu-}$, the molal activity is replaced by (9)

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

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

$$f_{x,B} x_{B} = v_{+}^{v_{+}} v_{-}^{v_{-}} f_{\pm}^{v_{x}} x_{\pm}^{v}$$
(15)

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

$$x_{+} = v_{+}x_{B}/[1+(v-1)x_{B}]; \qquad x_{-} = v_{-}x_{B}/[1+(v-1)x_{B}]$$
(16)

(ii) Solvent, A:

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

$$\phi = (\mu_A^* - \mu_A) / RT M_A \sum_{s=s}^{s} m_s$$
(17)

where μ_A^* is the chemical potential of the pure solvent. The *hational osmotic coefficient*, ϕ_x , is defined as (1):

$$\phi_{\mathbf{x}} = (\mu_{\mathbf{A}} - \mu_{\mathbf{A}}^{*}) / RT \ell n \mathbf{x}_{\mathbf{A}} = \phi M_{\mathbf{A}_{\mathbf{S}}} \sum_{\mathbf{S}}^{\Sigma m} / \ell n (1 + M_{\mathbf{A}_{\mathbf{S}}} \sum_{\mathbf{S}}^{\Sigma m})$$
(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:

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

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

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

$$d\mu_{i} = \sum_{j=2}^{\Sigma} G_{ij} dx_{j} - S_{i} dT + V_{i} dp$$
(21)

where (7)

.

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

where

$$H_{i}-H_{i}' = T(S_{i}-S_{i}')$$
 (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) Sqlubility 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

$$(1/x_{B}-n/x_{A}) \{1+(\frac{\partial \ln f_{B}}{\partial \ln x_{B}})_{T,P}\} dx_{B} = (nH_{A}+H_{B}-H_{AB}) dT/RT^{2}$$
(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):

$$RT \ln f_{\rm B} = w x_{\rm A}^2$$
 (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

$$nH_{A} + H_{B} - H_{AB}^{*} = \Delta H_{AB} + n(H_{A} - H_{A}^{*}) + (H_{B} - H_{B}^{*})$$
$$= \Delta H_{AB} + w(nx_{B}^{2} + x_{A}^{2})$$
(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

$$R d \ln\{x_{B}(1-x_{B})^{n}\} = -\Delta H_{AB} d(\frac{1}{T}) - w d(\frac{x_{A}^{2}+nx_{B}^{2}}{T})$$
(28)

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

where ΔC_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

$$\ln\{x_{\rm B}(1-x_{\rm B})^{\rm n}\} = A_1 + A_2/T + A_3 \ln T + A_4 (x_{\rm A}^{2} + nx_{\rm B}^{2})/T$$
(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

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

and (27) becomes

$$nH_{A} + H_{B} - H_{AB} = (nH_{A} + H_{B} - H_{AB}) + n(H_{A} - H_{A} +) + (H_{B} - H_{B}) = \Delta H_{AB} + w(nx_{B}^{2} + x_{A}^{2} - 1)$$
(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 $\Delta H_{AB}^{\infty}(T^*)$, $\Delta C_p(T^*)$ replacing ΔH_{AB}^{*} and ΔC_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

$$\mathbb{R}\nu(1/x_{B}-n/x_{A})\{1+(\partial \ln f_{\pm}/\partial \ln x_{\pm})_{T,P}\}dx_{B}/\{1+(\nu-1)x_{B}\}$$

$$= \{\Delta H_{AB}^{\omega} + n(H_{A} - H_{A}^{*}) + (H_{B} - H_{B}^{\omega})\}d(1/T)$$
(33)

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

$$\ln\{\frac{x_{B}^{\nu}(1-x_{B})^{n}}{1+(\nu-1)x_{D}^{-}n+\nu}\} = \ln\{\frac{n^{n}}{(n+\nu)^{n+\nu}}\} - \{\frac{\Delta H_{AB}^{\infty}(T^{*}) - T^{*}\Delta C_{D}^{*}}{R}\} (\frac{1}{T} - \frac{1}{T^{*}}) + \frac{\Delta C^{*}}{R}\ln(T/T^{*})$$

A similar equation (with v=2 and without the heat capacity terms) has been used to fit solubility data for some MOH=H₂O 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:

$$\mu_{A_{n}B}^{*} = \mu_{A_{n}B}(sln) = n\mu_{A} + \mu_{B}$$
(35)
= $(n\mu_{A}^{*} + \nu_{+}\mu_{+}^{\omega} + \nu_{-}\mu_{-}^{\omega}) + nRT \ln f_{A}x_{A}$
+ $\nu RT \ln \gamma_{+}m_{+}Q_{+}$ (36)

for a salt hydrate $A_n B$ which dissociates to water, (A), and a salt, B, one mole of which ionizes to give v_+ cations and v_- 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 $K_{s_0}^0$ in

$$\Delta G^{\infty} \equiv (\nu_{+}\mu_{+}^{\omega} + \nu_{-}\mu_{-}^{\omega} + n\mu_{A}^{\star} - \mu_{AB}^{\star})$$

$$= -RT \ln K_{S0}^{0}$$

$$= -RT \ln Q^{\nu}\gamma_{+}\nu_{m_{+}}^{\nu}m_{-}^{\nu}$$
(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_{go}^0 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 $C_{v} + A_{v} - nH_2O$ in the presence of other solutes is given by eqn (36) as

$$v \ln\{m_{\rm B}/m_{\rm B}(0)\} = -v \ln\{\gamma_{+}/\gamma_{+}(0)\} - n \ln(a_{\rm H_{2}O}/a_{\rm H_{2}O}(0))$$
(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 nonstoichiometric 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, selfexplanatory. 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

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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 $a\ell\ell$ 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.

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	R. Cohen-Adad, Villeurbanne, France J.W. Lorimer, London, Canada M. Salomon, Fair Haven, New
	August, 1979 Jersey, U.S.A.

COMPONENTS: (1) Lithium chloride; LiCl; [7447-41-8] (2) Formamide; CH ₃ NO; [75-12-7]	EVALUATOR: C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.
CRITICAL EVALUATION: Two investigations of the solubility of other measurements of salt solubility in thi	LiCl in formamide have been reported. As in s solvent, the accuracy of the data is likely

to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used so far in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹. It is likely that most of the solutions studied had water contents in the range 0.05 to 0.5 mol dm⁻³. Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility. In neither investigation was the recommended method of drying the LiC1 (by passing dry HC1) used, so that the purity of the solute is unknown.

Solubility at 298 K has been determined by Paul et al. (2) (250.6 g/kg of solvent) and by Berardelli et al. (3) (282 g/kg of solvent). Both groups of workers used purified solvent and took precautions to work in dry conditions. While the solvent used by Paul et al. (2) was probably the purer, no details are given of their analytical methods. The mean value of the two determinations is therefore recommended as a tentative value.

Tentative value at 298 K

266 - 16 g/kg of solvent

Tentative value at 308 K and 318 K

Paul et al. (2) measured the solubility of LiCl in formamide at 308 K and 318 K. The results reported may be taken as tentative values:

<u>308 K</u>

211 [±] 11 g/kg of solvent

318 K

185 - 10 g/kg of solvent.

References

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3. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. *Ric. Sci.*, <u>1968</u>, 38, 814-819.

1

For	namide
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Lithium chloride; LiC1;[7447-41-8] Formamide; CH₃NO; [75-12-7] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	. I
t/ ^o C	g/100 g solvent
25	25.1
35	21.1
45	18.5
AUXILIAR	2 INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
METHOD/APPARATUS/PROCEDURE: Saturated solutions of lithium chloride were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 $^{\circ}$ C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for chloride. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were	SOURCE AND PURITY OF MATERIALS: A.R. grade lithium chloride (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10 ⁻⁵ S m ⁻¹ was obtained. The solvent was stored in the dark.
METHOD/APPARATUS/PROCEDURE: Saturated solutions of lithium chloride were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for chloride. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were	SOURCE AND PURITY OF MATERIALS: A.R. grade lithium chloride (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10 ⁻⁵ S m ⁻¹ was obtained. The solvent was stored in the dark.
METHOD/APPARATUS/PROCEDURE: Saturated solutions of lithium chloride were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 $^{\circ}$ C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for chloride. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were	SOURCE AND PURITY OF MATERIALS: A.R. grade lithium chloride (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10 ⁻⁵ S m ⁻¹ was obtained. The solvent was stored in the dark. ESTIMATED ERROR:
METHOD/APPARATUS/PROCEDURE: Saturated solutions of lithium chloride were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 $^{\circ}$ C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for chloride. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were	SOURCE AND PURITY OF MATERIALS: A.R. grade lithium chloride (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements_1 A conductance of 5 x 10 ⁻⁵ S m ⁻¹ was obtained. The solvent was stored in the dark. ESTIMATED ERROR: Solubility ± 1%
METHOD/APPARATUS/PROCEDURE: Saturated solutions of lithium chloride were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 $^{\circ}$ C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for chloride. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a	SOURCE AND PURITY OF MATERIALS: A.R. grade lithium chloride (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10 ⁻⁵ S m ⁻¹ was obtained. The solvent was stored in the dark. ESTIMATED ERROR: Solubility ± 1% Temperature ±0.05 °C (author) REFERENCES: 1. Paul, R.C.; Singla, J.P.; Gill, D.S.;
METHOD/APPARATUS/PROCEDURE: Saturated solutions of lithium chloride were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for chloride. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were	SOURCE AND PURITY OF MATERIALS: A.R. grade lithium chloride (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements_1 A conductance of 5 x 10 ⁻⁵ S m ⁻¹ was obtained. The solvent was stored in the dark. ESTIMATED ERROR: Solubility ± 1% Temperature ±0.05 °C (author) REFERENCES:

OMPONENTS: (1) Lithium chloride; LiCl; [7447-41-8] (2) Formamide; CH ₃ NO; [75-12-7]	ORIGINAL MEASUREMENTS: Berardelli, M.L.; Pistoia, G.;	
	Remandalli VI + Distais C -	
(2) Formamide; CH ₃ NO; [75-12-7]	berarderii, M.L.; ristola, G.;	
J	Polcaro, A.M.	
	Ric. Sci., <u>1968</u> , 38, 814-819.	
ARIABLES:	PREPARED BY:	
One temperature: 25 °C	J.S. McKechnie	
Solubility of lithium chlor 28.2 g/100 g solv		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated lithium chloride solutions were prepared by dissolving excess of the halide in about 20-25 cm³ of solvent contained in a 50 cm³ Pyrex flask with a standard taper joint. The solution was then stored for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT 1) and, presumably, standard aqueous silver nitrate.

Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of formamide to aqueous solution (1:5).

The equivalence point remained the same (1).

SOURCE AND PURITY OF MATERIALS:

Lithium chloride, reagent grade, was dried under high vacuum before use. Commercial formamide (C. Erba, RP Product) was purified by storing over CaO for 2 days before twice distilling under reduced pressure. The conductance of the formamide varied from 0.9 to 1.40×10^{-4} S m⁻¹.

ESTIMATED ERROR:

Solubility

Temperature

REFERENCES:

 Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, 1967, 37, 1167-1172

± 1%

± 0.05 °C (author)

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COMPONENTS :	EVALUATOR:		
(1) Lithium bromide; LiBr; [7550-35-8] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978		
CRITICAL EVALUATION:			
Two investigations of the solubility of LiBr in formamide have been reported. As in			
other measurements of salt solubility in this solvent, the accuracy of the data is likely			
to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and			

hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below $2 \times 10^{-5} \text{ S m}^{-1}$. The most stringent procedure used so far in solubility studies has been that of Paul et al. (2) who obtained a conductance of $5 \times 10^{-5} \text{ S m}^{-1}$. It is likely that most of the solutions studied had water contents in the range 0.05 to 0.5 mol dm⁻³. Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility.

Solubility at 298 K has been determined by Paul et al. (2) (454.5 g/kg of solvent) and by Berardelli et al. (3) (738 g/kg of solvent). As both groups of workers used relatively pure solvent and dry conditions, there is no obvious explanation for this large discrepancy. The mean of these two determinations might be proposed as a very tentative value. Paul et al. (2) also measured the solubility at 308 K and 318 K: these are the only values available and should be taken as tentative.

<u>Tentative values</u> <u>298 K</u> 600 [±] 150 g/kg of solvent <u>308 K</u> 583 [±] 150 g/kg of solvent <u>318 K</u> 759 [±] 150 g/kg of solvent <u>References</u> 1. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), <u>1966</u>, 362-366

- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 3. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819.

Formamide

Formamide		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Lithium bromide; LiBr; [7550-35-8] (2) Formamide, CH ₃ NO; [75-12-7]	Paul, R.C.; Singla, J.P.; Lambda, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11,1024-1026	
VARIABLES:	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:		
t/°C	g/100 g solvent	
25	45.5	
35	58.3	
45	75.9	
	INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Saturated solutions of lithium bromide were prepared by adding excess of the powdered	A.R. grade lithium bromide (BDH) was re- prystallised, powdered and dried in vacuum at	
salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 $^{\circ}$ C	110-125 ^o C. Formamide (Reidel Pure) was distilled under	
above the temperature of the bath. The attainment of the equilibrium was checked	reduced pressure after extensive prior purification (1). Purity was checked by	
by intermittent analyses of the solutions.	conductance and density measurements. A	
The solutions were then filtered under nitrogen and analysed for chloride.	conductance of 5×10^{-5} S m ⁻¹ was obtained. The solvent was stored in the dark.	
No details of analytical methods were given. Transference of material, as		
far as possible, was carried out in a nitrogen filled dry box. Measurements	ESTIMATED ERROR:	
were made in triplicate.	Solubility ± 1%	
	Temperature ± 0.05 ^O C (author)	
	REFERENCES:	
	 Paul, R.C.; Singla, J.P.; Gill, D.S.; Narula, S.P. 	
	J. Chem. Soc., Dalton Trans., <u>1972</u> , 522–524.	

6 Forma	amide
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium bromide; LiBr; [7550-35-8] (2) Formamide; CH ₃ NO; [75-12-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of lithium bromide in for 73.8 g/ 100 g solvent.	mamide at 25 °C
AIIXTLIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated lithium bromide solutions were prepared by dissolving excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide	Lithium bromide, reagent grade, was dried under high vacuum before use. Commercial formamide (C. Erba, RP Product) was purified by storing over CaO for 2 days before twice distilling under reduced pressure. The conductance of the formamide varied from 0.9 to 1.40 x 10 ⁻⁴ S m ⁻¹ .
determined by a potentiometric method using a titrimeter (type Radiometer TTT 1) and, presumably, standard aqueous silver nitrate. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same	Solubility±1%Temperature±0.05°C (author)REFERENCES:1. Pistoia, G.; Pecci, G.; Scrosati, B.
value for dissolved halide. The applicability of the method was checked by increasing the ratio of formamide to aqueous solution (1:5).	<i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1167-1172

The equivalence point remained the same (1).

COMPONENTS:	EVALUATOR:
(1) Sodium fluoride; NaF; [7681-49-4] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

CRITICAL EVALUATION:

One investigation of the solubility of NaF in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. In this investigation of NaF, no attempt was made to dry or purify the solvent. Since preferential solvation of the ions by water is probable, the value given probably represents the higher limit of solubility.

Tentative value at 298 K

 $1.1 \stackrel{+}{-} 0.1 \text{ g dm}^{-3}$ of saturated solution

References

1. Colton, E.; Brooker, R.E. J. Phys. Chem., <u>1958</u>, 62, 1595-1596.

2. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366

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Formamide

Tomanide		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Sodium fluoride; NaF; [7681-49-4]	Colton, E.; Brooker, R.E.	
(2) Formamide, CH ₃ NO; [75-12-7]	J. Phys. Chem., <u>1958</u> , 62, 1595-1596	
VARIABLES:	PREPARED BY:	
One temperature: 25 ^o C	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of sodium fluoride in formamide at 25 ^O C		
1.1 g/1000 cm ³ of saturated solution.		

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of sodium fluoride in formamide were prepared in glass containers so that an excess of solid was always present. After tumbling for a minimum of 48 hours in a thermostatted water bath the solutions were filtered quickly and aliquots taken for analysis. The sodium was determined photometrically after decomposing the formamide. A Beckman DU spectrophotometric was used.	Reagent grade sodium fluoride. Commercial formamide (Fisher Scientific Co.) with a freezing point of 2.50 ^O C was used without further purification.
	ESTIMATED ERROR: Solubility ⁺ 5% (compiler) Temperature ⁺ 1 ^O C (author)
	REFERENCES:

COMPONENTS :	EVALUATOR:
(1) Sodium chloride; NaCl; [7647-14-5] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Four investigations of the solubility of NaCl in formamide have been reported. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm^{-3} and the conductance to below $2 \times 10^{-5} \text{ s m}^{-1}$. The most stringent procedure used so far in solubility studies has been that of Paul et al. (2) who obtained a conductance of $5 \times 10^{-5} \text{ s m}^{-1}$. It is likely that most of the solutions studied had water contents in the range 0.05 to 0.5 mol dm^{-3} . Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility.

The solubility reported by Colton and Brooker (3), a value of 99 g dm⁻³ of saturated solution at 298 K, cannot be compared with the molal solubilities given by the other workers since densities were not measured. However this result is likely to be high since the solvent was not purified and may have contained significant quantities of water.

Solubilities at 298 K determined by Paul et al. (2) (94.4 g/kg of solvent), Gopal and Husain (4) (93.2 g/kg of solvent) and by Berardelli et al. (5) (93.8 g/kg of solvent) are in good agreement. They have a mean value of 93.8 g/kg of solvent with a standard deviation of 0.8 g/kg of solvent, or just under 1%. There seems no good reason to favour one value over the others, and the mean value is therefore recommended.

Recommended value at 298 K

93.8 - 0.8 g/kg of solvent

Values at other temperatures

Both Paul et al. (2) and Gopal et al. (4) report solubilities at higher temperatures. Agreement between the two sets of results is good. In order to utilise all the data, a regression analysis of all the values in (2), (4) and (5) was undertaken in which the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. A correlation coefficient of -0.980 was found and from the analysis the following values are recommended.

Recommended value at 308 K

89.1 ⁺ 1.0 g/kg of solvent Recommended value at 318 K

84.8 - 1.0 g/kg of solvent

References

- 1. Notley, J.M.; Spiro, M., J. Chem. Soc. (B), 1966, 362-366.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 3. Colton, E.; Brooker, R.E. J. Phys. Chem., <u>1958</u>, 62, 1595-1596
- 4. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963, 40,272-274
- 5. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819.

0 Forma	amide
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; [7647-14-5] (2) Formamide; CH ₃ NO; [75-12-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, ¹⁰ 24-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C 25 35 45	g/100 g solvent 9.44 8.85 8.32
METHOD /APPARATUS /PROCEDURE :	INFORMATION SOURCE AND PURITY OF MATERIALS:
Saturated solutions of sodium chloride were prepared by adding excess of the powdered salt to formamide $(15-20 \text{ g})$ in Pyrex tubes $(15 \pm 2.5 \text{ cm})$ and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for chloride. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a	A.R. grade sodium chloride (BDH) was recrystallised, powdered and dried in vacuum at 110-125°. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10° S m ⁻¹ was obtained. The solvent was stored in the dark.
nitrogen filled dry box. Measurements were made in triplicate.	ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.05 °C (author)
	REFERENCES: 1. Faul, R.C.; Singla, J.P.; Gill, D.S.; Narula, S.P. J. Chem. Soc., Dalton Trans., 1972, 522-524.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; [7647-14-5] (2) Formamide; CH ₃ NO; [75-12-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.,<u>1968</u>, 38,</i> 814-819
VARIABLES:	PREPARED BY:
One Temperature: 25 ^O C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium chloride in formamide at 25 °C.

9.38 g/100 g solvent.

AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: Considerable effort was made to obtain water Sodium chloride, reagent grade, was dried free solutions and all the solutions were under high vacuum before use. Commercial formamide (C. Erba, RP Product) prepared in a dry box. Saturated sodium chloride solutions were prepared by dissolywas purified by storing over CaO for 2 days before twice distilling under reduced ing excess of the halide in about 20-25 cm pressure. of solvent contained in a 50 cm Pyrex flask The conductance of the formamide varied from 0.9 to 1.40 x 10⁻⁴ with a standard taper joint. The solution S m⁻¹. was then stored for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentio-ESTIMATED ERROR: metric method using a titrimeter (type ± 1% Solubility Radiometer TTT 1) and, presumably, standard aqueous silver nitrate. \pm 0.05 °C (author) Temperature Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive REFERENCES: periods of time, gave the same value for 1. Pistoia, G.; Pecci, G.; Scrosati, B. dissolved halide. The applicability of the method was checked by increasing the ratio Ric. Sci., 1967, 37, 1167-1172 of formamide to aqueous solution (1:5). The equivalence point remained the same (1).

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2 Forma	amide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; [7647-14-5] (2) Formamide; CH ₃ NO; [75-12-7]	Gopal, R.; Husain, M.M. J. Ind. Chem. Soc., <u>1963</u> , 40,272-274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
.0 .	
t/ ^o C	g/100 g solvent
25	9.32
30	9.05
35	8.91
40 45	8.71
45 50	8.55 8.42
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of NaCl were prepared at $10-15^{\circ}$ above the temperature at which the	A.R. grade sodium chloride.
solubility was to be measured before thermostatting at the required temperature. Aliquots $(3-5 \text{ cm}^3)$ were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P_2O_5 . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) Evaporation Method - Weighed samples were carefully heated to expel the solvent.	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner.	ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 ^o C (compiler)
(2) Precipitation Method - Weighed samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure was tested using known amounts of KCI in formamide and estimating gravimetrically. When experimental and theoretical values agreed within ±0.5% the procedure was applied to the samples of unknown solubility.	REFERENCES:

Formamide 13		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Sodium chloride; NaCl; [7647-14-5]	Colton, E.; Brooker, R.E.	
(2) Formamide; CH ₃ NO; [75-12-7]	J. Phys. Chem., <u>1958</u> , 62, 1595-1596	
5		
VARIABLES :	PREPARED BY:	
One temperature: 25 ^o C	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of sodium chloride ir	formamide at 25 $^{\circ}$ C	
99 g/1000 cm ³ of saturat		
99 g/1000 cm of saturat		
	INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Saturated solutions of sodium chloride in formamide were prepared in glass containers	Reagent grade sodium chloride.	
so that an excess of solid was always present	Commercial formamide (Fisher Scientific Co).	
After tumbling for a minimum of 48 hours in a thermostatted water bath the solutions	with a freezing point of 2.50 °C was used	
were filtered quickly and aliquots taken for analysis.	without further purification.	
The sodium was determined photometrically		
after decomposing the formamide. A Beckman DU spectrophotometric was used.		
l	ESTIMATED ERROR:	
	Solubility + 5% (compiler)	
	Temperature ⁺ 1 ^O C (author)	
	REFERENCES:	

	COMPONENTS :	EVALUATOR:
	(1) Sodium bromide; NaBr; [7647-15-6] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews St. Andrews, Fife, Scotland. December 1978

CRITICAL EVALUATION:

Five investigations of the solubility of NaBr in formamide have been reported. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used so far in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹. It is likely that most of the solutions studied had water contents in the range 0.05 to 0.5 mol dm⁻³. Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility.

The solubility reported by Colton and Brooker (3) (a value of 439 g dm⁻³ of saturated solution at 298 K, which is equivalent to approximately 430 g/kg of solvent) is very much higher than that found by other workers. This result is rejected since no purification of the solvent was attempted.

Solubilities at 298 K determined by Paul et al. (2) (348.9 g/kg of solvent), Pavlopoulos and Strehlow (4) (304.7 g/kg of solvent), Gopal and Husain (5) (358.4 g/kg of solvent) and Berardelli et al. (6) (353 g/kg of solvent) have a mean value of 341.3 g/kg of solvent with a standard deviation of 25 g/kg of solvent, or just over 7%. By rejecting the value of Pavlopoulos and Strehlow on statistical grounds, a mean value of $353.4 \stackrel{+}{-} 5.0$ g/kg of solvent is found. This is consistent with the calculated value of 354.7 g/kg determined using a linear regression analysis of the logarithms of solubilities between 298 K and 323 K as a function of the reciprocal of the absolute temperature. Recommended value at 298 K

353.4 - 4.8 g/kg of solvent

Tentative values at other temperatures

Both Paul et al. (2) and Gopal and Husain (5) report solubilities at higher temperatures. Agreement between the two sets of values is fair. While the data of Paul et al. may be slightly more reliable at higher temperatures since their purer solvent would be less liable to decomposition, no information is available concerning their analytical procedures. In order to utilise all the variable temperature data available a regression analysis of all the values given in (2), (5) and (6) was undertaken in which the logarithm of the solubility was assumed to be a linear function of the inverse absolute temperature. A correlation coefficient of -0.982 was found. From this analysis the following tentative values at 308 K and 318 K are given.

<u>308 K</u>

387 ⁺ 5 g/kg of solvent <u>318 K</u> 421 ⁺ 6 g/kg of solvent.

(continued)

COMPONENTS:	EVALUATOR:
 Sodium bromide; NaBr; [7647-15-6] Formamide; CH₃NO; [75-12-7] 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife,
_	Scotland. December 1978
CRITICAL EVALUATION: (continued)	December 1978
References	
1. Notley, J.M.; Spiro, M. J. Chem. Soc. (B)	. 1966. 362-366
2. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem.,	
<u>1973, 11, 1024-1026</u>	
3. Colton, E.; Brooker, R.E. J. Phys. Chem.	
4. Pavlopoulos, T.; Strehlow, H. Z. Phys. C	
5. Gopal, R.; Husain, M.M. J. Indian Chem.	
6. Berardelli, M.L.; Pistoia, G.; Polcaro,	A.M. Ric. Sci., 1968, 38, 814-819.

16 Form	amide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; [7647-15-6] (2) Formamide; CH ₃ NO; [75-12-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P.
	Indian J. Chem., <u>1973</u> , 11, 1024–1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/°c	g/100 g solvent
25	34.9
35	38.1
45	41.2
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of sodium bromide were prepared by adding excess of the powdered salt to formamide $(15-20 \text{ g})$ in Pyrex tubes $(15 \times 2.5 \text{ cm})$ and heating to nearly 10 °C. above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for bromide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a	A.R. grade sodium bromide (BDH) was recrystallised, powdered and dried in vacuo at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5×10^{-5} S m was obtained. The solvent was stored in the dark.
nitrogen filled dry box. Measurements	ESTIMATED ERROR:
were made in triplicate.	Solubility ± 1% Temperature ± 0.05 ^o C (author)
	REFERENCES: 1. Paul, R.C.; Singla, J.P.; Gill, D.S.; Narula, S.P. J. Chem. Soc., Dalton Trans., <u>1972</u> ,522-524.

Formamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; [7647-15-6] (2) Formamide; CH ₃ NO; [75-12-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium bromide in f	ormamide at 25 ⁰ C.
35.3 g/ 100 g solvent	
	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water free solutions and all the solutions were	Sodium bromide, reagent grade, was dried under high vacuum before use.
prepared in a dry box. Saturated sodium bromide solutions were prepared by dissolving	Commercial formamide (C. Erba, RP Product)
excess of the halide in about 20-25 cm ³ of	before twice distilling under reduced
solvent contained in a 50 cm^3 Pyrex flask with a standard taper joint. The solution	pressure. The conductance of the formamide varied from 0.9 to $1.40 \times 10^{-4} \text{ S m}^{-1}$.
was then stored for three or more days in a thermostatted bath. With the aid of a	
syphon provided with a terminal G-3 gooch.	
an aliquot of the saturated solution was diluted with water (1:20) and the amount	
of dissolved halide determined by a potentiometric method using a titrimeter	ESTIMATED ERROR:
(type Radiometer TTT 1) and, presumably, standard aqueous silver nitrate.	Solubility ± 1%
Equilibrium between solid and solution	Temperature ± 0.05 ^O C (author)
phases was considered to be attained when analysis of the solution, made at progressive	REFERENCES :
periods of time, gave the same value for dissolved halide. The applicability of	1. Pistoia, G.; Peccí, G.; Scrosati, B.
the method was checked by increasing the	Ric. Sci., 1967, 37, 1167-1172
ratio of formamide to aqueous solution (1:5).	
The equivalent point remained the same (1).	
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18 Form	amide
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; [7647-15-6]	Gopal, R.; Husain, M.M.
(2) Formamide; CH ₃ NO; [75-12-7]	J. Ind. Chem. Soc., <u>1963</u> , 40, 272–274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	I
t/ [°] C	g/100 g solvent
25	35.8
30	38.0
35	39.1
40	40.1
45	42.4
50	44.1
AIIXTLTARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of NaBr were prepared at	
10-15 ⁰ above the temperature at which the solubility was to be measured before	A.R. grade sodium bromide. Commercial formamide was redistilled
thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₀ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent.	and stored out of contact with light in amber-coloured bottles until used.
The samples were then cooled and weighed in a dry atmosphere. The process was	ESTIMATED ERROR:
repeated to obtain a constant weight. All three samples taken were treated in a similar manner.	Solubility ± 1% Temperature ± 0.1 °C (compiler)
(2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with	DEPERFUSING .
water and the halide content was determined	REFERENCES :
gravimetrically. Reliability of procedure was tested using known amounts of KCl in formamide and estimating gravimetrically. When experimental and theoretical values agreed within ±0.5% the procedure was	
applied to the samples of unknown solubility.	

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; [7647-15-6]	Colton, E.; Brooker, R.E.
(2) Formamide; CH ₃ NO; [75-12-7]	J. Phys. Chem., <u>1958</u> , 62, 1595-1596
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	1
Solubility of sodium bromide in formamide at 25 ^O C	
439 g/1000 cm ³ of saturated	solution
AUXILIARY INFORMATION	
ME THOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of sodium bromide in formamide were prepared in glass containers	Reagent grade sodium bromide.
so that an excess of solid was always	Commercial formamide (Fisher Scientific Co) with a freezing point of 2.50 °C was used
present. After tumbling for a minimum of 48 hours in a thermostatted water bath the	with a freezing point of 2.50° c was used without further purification.
solutions were filtered quickly and aliquots taken for analysis. Duplicate samples were	without further purification.
run.	
The sodium was determined photo-	
metrically after decomposing the formamide. A Beckman DU spectrophotometer was used.	
	ESTIMATED ERROR: Solubility ± 5% (compiler)
1	Solubility <u>-</u> 5% (compiler) Temperature <u>+</u> 1 ^o C (author)
	REFERENCES :
]	

) Formamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; [7647-15-6]	Pavlopolous, T.; Strehlow, H.
(2) Formamide; CH ₃ NO; [75-12-7]	2. Phys. Chem. (Wiesbaden), <u>1954</u> , 2, 89-103
VARIABLES :	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium bromide in fo	rmamide at 25 °C
340 g/1000 cm ³ of saturat	ed solution.
	······
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Water free solutions (\$ 0.005%) were prepared in a special glass apparatus equipped with a	240 °C for 4-5 hours in a furnace.
magnetic stirrer which was placed in a thermostatted bath for several hours. The	Formamide (Merck) was dried over P ₂ 0 ₅ and twice distilled, using a long column of glass
saturated solution was then forced out with dry air through a sinter, into a 50 cm^3	helices, before use. The water content of the solvent was determined by Karl Fischer
measuring flask and weighed. The air was	titration.
dried over CaCl ₂ . A known volume was evaporated to dryness, redissolved in water	
and the halogen content determined potentiometrically. No details of the cell	
were given (1). The results obtained were checked by density measurements.	
include by denotely medicatementer.	ESTIMATED ERROR:
	Solubility $\frac{1}{7}$
	Temperature ⁺ 0.1 ^o C (author)
	REFERENCES:
	1. Pavlopoulos, T.; Strehlow, H. Z. Phys. Chem. (Leipzig), <u>1953</u> , 202,
Į	474-479.
1	

COMPONENTS:	EVALUATOR:
(1) Sodium iodide; NaI; [7681-82-5] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

Four investigations of the solubility of NaI in formamide have been reported. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used so far in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹. It is likely that most of the solutions studied had water contents in the range 0.05 to 0.5 mol dm⁻³. Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility.

The solubility reported by Colton and Brooker (3), a value of 752 g dm⁻³ of saturated solution at 298 K, cannot be compared with the molal solubilities given by the other workers since densities were not measured. However this result is likely to be high since the solvent was not purified and may have contained significant quantities of water.

The solubilities determined by Paul et al. (2) and by Gopal and Husain (4) are in fair agreement over the temperature range studied. The greatest discrepancy occurs at 298 K, where solubilities of 656.3 g/kg of solvent and 566.2 g/kg of solvent respectively, are reported. Berardelli et al. (5) suggest the even more discrepant value of 850 g/kg of solvent. No simple explanation for this lack of agreement can be given. Analysis of the data suggested rejecting the result of Berardelli et al. (5). All the values from (2) and (4) were then subjected to a regression analysis where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was found (correlation coefficient of -0.986), and from the analysis the following tentative values are suggested.

Tentative value at 298 K

600 [±] 20 g/kg of solvent <u>Tentative value at 308 K</u>

788 [±] 10 g/kg of solvent <u>Tentative value at 318 K</u>

1038 [±] 15 g/kg of solvent.

References

1. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.

- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 3. Colton, E.; Brooker, R.E. J. Phys. Chem., 1958, 62, 1595-1596.
- 4. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963,40, 272-274.

5. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M., Ric. Sci., 1968, 38, 814-819.

22 Form	amide
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium iodide; NaI; [7681-82-5] (2) Formamide; CH ₃ NO; [75-12-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026.
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	65.6
35 45	80.0 162
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were prepared by adding excess of the powdered salt to formamide (15-20 g) in three Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for iodide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate	SOURCE AND PURITY OF MATERIALS: A.R. grade sodium iodide (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10 ⁻⁵ S m ⁻¹ was obtained. The solvent was stored in the dark. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.05 °C (author) REFERENCES: 1. Paul, R.C.; Singla, J.P.; Gill, D.S.; Narula, S.P. J. Chem. Soc., Dalton Trans., 1972, 522-524

		2:
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Sodium iodide; NaI; [7681-82-5]	Berardelli, M.L.; Pistoia, G.;	
(2) Formamide; CH ₃ NO; [75-12-7]	Polcaro, A.M.	
	Ric. Sci., 1968 38, 814-819	
VARIABLES:	PREPARED BY:	
One temperature: 25 °C	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of sodium i	odide in formamide at 25 ^O C.	
85.0 g/100 g solvent.		
}		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated sodium iodide solutions were prepared by dissolving excess of the halide in about 20-25 cm³ of solvent contained in a 50 cm³ Pyrex flask with a standard taper joint. The solution was then stored for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT 1) and,

presumably, standard aqueous silver nitrate. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of formamide to aqueous solution (1:5). The equivalent point remained the same (1).

SOURCE AND PURITY OF MATERIALS:

Sodium iodide, reagent grade, was dried under high vacuum before use. Commercial formamide (C. Erba, RP Product) was purified by sorting over CaO for 2 days before twice distilling under reduced pressure. The conductance of the formamide varied from 0.9 to 1.40×10^{-4} s m⁻¹.

ESTIMATED ERROR:

Solubility	
Temperature	

REFERENCES:

1. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172

± 1%

 ± 0.05 °C (author)

24 Form	amide
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium iodide; NaI; [7681-82-5]	Gopal, R.; Husain, M.M.
(2) Formamide; CH ₃ NO; [75-12-7]	J. Ind. Chem. Soc., <u>1963</u> , 40, 272-274
-	
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	L
t/ [°] C	g/100 g solvent
25	56.6
30	67.7
35 40	78.0 89.7
45	103
50	119
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of NaI were prepared at	A.R. grade sodium iodide.
10-15 ⁰ above the temperature at which the solubility was to be measured before	Commercial formamide was redistilled and stored out of contact with light
thermostatting at the required temperature. Aliquots $(3-5 \text{ cm}^3)$ were withdrawn with a	in amber-coloured bottles until used.
pro-pipette and weighed in a closed silica	
dish protected from moisture with P _. 0 _. Two more samples were taken for each ⁵	
temperature and the amount of solute present determined by one of the following methods	
determined by one of the following methods.(1) Evaporation Method - Weighed samples	-
were carefully heated to expel the solvent. The samples were then cooled and weighed in	ESTIMATED ERROR:
a dry atmosphere. The process was repeated	_
to obtain a constant weight. All three samples taken were treated in a similar	Solubility ± 1% Temperature ± 0.1 ^o C (compiler)
manner. (2) Precipitation Method - Weighed samples	
of saturated solutions were diluted with	REFERENCES :
water and the halide content was determined gravimetrically. Reliability of procedure	
was tested using known amounts of KC1 in	
formamide and estimating gravimetrically. When experimental and theoretical values	
agreed within ±0.5% the procedure was applied to the samples of unknown solubility.	
	L

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium iodide; NaI; [7681-82-5] (2) Formamide; CH ₃ NO; [75-12-7]	Colton, E.; Brooker, R.E. J. Phys. Chem., <u>1958</u> , 62, 1595-1596
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium iodide in formamide at 25 °C

752 g/1000 cm³ of saturated solution.

AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide in formamide were prepared in glass containers so that an excess of solid was always present. After tumbling for a minimum of 48 hours in a thermostatted water bath the solutions were filtered quickly and aliquots taken for analysis. Duplicate samples were run. The sodium was determined photometrically after decomposing the formamide. A Beckman	SOURCE AND PURITY OF MATERIALS: Reagent grade sodium iodide. Commercial formamide (Fisher Scientific Co) with a freezing point of 2.50 ^o C was used without further purification.	
DU spectrophotometer was used.	ESTIMATED ERROR: Solubility ⁺ 5% (compiler) Temperature ⁺ 1 ^o C (author)	
	REFERENCES :	

6 Formamide	
COMPONENTS:	EVALUATOR:
(1) Sodium thiocyanate; NaCNS;[540-72-7] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland December 1978
CRITICAL EVALUATION:	
the solute concentration. Measurements were made at 298, 308 and 31	solvent, the accuracy of the data is likely ne solvent used, rather than by the precision mermally unstable, photosensitive and fication procedure (2) is required to and the conductance to below $2 \times 10^{-5} \text{ sm}^{-1}$. mabled a conductance of $5 \times 10^{-5} \text{ sm}^{-1}$ to be of the analytical methods used to determine 18 K. As a check for internal consistency,
the logarithms of the solubilities were plotte absolute temperature. An excellent linear re of only one investigation are available, it we	elationship was obtained. While the results
with sufficient care for them to be taken as a	
<u>Recommended values</u> <u>298 K</u> 1435.0 [±] 10.0 g/kg of solvent <u>308 K</u> 891.0 [±] 7.0 g/kg of solvent <u>318 K</u> 575.1 [±] 4.0 g/kg of solvent <u>References</u> 1. Paul, R.C.; Singla, J.P.; Lamba, M.S.; C	Sill, D.S.; Narula, S.P. Indian J. Chem.,
<u>1973,</u> 11, 1024-1026, 2. Noltley, J.M.; Spiro, M. <i>J. Chem. Soc.(B)</i>	, <u>1966</u> , 362-366.

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Formamide	
ORIGINAL MEASUREMENTS:	
Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024.	
PREPARED BY:	
J.S. McKechnie	
L	
g/100 g solvent	
144 89.1	
57.5	
INFORMATION	
SOURCE AND PURITY OF MATERIALS;	
A.R. grade sodium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at >100 °C. Formamide (Reidel Pure) was finally distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5×10^{-5} S m ⁻¹ was obtained. The solvent was stored in the dark. ESTIMATED ERROR: Solubility ± 1%	
Temperature ± 0.05 °C (author)	
REFERENCES: 1. Paul, R.C.; Singla, J.P.; Gill, D.S.; Narula, S.P. <i>J. Chem. Soc.</i> , Dalton Trans., <u>1972</u> , 522-524.	

COMPONENTS :	EVALUATOR:
(1) Potassium chloride; KC1; [7447-40-7] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife. Scotland. December 1978

CRITICAL EVALUATION:

Five investigations of the solubility of KCl in formamide have been reported. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used so far in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹. It is likely that most of the solutions studied had water contents in the range 0.05 to 0.5 mol dm⁻³. Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility.

The solubility reported by Colton and Brooker (3) (a value of 80.8 g dm⁻³ of saturated solution at 298 K, which is equivalent to approximately 74.7 g/kg of solvent) is considerably higher than that found by other workers. This result is rejected since no purification of the solvent was attempted.

Solubilities at 298 K determined by Paul et al. (2) (62.9 g/kg of solvent), Pavlopoulos and Strehlow (4) (61.3 g/kg of solvent), Gopal and Husain (5) (63.0 g/kg of solvent) and Berardelli et al. (6) (61.8 g/kg of solvent) have a mean value of 62.3 g/kg of solvent with a standard deviation of 0.83 g/kg of solvent, or just over 1%. The results seem to fall into two groups with mean values of 61.5 ± 0.3 g/kg of solvent and 62.9 ± 0.1 g/kg of solvent, but it is not possible to select one of these as the better estimate. While the solvent used by Paul et al. (2) was particularly pure, no details are given of their analytical methods. The solvent used by Gopal and Husain (5) was unlikely to have been very dry. Both Pavlopoulos and Strehlow (4) and Berardelli et al. (6) took special precautions to prepare pure solvent and to work in a dry atmosphere. The latter groups used potentiometric titration to determine the chloride ion in solution.

Recommended value at 298 K

62.3 - 0.8 g/kg of solvent

Tentative values at other temperatures

Both Paul et al. (2) and Gopal and Husain (5) report solubilities at higher temperatures. Agreement between the two sets of data is very good at 298 K, but the values diverge as the temperature is raised until by 318 K the difference is over 3%. While the data of Paul et al. may be slightly more reliable at higher temperatures since their purer solvent would be less liable to decomposition, mean values are reported below as tentative best values. No significant difference is found between these and values determined using a linear regression analysis of the logarithm of all the solubilities as a function of the reciprocal of the absolute temperature. The only measurement reported at temperatures lower than 298 K is one at 291 K by Pavlopoulos and Strehlow (4). Their value of 60.3 g/ kg of solvent compares with 61.2 g/kg of solvent obtained by extrapolation of the best straight line through all the other points, and thus may be somewhat low. The error in the solubilities at temperatures other than 298 K is estimated to be -1.5%.

(continued)

CONMPONENTS:	EVALUATOR:	
(1) Potassium chloride; KC1; [7447-40-7] (2) Formamide; CH ₃ NO; [75-12.7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife, Scotland. December 1978.	
CRITICAL EVALUATION: (continued)		
<u>291 K</u>		
$61.2 \stackrel{+}{=} 1.0 \text{ g/kg of solvent}$		
<u>308 K</u>		
64.5 [±] 1.0 g/kg of solvent		
<u>318 K</u>		
66.7 [±] 1.0 g/kg of solvent		
References		
1. Notley, J.M.; Spiro, M.; J. Chem. Soc. (B), <u>1966</u> , 362-366.		
2. Paul, R.C.; Singla, J.P.; Lamba, M.S.;	Gill, D.S.; Narula, S.P. Indian J. Chem.,	
<u>1973</u> , <i>11</i> , 1024-1026.		
3. Colton, E.; Brooker, R.E. J. Phys. Chem.	, <u>1958</u> , <i>62</i> , 1595-1596.	
4. Pavlopoulos, T.; Strehlow, H. Z. Phys. (Them. (Weisbaden), <u>1954</u> , 2, 89–103.	
5. Gopal, R.; Husain, M.M. J. Indian Chem. S	50c., <u>1963</u> , <i>40</i> , 272-274.	

6. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819

30 For	mamide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KCl;[7447-40-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.;
(2) Formamide; CH ₃ NO; [75-12-7]	Gill, D.S.; Narula, S.P.
3	Indian J. Chem., <u>1973</u> , 11 , 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o c g/	100 g solvent
25	6.29
35	6.48
45	6.78
AUXILIA	RY INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium chloride	SOURCE AND PURITY OF MATERIALS:
were prepared by adding excess of the powder	A.R. grade potassium chloride (BDH) was red recrystallised, powdered and dried in
salt to formamide (15-20 g) in Pyrex tubes $(15 \times 2.5 \text{ cm})$ and heating to nearly 10 $^{\circ}$ C abo	vacuum at 110-125°C.
the temperature of the bath. The attainment	t reduced pressure after extensive prior
of the equilibrium was checked by intermittent analyses of the solutions. The solution	purification (1). Purity was checked by
were then filtered under nitrogen and	conductance of 5 x 10^{-5} S m ⁻¹ was obtained.
analysed for chloride. No details of anal- ytical methods were given. Transference of	The solvent was stored in the dark.
material, as far as possible, was carried o	ut
in a nitrogen filled dry box. Measurements were made in triplicate.	
	ESTIMATED ERROR:
	Solubility + 1%
	Temperature $+$ 0.05 °C (author)
	REFERENCES :
	1. Paul, R.C., Singla, J.P., Gill, D.S.,
	Narula, S.P. J. Chem. Soc., Dalton Trans
	<u>1972</u> , 522-524

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KCl; [7447-40-7] (2) Formamide; CH ₃ NO;[75-12-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	

Solubility of potassium chloride in formamide at 25 °C 6.18 g/100 g solvent

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated potassium chloride solutions were prepared by dissolving excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stored for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT 1) and, presumably, standard aqueous silver nitrate. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of formamide to aqueous solution (1:5). The equivalence point remained the Same (1).	SOURCE AND PURITY OF MATERIALS: Potassium chloride, reagent grade, was dried under high vacuum before use. Commercial formamide (C. Erba, RP product) was purified by storing over CaO for 2 days before twice distilling under reduced pressure, The conductance of the formamide varied from 0.9 to 1.40 x 10 ⁻⁴ S m ⁻¹ . ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.05 °C (author) REFERENCES: 1. Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1167-1172
1	1

32 Forma	amide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KC1; [7447-40-7] (2) Formamide; CH ₃ NO; [75-12-7]	Gopal, R.; Husain, M.M. J. Indian Chem. Soc., <u>1963</u> , 40, 272-274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ [°] C	g/100 g solvent
25	6.30
30	6.30
35	6.41
40	6.49
45	6.56
50	6.69
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS;
Saturated solutions of KCl were prepared at 10-15 °C above the temperature at which the solubility was to be measured before thermo- statting at the required temperature. Aliquots $(3-5 \text{ cm}^3)$ were withdrawm with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent.	A.R. grade potassium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner.	ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 [°] C (compiler)
(2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure was tested using known amounts of KCl in formamide and estimating gravimetrically. When experimental and theoretical values agreed within $\pm 0.5\%$ the procedure was applied to the samples of unknown solubility.	REFERENCES :

	Formamide	3:
COMPONENTS :	ORIGINAL MEASUREMENTS:	
(1) Potassium chloride; KC1; [7447-40-7	7] Colton, E.; Brooker, R.E.	
(2) Formamide; CH ₃ NO; [75-12-7]	J. Phys. Chem., <u>1958</u> , 62, 1595-1596	
VARIABLES:	PREPARED BY:	
One temperature: 25 ^o C	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of potassium chlorid	de in formamide at 25 ^O C	
81 g/1000 cm ³ of saturated solution.		

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium chloride in formamide were prepared in glass containers so that an excess of solid was always present. After tumbling for a minimum of 48 hours in a thermostatted water bath the solutions were filtered quickly and aliquots taken for analysis. Duplicate samples were run. The potassium was determined photo- metrically after decomposing the formamide. A Beckman DU spectrophotometer was used. Potassium was also determined gravimetrically with tetraphenylboron in the decomposed formamide solution.	SOURCE AND PURITY OF MATERIALS: Reagent grade potassium chloride. Commercial formamide (Fischer Scientific Co) with a freezing point of 2.50 °C was used without further purification. ESTIMATED ERROR: Solubility ± 5% (compiler) Temperature ± 1 °C (author) REFERENCES:

34 Forma	amide
COMPONENTS :	ORIGINAL MEASUREMENTS:
<pre>(1) Potassium chloride; KC1; [7447-40-7] (2) Formamide; CH₃NO; [75-12-7]</pre>	Pavlopoulos, T.; Strehlow, H. 2. Phys. Chem. (Weisbaden), <u>1954</u> , 2, 89-103
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	L
Solubility of potassium chl temperatures.	oride in formamide at two different
t/ ^o C	g/1000 cm^3 of saturated solution.
18	66.4
25	67.1
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Water free solutions (≤ 0.005%) were prepared in a special glass apparatus equipped with a magnetic stirrer which was placed in a thermostatted bath for several hours. The saturated solution was then forced out with dry air through a sinter, into a 50 cm ³ measuring flask and weighed. The air was dried over CaCl ₂ . A known volume was evaporated to dryness, redissolved in water and the halogen content determined potentiometrically. No details of the cell were given (1). The results obtained were	SOURCE AND PURITY OF MATERIALS: Reagent grade potassium chloride was dried at 240 °C for 4-5 hours in a furnace. Formamide (Merck) was dried over P ₂ O ₅ and twice distilled, using a long column of glass helices before use. The water content of the solvent was determined by Karl Fischer titration.
checked by density measurements.	ESTIMATED ERROR:
	Solubility [±] 1% Temperature [±] 0.1 ^o C (author)
	<pre>REFERENCES: 1. Pavlopoulos, T.; Strehlow, H. 2. Phys. Chem. (Leipzig), 1953,202, 474-479</pre>

COMPONENTS :	EVALUATOR:
(1) Potassium bromide; KBr; [7758-02-3] (2) Formamide; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

Three investigations of the solubility of KBr in formamide have been reported. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used so far in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹. It is likely that most of the solutions studied had water contents in the range 0.05 to 0.5 mol dm⁻³. Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility.

Solubilities at 298 K determined by Paul et al. (2) (216.1 g/kg of solvent), Gopal and Husain (3) (213.8 g/kg of solvent) and by Berardelli et al. (4) (216 g/kg of solvent) are in excellent agreement. They have a mean value of 215.3 g/kg of solvent with a standard deviation of 1.3 g/kg of solvent, or 0.6 %. There seems no good reason to favour one value over the others, and the mean value is therefore recommended.

Recommended value at 298 K

215.3 - 1.3 g/kg of solvent

Values at other temperatures

Both Paul et al. (2) and Gopal and Husain (3) report solubilities at higher temperatures. The temperature coefficient was found to be small. Agreement between the two sets of results is excellent. In a regression analysis of all the results where the logarithm of the solubility was assumed to be a linear function of the inverse absolute temperature, a correlation coefficient of -0.961 was found. The mean values of the two determinations at 308 K and 318 K are therefore recommended. Recommended value at 308 K

217.8 [±] 1.3 g/kg of solvent

Recommended value at 318 K

221.4 - 1.3 g/kg of solvent.

References

1. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.

- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026,
- 3. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963, 40, 272-274.
- 4. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819

36 Form	amide
COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Potassium bromide; KBr; [7758-02-3] (2) Formamide; CH_{.3}NO; [75-12-7]</pre>	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	l
t/ ^o C	g/100 g solvent
25	21.6.
35 45	21.8 ⁻ 22.2
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Satured solutions of potassium bromide were prepared by adding excess of the powdered salt to formamide $(15-20 \text{ g})$ in Pyrex tubes $(15\times2.5 \text{ cm})$ and heating to nearly 10 $^{\text{O}}\text{C}$ above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for bromide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	A.R. grade potassium bromide (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density _5
	J. Chem. Soc., Dalton Trans., <u>1972</u> ,522-524

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium bromide; KBr;[7758-02-3] (2) Formamide; CH ₃ NO;[75-12-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES:	PREPARED BY:
One Temperature: 25 ^O C	J.S. McKechnie
EXPERIMENTAL VALUES:	

Solubility of potassium bromide in formamide at 25 $^{\circ}\mathrm{C}.$

21.6 g/ 100 g solvent

AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated potassium bromide solutions were prepared by dissolving excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stored for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of	Potassium bromide, reagent grade, was dried under high vacuum before use. Commercial formamide (C. Erba, RP Product) was purified by storing over CaO for 2 days before twice distilling under reduced pressure. The conductance of the formamide varied from 0.9 to 1.40 x 10 ⁻⁴ S m ⁻¹ .	
dissolved halide determined by a Potentiometric method using a titrimeter	ESTIMATED ERROR:	
(type Radiometer TTT 1) and, presumably,	Solubility ± 1%	
standard aqueous silver nitrate. Equilibrium between solid and solution	Temperature ± 0.05 °C (author)	
phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of formamide to aqueous solution (1:5). The equivalence point remained the same (1).	REFERENCES: 1. Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , 37, 1167-1172	

8 Formamide	
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium bromide; KBr; [7758-02-3] (2) Formamide; CH ₃ NO; [75-12-7]	Gopal, R.; Husain, M.M. J. Ind. Chem. Soc., <u>1963</u> , 40, 272-274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	L
t/ ^o C	g/100 g solvent
25	21.4
30	21.7
35	21.7
40	21.9
45	22.1
50	22.2
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of KBr were prepared at $10-15$ °C above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pre-pipette and weighed in a closed silica dish protected from moisture with P ₀ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) Evaporation Method - Weighed samples	A.R. grade potassium bromide. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
 (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure was tested using known amounts of KC1 in formamide and estimating gravimetrical values agreed within ±0.5% the procedure was applied to the samples of unknown solubility. 	ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 ^O C (compiler) REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Potassium iodide; KI;[7681-11-0] (2) Formamide; CH ₃ NO;[75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

Five investigations of the solubility of KI in formamide have been reported. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used so far in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹. It is likely that most of the solutions studied had water contents in the range 0.05 to 0.5 mol dm⁻³. Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility.

Solubilities at 298 K determined by Paul et al. (2) (685.0 g/kg of solvent), Colton and Brooker (3)(692 g/kg of solvent - evaluator, using density data from (4)), Pavlopoulos and Strehlow (4) (669.4 g/kg of solvent), Gopal and Husain (5) (692.2 g/kg of solvent) and by Berardelli et al. (6) (678 g/kg of solvent) have a mean value of 683 g/kg of solvent) with a standard deviation of 10 g/kg of solvent. It is difficult to select the best determination. The two highest values occur with solvents where least care has been taken to eliminate water and these results are probabley greater than the true value. On the other hand, values given by Pavlopoulos and Strehlow (4) for KCl and KI in formamide have been lower than those reported by other workers using dry solvents. It has therefore been decided to recommend the mean value. Recommend value at 298 K

683 [±] 10 g/kg of solvent

Tentative values at other temperatures

Both Paul et al. (2) and Gopal and Husain (5) report solubilities at higher temperatures. The temperature coefficient for the solubility of KI in formamide is very small and the two sets of results are in good agreement. However, in consideration of the significantly lower solubilities reported at 298 K by other workers only tentative values are suggested. In order to give some weighting to the results of Berardelli et al. (6) and Pavlopoulos and Strehlow (4) a regression analysis of all the values was undertaken in which the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. From this analysis the following tentative values at 308 K and 318 K are given.

<u>308 K</u>

691 - g/kg of solvent

<u>318 K</u>

698 - 10 g/kg of solvent.

References

- 1. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 3. Colton, E.; Brooker, R.E. J. Phys. Chem., 1958, 62, 1595-1596. (continued)

Formamic	łe.

COMPONENTS:	EVALUATOR:	
(1) Potassium iodide; KI; [7681-11-0] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife, Scotland. December 1978.	
CRITICAL EVALUATION: (continued)	J	

- 4. Pavlopoulos, T.; Strehlow, H., Z. Phys. Chem. (Weisbaden), 1954, 2, 89-103.
- 5. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963, 40, 272-274.
- 6. Berardelli, M.L.; Pistoia, G.; Polcaro. A.M. Ric. Sci., 1968, 38, 814-819.

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For	mamide 4
OMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0] (2) Formamide; CH ₃ NO; [75-12-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026
ARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
XPERIMENTAL VALUES:	
t/ ^o C	g/100 g Solvent
25	68.5
35	69.2
45	69.8
AUXILIAR	Y INFORMATION
ETHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of potassium iodide Vere prepared by adding excess of the	A.R. grade potassium iodide (BDH) was
powdered salt to formamide (15-20 g) in	recrystallised, powdered and dried in vacuum at 110-125°C.
Pyrex tubes (15 x 2.5 cm) and heating to hearly 10 °C above the temperature of	Formamide (Reidel Pure) was distilled
the bath. The attainment of the	under reduced pressure after extensive prior purification (1). Purity was
quilibrium was checked by intermittent unalyses of the solutions. The solutions	checked by conductance and density
ere then filtered under nitrogen and	measurements. A conductance of $5 \times 10^{-5} \text{ S m}^{-1}$ was obtained. The solvent
nalysed for iodide. No details of nalytical methods were given.	was stored in the dark.
ransference of material, as far as possibl as carried out in a nitrogen filled dry	
ox. Measurements were made in triplicate	
	Solubility $\pm 1\%$
	Temperature [±] 0.05 ^o C (author)
	REFERENCES :
	 Paul, R.C.; Singla, J.P.; Gill, D.S.; Narula, S.P.
	J. Chem. Soc., Dalton Trans.,

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0] (2) Formamide; CH ₃ NO; [75-12-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> ,814-819
VARIABLES: One temperature: 25 ^O C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium iodide in formamide at 25 °C

67.8 g/100 g solvent.

AUXILIARY INFORMATION

METHOD / APPARATUS / PROCEDURE : Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated potassium iodide solutions were prepared by dissolving excess of the halide in about $20-25 \text{ cm}^3$ of solvent contained in a 50 cm³ Pyrex flask with a standard taper joint. The solution was then stored for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of the dissolved halide ddtermined by a potentiometric method using a titrimeter (type Radiometer TTT 1) and, presumably, standard aqueous silver nitrate.

Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of formamide to aqueous solution (1:5). The equivalence point remained the same (1).

SOURCE AND PURITY OF MATERIALS: Potassium iodide, reagent grade, was dried under high vacuum before use. Commercial formamide (C. Erba, RP Product) was purified by storing over CaO for 2 days before twice distilling under reduced pressure. The conductance of the formamide varied from 0.9 to 1.40 x 10^{-4} S m⁻¹

ESTIMATED ERROR:

Solubility

Temperature

REFERENCES :

Pistoia, G.; Pecci, G.; Scrosati, B.
 Ric. Sci., <u>1967</u>, *37*, 1167-1172.

± 1%

 \pm 0.05 °C (author)

Fn	rm	am	ide
10		am	luç

Formamide	
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0]	Gopal, R.; Husain, M.M.
(2) Formamide; CH ₃ NO; [75-12-7]	J. Ind. Chem. Soc., <u>1963</u> , 40, 272-274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/°C	g/100 g solvent
25	69.2
30	69.3
35	69.4
40	69.6
45	69.7
50	69,8
	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
METHOD/APPARATUS/PROCEDURE: Saturated solutions of KI were prepared at 10-15 ° above the temperature at which the	SOURCE AND PURITY OF MATERIALS: A.R. grade potassium iodide.
METHOD/APPARATUS/PROCEDURE: Saturated solutions of KI were prepared at 10-15 o above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent.	SOURCE AND PURITY OF MATERIALS: A.R. grade potassium iodide. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
METHOD/APPARATUS/PROCEDURE: Saturated solutions of KI were prepared at 10-15 o above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in	SOURCE AND PURITY OF MATERIALS: A.R. grade potassium iodide. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
METHOD/APPARATUS/PROCEDURE: Saturated solutions of KI were prepared at 10-15 ° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent.	SOURCE AND PURITY OF MATERIALS: A.R. grade potassium iodide. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.

44 Forma	Formamide		
COMPONENTS :	ORIGINAL MEASUREMENTS:		
(1) Potassium iodide; KI; [7681-11-0]	Colton, E.; Brooker, R.E.		
(2) Formamide; CH ₃ NO; [75-12-7]	J. Phys. Chem., 1958. 62, 1595-1596		
VARIABLES:	PREPARED BY:		
One temperature: 25 °C	J.S. McKechnie		
EXPERIMENTAL VALUES:			
Solubility of potassium iodide in f			
628 g/1000 cm ³ of saturated	l solution.		
AUXILIARY	INFORMATION		
METHOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:		
Saturated solutions of potassium iodide in	Reagent grade potassium iodide.		
formamide were prepared in glass containers so that an excess of solid was always	Commercial formamide (Fisher Scientific Co) with a freezing point of 2.50 °C was used		
present. After tumbling for a minimum of 48 hours in a thermostatted water bath the	without further purification.		
solutions were filtered quickly and			
aliquots taken for analysis. Duplicate samples were run.			
The potassium was determined photo-			
metrically after decomposing the formamide. A Beckman DU spectrophotometer was used.			
Potassium was also determined gravi- metrically with tetraphenylboron, in the	ESTIMATED ERROR:		
decomposed formamide solution.	Solubility ⁺ 5% (compiler) Temperature ⁺ 1 ^o C (author)		
	REFERENCES :		
	1		

COMPONENTS:	ORIGINAL MEASUREMENTS:
 Potassium iodide; KI; [7681-11-0] Formamide; CH₃NO; [75-12-7] 	Pavlopoulos, T.; Strehlow, H. Z. Phys. Chem. (Wiesbaden), <u>1954</u> , 2, 89-103
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium iodide in formamide at 25 $^{\rm O}{\rm C}$

616 g/1000 cm³ of saturated solution.

	INFORMATION
METHOD/APPARATUS/PROCEDURE: Water free solutions (≤ 0.005%) were prepared in a special glass apparatus equipped with a magnetic stirrer which was placed in a thermostatted bath for several hours. The saturated solution was then forced out with dry air through a sinter, into a 50 cm ³ measuring flask and weighed. The air was dried over CaCl ₂ . A known volume was evaporated to dryness, redissolved in water and the halogen content determined potentiometrically. No details of the cell were given (1). The results obtained were checked by density measurements.	SOURCE AND PURITY OF MATERIALS: Reagent grade potassium iodide was dried at 240 °C for 4-5 hours in a furnace. Formamide (Merck) was dried over P ₂ O ₅ and twice distilled, using a long column of glass helices, before use. The water content of the solvent was determined by Karl Fischer titration. ESTIMATED ERROR: Solubility ⁺ / ₊ 1% Temperature ⁺ / ₋ 0.1 °C (author) REFERENCES: 1. Pavlopoulos, T.; Strehlow, H.; 2. Phys. Chem. (Leipzig), <u>1953</u> ,202, 474-479.

COMPONENTS:	EVALUA
 (1) Potassium cyanide; KCN; [151-50-8] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. V Depart Univer St. An Scotla

TOR:

Vincent, tment of Chemistry. rsity of St.Andrews. ndrews, Fife, and. December 1978

CRITICAL EVALUATION:

One investigation of the solubility of KCN in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2 x 10⁻⁵ S m⁻¹. In this investigation of KCN, no attempt was made to dry or purify the solvent. Since preferential solvation of the ions by water is probable, the value given probably represents the higher limit of solubility.

Tentative value at 298 K

146 \div 8 g dm⁻³ of saturated solution

References

1. Colton, E.; Brooker, R.E. J. Phys. Chem., 1958, 62, 1595-1596. 2. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.

COMPONENTS:	ORIGINAL MEASUREMENTS:
 Potassium cyanide; KCN; [151-50-8] Formamide; CH₃NO; [75-12-7] 	Colton, E.; Brooker, R.E. J. Phys. Chem., <u>1958</u> , 62, 1595-1596
VARIABLES: One temperature: 25 ^O C	PREPARED BY: J.S. McKechnie

Solubility of potassium cyanide in formamide at 25 $^{\rm O}{\rm C}.$

146 g/1000 cm^3 of saturated solution.

	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium cyanide in formamide were prepared in glass containers so that an excess of solid was always present After tumbling for a minimum of 48 hours in a thermostatted water bath the solutions were filtered quickly and aliquots taken for analysis. Duplicate samples were run.	SOURCE AND PURITY OF MATERIALS: Reagent grade potassium cyanide, Commercial formamide (Fisher Scientific Co) with a freezing point of 2.50 ^o C was used without further purification.
The potassium was determined photo- metrically after decomposing the formamide. A Beckman DU spectrophotometer was used. Potassium was also determined gravimetrically with tetraphenylboron in the decomposed formamide solution.	ESTIMATED ERROR: Solubility <u>+</u> 5% (compiler) Temperature <u>+</u> 1 ^O C (author) REFERENCES:

40 10111	
COMPONENTS :	EVALUATOR:
 (1) Potassium thiocyanate; KCNS; [330-20-0] (2) Formamide; [75-12-7] 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

CRITICAL EVALUATION:

Three investigations of the solubility of KCNS in formamide have been reported. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used so far in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹. It is likely that most of the solutions studied had water contents in the range 0.05 to 0.5 mol dm⁻³. Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility.

The solubility reported by Colton and Brooker (3), a value of 659 g dm⁻³ of saturated solution at 298 K, cannot be compared with the molal solubilities given by the other workers since densities were not measured. The solubilities at 298 K determined by Paul et al. (2) (1435.0 g/kg of solvent) and by Becker (4) (886 g/kg of solvent) are so far apart that it is difficult to recommend even a tentative value for this solute. Measurements at 308 and 318 K by Paul et al. (2) indicate that the temperature coefficient of solubility is very small. However the rate of dissolution may be significantly affected by temperature. Paul et al. heat their samples initially at 10 °C above the temperature of the bath before cooling to the equilibration temperature. Becker (4) maintained his solutions at 25 °C throughout the 48 hour equilibration period and it is therefore possible that his solutions did not saturate because of slow dissolution kinetics. Tentative values 298 to 318 K

1420 - 20 g/kg of solvent.

References

- 2. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973; 11, 1024-1026
- 3. Colton, E.; Brooker, R.E. J. Phys. Chem., 1958, 62, 1595-1596.
- 4. Becker, B. J. Chem. Eng. Data, 1970, 15, 31-32.

^{1.} Notley, J.M.; Spiro, M.; J. Chem. Soc. (B), 1966, 362-366.

Form	amide 4
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Potassium thiocyanate; KCNS;[333-20-0] Formamide; CH₃NO;[75-12-7] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian, J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
XPERIMENTAL VALUES:	
t/ ^o C 25 35 45	g/100 g solvent 143.5 141.9 141.0
AUXILIARY	INFORMATION
METHOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of potassium thiocyanate were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for chloride. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	A.R. grade potassium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10 ⁻⁵ S

50 Forma	ımide
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium thiocyanate; KCNS;[333-20-0]	Becker, B. J. Chem. Eng. Data, 1970, 15(1),
(2) Formamide; CH ₃ NO; [75-12-7]	31-32
5	
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of potassium thioc	yanate in formamide at 25 °C.
89 g/100 g solvent	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS;
Saturated solutions were prepared by shaking	
excess potassium thiocyanate with the solvent for at least 48 hours in a thermostatted	was used without further purification.
bath. The formamide was exposed to air only briefly during addition. The mixture	Its melting point of 2.5 °C - 0.1 °C was in good agreement with the literature
was filtered rapidly in air at 25 °C and a weighed aliquot of the filtrate was taken	value of 2.55 °C.
for analysis. Two or three separate solubility determinations were made for each	
compound. Before analysis, formamide was	
removed by evaporating an aqueous solution of the sample to dryness. Potassium was	
determined by the Perrin method (1), which involved weighing the potassium precipitated	ESTIMATED ERROR:
as K2 ^{PtC1} 6.	Solubility + 5% (compiler)
	Temperature ⁺ 1 ^o C (author)
	REFERENCES:
	(1) Perrin, C.H. Anal. Chem., <u>1949</u> , 21,984

Formamide 5	
COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Potassium thiocyanate; KCNS; [333-20-0] (2) Formamide; CH₃NO; [75-12-7]</pre>	Colton, E.; Brooker, R.E.; J. Phys. Chem., <u>1958</u> , 62, 1595-1596
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J. S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of potassium thin	cyanate in formamide at 25 ⁰ C
659 g/1000 cm ³ of sat	urated solution.
	INFORMATION
METHOD /APPARATUS / PROCEDURE :	
Saturated solutions of potassium thiogyapate	SOURCE AND PURITY OF MATERIALS; Reagent grade potassium thiocyanate,
so that an excess of solid was always present	
	with a freezing point of 2.50 °C was used
were filtered quickly and aliquots taken for	without further purification.
analysis. Duplicate samples were run.	
The potassium was determined photo-	
A Beckman DU spectrophotomator use used	
	ESTIMATED ERROR:
with tetraphenylboron in the decomposed formamide solution.	Solubility - 5% (compiler)
	Temperature $\frac{+}{2}$ 1 °C (author)
	REFERENCES:

52 For	Formamide	
COMPONENTS:	EVALUATOR:	
(1) Rubidium bromide; RbBr; [7789-39-1] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978	

One investigation of the solubility of RbBr in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. In the present investigation stringent precautions were taken to purify and dry the solvent. However because of large discrepancies between solubilities of other salts in formamide reported in this work and those reported elsewhere (3), the value given for the solubility of RbBr at 298 K is recommended as a tentative value only.

Tentative value at 298 K

 274.5 ± 3.0 g dm⁻³ of saturated solution.

References

- 1. Pavlopoulos, T.; Strehlow, H. Z. Phys. Chem. (Wiesbaden), 1954, 2,89-103.
- 2. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.
- 3. Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u>, 89, 3703-3712.

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AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Water free solutions (\$0.005%) were prepared in a special glass apparatus equipped with a magnetic stirrer which was placed in a thermostatted bath for several hours. The saturated solution was then forced out with dry air through a sinter, into a 50 cm ³ measuring flask and weighed. The air was dried over CaCl ₂ . A known volume was evaporated to dryness, redissolved in water and the halogen content determined potentiometrically. No details of the cell were given (1). The results obtained	SOURCE AND PURITY OF MATERIALS: Reagent grade rubidium bromide was dried at 240 $^{\circ}$ C for 4-5 hours in a furnace. Formamide (Merck) was dried over P_2O_5 and twice distilled, using a long column of glass helices, before use. The water content of the solvent was determined by Karl Fischer titration.
were checked by density measurements.	ESTIMATED ERROR: Solubility ⁺ 1% Temperature ⁺ 0.1 [°] C (author)
	REFERENCES: 1. Pavlopoulos, T.; Strehlow, H. 2. Phys. Chem. (Leipzig), <u>1953</u> , 202, 474-479.

54 Forr	mamide	
COMPONENTS:	EVALUATOR:	
(1) Caesium chloride; CsC1; [7647-17-8] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978	

CRITICAL EVALUATION:

One investigation of the solubility of CsCl in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2 x 10^{-5} S m⁻¹. In the present investigation stringent precautions were taken However because of large discrepancies between to purify and dry the solvent. solubilities of other salts in formamide reported in this work and those reported elsewhere (3), the value given for the solubility of CsCl at 298 K is recommended as a tentative value only.

Tentative value at 298 K

91.5 $\stackrel{+}{=}$ 2.0 g dm⁻³ of saturated solution.

References

- 1. Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., 1967, 89, 3703-3712.
- 2. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.
- 3. Pavlopoulos, T.; Strehlow, H. Z. Phys. Chem. (Wiesbaden), 1954, 2, 89-103.

COMPONENTS: (1) Caesium chloride; CsC1; [7647-17-8] (2) Formamide; CH ₃ NO; [75-12-7]	ORIGINAL MEASUREMENTS: Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u> , 89, 3703-3712
VARIABLES:	PREPARED BY:
One temperature: 25°C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium chloride in formamide at 25 °C

91.5 g/1000 cm^3 of saturated solution

Value calculated from solubility product data given as:

 $\log (K_o/mol dm^{-3}) = -0.53$

AUXILIARY	INFORMATION

METHOD / APPARATUS / PROCEDURE : Saturated solutions of caesium chloride were prepared by shaking the solid with solvent in a stoppered flask at 35 °C for 24 hours. The flask was then shaken for a further 24 hours at 25 °C. The liquid phase was analysed for halide potentiometrically using silver concentration cells with a tetraethyl ammonium picrate salt bridge. The cells were wrapped in aluminium foil and immersed in a 25 °C thermostat and stirred magnet-ically, as standard 0.01 mol dm⁻³ silver nitrate was added. The emf was measured on a Radiometer pH meter (type PHM 22r). It was established early in the work that effectively the same results were obtained no matter whether measurements were made in the dark on freshly prepared solutions, on solutions exposed to the atmosphere for 30 minutes, or on solutions kept under dry nitrogen during preparation and titration. The usual procedure was to titrate 0.01 mol dm-3AgNO solution into a half-cell containing the silver wire immersed in 20 cm³ of 0.01 mol dm-3caesium chloride in formamide. The reference half-cell was a silver wire in $0.01 \text{ mol } dm^{-3} \text{ AgNO}_3$ in the same solvent.

SOURCE AND PURITY OF MATERIALS: A.R. grade caesium chloride was used without further purification. Formamide was dried with Type 4A molecular sieves and fractionated twice under a reduced pressure of dry nitrogen. It was stored over molecular sieves in dark bottles and was used within 7 days of purification. The silver nitrate solution turned brown, slowly, but the concentration of silver ion when estimated by titration with potassium bromide, was as required by the stoichiometric amount of silver nitrate.

ESTIMATED ERROR:

Solubility ⁺ 2% (compiler) Temperature ⁺ 0.1 ^oC (compiler)

REFERENCES :

56 F	ormamide
COMPONENTS :	EVALUATOR:
(1) Caesium bromide; CsBr; [7787-69-1] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of CsBr in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2 x 10^{-5} s m⁻¹ In the present investigation stringent precautions were taken to purify and dry the solvent However because of large discrepancies between solubilities of other salts in formamide reported in this work and those reported elsewhere (3), the value given for the solubility of CsBr at 298 K is recommended as a tentative value only.

 $\frac{\text{Tentative value at 298 K}}{152.4 + 3.0 \text{ g dm}^{-3}} \text{ of saturated solution.}$

References

1. Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., 1967, 89, 3703-3712.

- 2. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.
- 3. Pavlopoulos, T.; Strehlow, H. Z. Phys. Chem. (Wiesbaden), 1954, 2, 89-103.

	rmamide 5
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Caesium bromide; CsBr; [7787-69-1] (2) Formamide; CH ₃ NO; [75-12-7]	Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J.
(3) = 0 = 0 = 0 = 0 = 0 = 0 = 0 = 0 = 0 =	J. Am Chem. Soc., <u>1967</u> , 89, 3703-3712
VARIABLES:	
	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of caesium bromide in form	amide at 25 °C
152.4 g/1000 cm^3 of saturated solu	tion.
Value calculated from solubility prod	
$\log (\kappa_{s} o/mol dm^{-3}) = -0.29$	
AT1771	
METHOD /APPARATUS / PROCEDURE :	RY INFORMATION
Saturated solutions of caesium bromide wer	SOURCE AND PURITY OF MATERIALS: A.R.grade caesium bromide was used without
prepared by shaking the solid with solvent	further purification.
in a stoppered flask at 35 °C for 24 hours The flask was then shaken for a further 24	Formamide was dried with Type 4A molecular sieves and fractionated twice under a reduce
nours at 25 °C. The liquid phase was	pressure of dry nitrogen. It was stored ove
analysed for halide potentiometrically usi silver concentration cells with a tetraeth	ng molecular sieves in dark bottles and was use
ammonium picrate salt bridge. The cells w	ere nitrate solution turned brown slowly but the
wrapped in aluminium foil and immersed in	a concentration of silver ion when estimated h
25 °C thermostat and stirred magnetically, as standard 0.01 mol dm silver nitrate w	the titration with potassium bromide, was as as required by the stoichiometric amount of
added. The emf was measured on a Radiome	ter silver nitrate.
PH meter (type PHM 22r). It was establish early in the work that effectively the sam	ed ESTIMATED ERROR:
^{results} were obtained no matter whether	$\int c_0 1 u h i 1 i h m + 27 (compailer)$
measurements were made in the dark on fres prepared solutions, on solutions exposed to	hlvl
the atmosphere for 30 minutes, or on	DEPEDENCES .

REFERENCES:

the atmosphere for 30 minutes, or on solutions kept under dry nitrogen during preparation and titration. The usual procedure was to titrate 0.01 mol dm⁻³ AgNO₃ solution into a half-cell containing the silver wire immersed in 20 cm⁻³ of 0.01 mol dm⁻³caesium bromide in formamide. The reference half-cell was a silver wire in 0.01 mol dm⁻³ AgNO₃ in the same solvent.

58	Formamide	
COMPONENTS :	EVALUATOR:	
(1) Caesium iodide; CsI; [7789-17-5] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978	

CRITICAL EVALUATION:

Investigations of the solubility of CsI in formamide have been reported by Paylopoulos and Strehlow (1) and by Alexander et al. (2). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (3) is required to lower the water content to below 0.01 mol dm^{-3} and the conductance to below $2 \times 10^{-5} \text{ sm}^{-1}$.

In both investigations of CsI solubility, care was taken to purify and dry the solvent. In each case the halide ions were determined potentiometrically. Values of solubility at 298 K were reported as 431.3 g dm⁻³ of saturated solution (1) and 199.4 g dm⁻³ of saturated solution (2). The discrepancy between these values is so large that not even a tentative solubility is recommended.

References

- 1. Pavlopoulos, T.; Strehlow, H. Z. Phys. Chem. (Wiesbaden), 1954, 2, 89-103.
- Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u>, 89, 3703-3712.
- 3. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.

COMPONENTS:	
(1) Caesium iodide; CsI; [7789-17-5] (2) Formamide; CH ₃ NO; [75-12-7]	ORIGINAL MEASUREMENTS: Alexander R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u> , 89, 3703-3712
VARIABLES: One temperature: 25 ^O C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium iodide in formamide at 25 °C

199.4 g/1000 cm³ of saturated solution.

Value calculated from solubility product data given as:

 $\log (K_o/mol dm^{-3}) = -0.23$

AUXILIARY INFORMATION

METHOD / APPARATUS / PROCEDURE : Saturated solutions of caesium iodide were prepared by shaking the solid with solvent in a stoppered flask at 35 °C for 24 hours. The flask was then shaken for a further 24 hours at 25 $^{\circ}$ C. The liquid phase was analysed for halide potentiometrically using silver concentration cells with a tetraethylammonium picrate salt bridge. The cells were wrapped in aluminium foil and immersed in a 25 °C thermostat and stirred magnetically, as standard 0.01 mol dm ′silver nitrate was added. The emf was measured on a Radiometer pH meter (type PHM 22r). It was established early in the work that effectively the same results were obtained no matter whether measurements were made in the dark on freshly prepared solutions, on solutions exposed to the atmosphere for 30 minutes, or on solutions kept under dry nitrogen during preparation and titration. The usual procedure was to titrate 0.01 mol dm-3AgNO3 solution into a half-cell containing the silver wire immersed in 20 cm³ of 0.01 mol dm⁻³caesium iodide in formamide. The reference half-cell was a silver wire in 0.01 mol dm^{-3} AgNO₃ in the same solvent.

SOURCE AND PURITY OF MATERIALS: A.R. grade caesium iodide was used without further purification. Formamide was dried with Type 4A molecular sieves and fractionated twice under a reduced pressure of dry nitrogen. It was stored over molecular sieves in dark bottles and was used within 7 days of purification. The silver nitrate solution turned brown, slowly, but the concentration of silver ion when estimated by titration with potassium bromide, was as required by the stoichiometric amount of silver nitrate. ESTIMATED ERROR:

Solubility ⁺ 2% (compiler) Temperature ⁺ 1 ^oC (compiler)

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
	Pavlopoulos, T.; Strehlow, H. Z. Phys. Chem. (Wiesbaden), <u>1954</u> , 2, 89-103
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium iodide in formamide at 25 °C

431 g/1000 cm³ of saturated solution.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Water free solutions ($\leq 0.005\%$) were prepared in a special glass apparatus equipped with a magnetic stirrer which was placed in a thermostatted bath for several hours. The saturated solution was then forced out, with dry air through a sinter, into a 50 cm² measuring flask and weighed. The air was dried over CaCl₂. A known volume was evaporated to dfyness, redissolved in water and the halogen content determined potentiometrically. No details of the cell were given (1). The results obtained were checked by density measurements.

SOURCE AND PURITY OF MATERIALS:

Reagent grade caesium iodide was dried at 240 °C for 4-5 hours in a furnace. Formamide (Merck) was dried over P_2O_5 and twice distilled, using a long column of glass helices, before use. The water content of the solvent was determined by Karl Fischer titration.

ESTIMATED ERROR:

Solubility $\stackrel{+}{=}$ 1% Temperature $\stackrel{+}{=}$ 0.1 ^oC (author)

REFERENCES:

 Pavlopoulos, T.; Strehlow, H.
 Z. Phys. Chem. (Leipzig), <u>1953</u>, 202, 474-479.

COMPONENTS:	EVALUATOR:
(1) Ammonium chloride; NH ₄ Cl; [12125-02-9] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

CRITICAL EVALUATION:

Two sets of values have been reported for the solubility of NH_4C1 in formamide. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹.

Measurements on the solubility of NH_4CI were made at three temperatures by Paul et al. (2) and at six temperatures by Gopal and Husain (3). Evaluations at common temperatures agree to better than 0.6%. In a regression analysis of all nine results where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature, a correlation coefficient of -0.994 was found. On the basis of this analysis the following solubility values are recommended.

Recommended values

298 K 109.5 ⁺ 0.7 g/kg of solvent
308 K 116.7 ⁺ 0.7 g/kg of solvent
318 K 123.5 ⁺ 0.7 g/kg of solvent
References
1. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), <u>1966</u>, 362-366.
2. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
3. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., <u>1963</u>, 40, 272-274.

62 Form	amide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Ammonium chloride; NH ₄ Cl; [12125-02-9] (2) Formamide; CH ₃ NO; [75-12-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> ,11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/°C 25	g/100 g solvent 10.9
35	11.6
45	12.3
	:
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of ammonium chloride were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for chloride. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	 A.R. grade ammonium chloride (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10⁻⁵ S m⁻¹ was obtained. The solvent was stored in the dark.
	Solubility ± 1% Temperature ± 0.05 ^O C (author)
	<pre>REFERENCES: 1. Paul, R.C.; Singla, J.P.; Gill, D.S.; Narula, S.P. J. Chem. Soc., Dalton Trans., 1972, 522-524.</pre>

Formamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Ammonium chloride; NH₄Cl; [12125-02-9] (2) Formamide; CH₃NO; [75-12-7]</pre>	Gopal, R.; Husain, M.M. J. Ind. Chem. Soc., <u>1963</u> , 40,272-274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	<u>I</u>
t/ ^o c	g/100 g solvent
25	11.0
30	11.0
35	11.7
40	12.1
45	12.4
50	12.8
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of NH_Cl were prepared at 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm') were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P_05. Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) Evaporation Method - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure was tested using known amounts of KCl in formamide and estimating gravimetrically. When experimental and theoretical values agreed within ±0.5% the procedure was applied to the samples of means of the samples of	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 °C (compiler) REFERENCES:
to the samples of unknown solubility.	

COMPONENTS: (1) Ammonium bromide; NH₄Br; [12124-97-9] (2) Formamide; CH₃NO; [75-12-7] EVALUATOR: C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Two sets of values have been reported for the solubility of NH₄Br in formamide. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹.

Measurements on the solubility of NH_4Br were made at three temperatures by Paul et al. (2) and at six temperatures by Gopal and Husain (3). Evaluations at common temperatures agree to better than 1.3% In a regression analysis of all nine results where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature, a correlation coefficient of -0.923 was found. On the basis of this analysis the following solubility values are recommended.

Recommended values

298 K

361.0 [±] 5.0 g/kg of solvent <u>308 K</u> 384.0 [±] 5.0 g/kg of solvent <u>318 K</u> <u>394.5 [±] 5.0 g/kg of solvent</u>

References

- 1. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.
- 2. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 3. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963, 40, 272-274.

Forma	amide 65
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ammonium bromide; NH ₄ Br;[12124-97-9] (2) Formamide; CH ₃ NO; [75-12-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES: Temperature	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ⁰ C 25 35 45	g/100 g solvent 37.2 38.9 39.5
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of ammonium bromide were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for bromide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	A.R. grade ammonium bromide (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5×10^{-5} S m ⁻¹ was obtained. The solvent was stored in the dark. ESTIMATED ERROR: Solubility \pm 1% Temperature \pm 0.05 °C (author) REFERENCES: 1. Paul, R.C.; Singla, J.P.; Gill, D.S.;
	Narula, S.P. J. Chem. Soc., Dalton Trans., <u>1972</u> , 522-524

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Formamide		
COMPONENTS: ORIGINAL MEASUREMENTS:		
(1) Ammonium bromide; NH ₄ Br; [12124-97-9] (2) Formamide; CH ₃ NO; [75-12-7]	Gopal, R.; Husain, M.M. J. Ind. Chem. Soc., <u>1963</u> , 40, 272-274	
VARIABLES:	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:		
t/ ^o C	g/100 g solvent	
25	36.1.	
30	37.0	
35	37.9	
40	38.7	
45	39.4	
50	40.4	
AUXILIARY	INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Saturated solutions of NH, Br were prepared at 10-15° above the temperature at which the	A.R. grade ammonium bromide	
solubility was to be measured before	Commercial formamide was redistilled and	
thermostatting at the required temperature. Aliquots (3-4 cm ³) were withdrawn with a	stored out of contact with light in amber-coloured bottles until used.	
pro-pipette and weighed in a closed silica	amber-coroured bottles until used.	
dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each		
temperature and the amount of solute present		
determined by one of the following methods. (1) Evaporation Method - Weighed samples		
were carefully heated to expel the solvent.		
The samples were then cooled and weighed in a dry atmosphere. The process was repeated	ESTIMATED ERROR: Solubility 1%	
to obtain a constant weight. All three samples taken were treated in a similar		
manner.	Temperature 0.1 °C (compiler)	
(2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water	REFERENCES :	
and the halide content was determined		
gravimetrically. Reliability of procedure was tested using known amounts of KCl in		
formamide and estimating gravimetrically.		
When experimental and theoretical values agreed within ±0.5% the procedure was applied		
to the samples of unknown solubility.		

COMPONENTS:	EVALUATOR:	
(1) Ammonium iodide; NH ₄ I; [12027-06-4] (2) Formamide; CH ₃ NO; {75-12-7]	C. A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978	
CRITICAL EVALUATION:		
Two sets of values have been reported for the	e solubility of NH_4^{I} in formamide. As in	
other measurements of salt solubility in this	s solvent, the accuracy of the data is	

likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. The most stringent procedure used in solubility studies has been that of Paul et al. (2) who obtained a conductance of 5×10^{-5} S m⁻¹.

Measurements on the solubility of NH_4I were made at three temperatures by Paul et al. (2) and at six temperatures by Gopal and Husain (3). Evaluations at common temperatures agree to better than 0.2% In a regression analysis of all nine results where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature, a correlation coefficient of -0.961 was found. On the basis of this analysis the following solubility values are recommended.

Recommended values

298 K 1043.5 [±] 2.0 g/kg of solvent 308 K 1050.0 [±] 2.0 g/kg of solvent <u>318 K</u> 1060.5 [±] 2.0 g/kg of solvent <u>References</u> 1. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), <u>1966</u>, 362-366. 2. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.

3. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963, 40, 272-274

Formamide		
COMPONENTS :	ORIGINAL MEASUREMENTS:	
(1) Ammonium iodide; NH ₄ I;[12027-06-4] (2) Formamide; CH ₃ NO; [75-12-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026	
VARIABLES:	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:		
t/ [°] C	g/100 g solvent	
25	104	
35	105	
45	106	
	INFORMATION	
METHOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:	
Saturated solutions of ammonium iodide were prepared by adding excess of the powdered salt to formamide $(15-20 \text{ g})$ in Pyrex tubes $(15 \times 2.5 \text{ cm})$ and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for iodide. No details of analytical methods were given. Transference of material, as far as	A.R. grade ammonium iodide (BDH) was recrystallised, powdered and dried in vacuum at 110-125 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of $5 \times 10^{-5} \text{ S m}^{-1}$ was obtained. The solvent was stored in the dark.	
possible, was carried out in a nitrogen filled dry box. Measurements were	ESTIMATED ERROR:	
made in triplicate.	Solubility ± 1% Temperature ± 0.05 ^o C (author)	
	REFERENCES: 1. Paul, R.C.; Singla, J.P.; Gill, D.S.; Narula, S.P. J. Chem. Soc., Dalton Trans., 1972, 522-524	

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Formamide		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
	Caral D. A. Hussin M.M.	
(1) Ammonium iodide; NH_4I ; [12027-06-4] (2) Formeridae (UNO) [75-12-7]	Gopal, R.; Husain, M.M.	
(2) Formamide; CH ₃ NO; [75-12-7]	J. Ind. Chem. Soc., <u>1963</u> , 40, 272-274	
VARIABLES:	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:	l	
t/ ^o C	g/100 g solvent	
25	104	
30	105	
35	105	
40	105	
45	106	
50	106	
METHOD/APPARATUS/PROCEDURE:	INFORMATION SOURCE AND PURITY OF MATERIALS:	
Saturated solutions of NH_4I were prepared as $10-15^\circ$ above the temperature at which the		
solubility was to be measured before	A.R. grade ammonium iodide	
thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.	
were carefully heated to expel the solvent. The samples were then cooled and weighed		
in a dry atmosphere. The process was repeated to obtain a constant weight.	ESTIMATED ERROR:	
All three samples taken were treated in a similar manner.	Solubility $\pm 1\%$ Temperature ± 0.1 °C (compiler)	
(2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with	REFERENCES :	
water and the halide content was determined gravimetrically. Reliability of procedure was tested using known amounts of KC1 in formamide and estimating gravimetrically. When experimental and theoretical values agreed within ±0.5% the procedure was		
applied to the samples of unknown solubility	7	

COMPONENTS :	EVALUATOR:
(1) Ammonium thiocyanate; NH ₄ CNS;[1762-95-4] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland December 1978

CRITICAL EVALUATION:

One investigation of the solubility of NH_4CNS in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. In this study, careful solvent purification enabled a conductance of 5×10^{-5} S m⁻¹ to be obtained. Unfortunately no detail is given of the analytical methods used to determine the solute concentration.

Measurements were made at 298, 308, and 318 K. As a check for internal consistency, the logarithms of the solubilities were plotted as a function of the reciprocal of the absolute temperature. An excellent linear relationship was obtained. While the results of only one investigation are available, it would appear that they have been determined with sufficient care for them to be taken as recommended values.

Recommended values

298 K 1015.5 [±] 6.0 g/kg of solvent 308 K 1102.0 [±] 6.0 g/kg of solvent 318 K 1163.0 [±] 6.0 g/kg of solvent <u>References</u> 1. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026

2. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.

COMPONENTS:	ORIGINAL MEASUREMENTS:
OMPONENTS:	ORIGINAL MEASUREMENTS:
 Ammonium thiocyanate; NH₄CNS;[1762-95-4] 	
(2) Formamide; CH ₃ NO; [75-12-7]	Gill, D.S.; Narual, S.P.
	Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
XPERIMENTAL VALUES:	
t/ ^o c	g/100 g solvent
25	102
35	110
45	116
AUXILIARY	INFORMATION
	INFORMATION SOURCE AND PURITY OF MATERIALS:
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH)
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions Were then filtered under nitrogen and analysed for thiocyanate. No details of	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for thiocyanate. No details of analytical methods were given. Transference of material, as far as	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10 ⁻⁵ S m ⁻¹ was obtained. The
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for thiocyanate. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made	<pre>SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of 5 x 10⁻⁵ S m⁻¹ was obtained. The solvent was stored in the dark. ESTIMATED ERROR:</pre>
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysical methods were given. Transference of material, as far as possible, was carried out in a nitrogen	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of $5 \times 10^{-5} \text{ sm}^{-1}$ was obtained. The solvent was stored in the dark.
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide $(15-20 \text{ g})$ in Pyrex tubes $(15 \times 2.5 \text{ cm})$ and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for thiocyanate. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of $5 \times 10^{-5} \text{ Sm}^{-1}$ was obtained. The solvent was stored in the dark. ESTIMATED ERROR: Solubility $\pm 1\%$
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for thiocyanate. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of $5 \times 10^{-5} \text{ Sm}^{-1}$ was obtained. The solvent was stored in the dark. ESTIMATED ERROR: Solubility $\pm 1\%$ Temperature ± 0.05 °C (author)
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide $(15-20 \text{ g})$ in Pyrex tubes $(15 \times 2.5 \text{ cm})$ and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for thiocyanate. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised, powdered and dried in vacuum at > 100 °C. Formamide (Reidel Pure) was distilled under reduced pressure after extensive prior purification (1). Purity was checked by conductance and density measurements. A conductance of $5 \times 10^{-5} \text{ Sm}^{-1}$ was obtained. The solvent was stored in the dark. ESTIMATED ERROR: Solubility \pm 1% Temperature \pm 0.05 °C (author) REFERENCES:
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to formamide $(15-20 \text{ g})$ in Pyrex tubes $(15 \times 2.5 \text{ cm})$ and heating to nearly 10 °C above the temperature of the bath. The attainment of the equilibrium was checked by intermittent analyses of the solutions. The solutions were then filtered under nitrogen and analysed for thiocyanate. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made	SOURCE AND PURITY OF MATERIALS:A.R. grade ammonium thiocyanate (BDH)was recrystallised, powdered and driedin vacuum at > 100 °C.Formamide (Reidel Pure) was distilledunder reduced pressure after extensiveprior purification (1). Purity waschecked by conductance and densitymeasurements. A conductance of $5 \times 10^{-5} \text{ Sm}^{-1}$ was obtained. Thesolvent was stored in the dark.ESTIMATED ERROR:Solubility \pm 1%Temperature \pm 0.05 °C (author)REFERENCES:1. Paul, R.C.; Singla, J.P.; Gill, D.S.;

1	COMPONENTS :	EVALUATOR:
	(1) Magnesium chloride; MgC1 ₂ ; [7786-30-3] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland.
		December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of $MgCl_2$ in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. In this study, the conductance of the formamide was in the range 0.9 to 1.4×10^{-4} S m⁻¹.

While the result of only this one investigation is available, it would appear that the measurement has been undertaken with sufficient care for it to be taken as a recommended value. Good agreement has been shown between the results for CaCl₂ obtained by the above workers and those of Gopal and Husain (3). However the recommended method of preparing anhydrous MgCl₂ (by passing dry HCl over the hydrated double salt, MgCl₂. NH₄Cl) was not followed and so the purity of the solute is unknown. Hence only a tentative value for the solubility is recommended.

Tentative value at 298 K

 $84 \stackrel{+}{=} 4 \text{ g/kg of solvent.}$

References

- 1. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., <u>1968</u>, 38, 814-819.
- 2. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.
- 3. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963, 40, 272-274.

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COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Magnesium chloride; MgCl ₂ ;[7786-30-3] (2) Formamide; CH ₃ NO; [75-12-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of magnesium chloride in formamide at 25 $^{\circ}$ C

8.40 g/100 g solvent

	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated magnesium chloride solutions were prepared by dissolving excess of the halide in about 3 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and	Magnesium chloride, reagent grade, was dried under high vacuum before use. Commercial formamide (C. Erba, RP Product) was purified by storing over CaO for 2 days before twice distilling under reduced pressure. The conductance of the formamide varied from 0.9 to 1.40 x 10 ⁻⁴ S m ⁻¹ .
the amount of dissolved halide determined	ESTIMATED ERROR:
by a potentiometric method using a	Solubility ± 1%
titrimeter (type Radiometer TTT 1) and, presumably, standard aqueous silver nitrate. Equilibrium between solid and solution	Temperature ± 0.05 ^O C (author)
phases was considered to be attained when	REFERENCES:
analysis of the solution, made at progressive periods of time, gave the same	1. Pistoia, G.; Pecci, G.; Scrosati, B.
value for dissolved halide. The applicability of the method was checked by increasing the ratio of formamide to aqueous	Ric. Sci., <u>1967</u> ,37, 1167-1172
solution (1:5).	
The equivalence point remained the same (1)	

COMPONENTS: EVALUATOR: (1) Calcium chloride; CaCl₂; [10043-52-4] C.A. Vincent, Department of Chemistry, (2) Formamide; CH₂NO; [75-12-7] University of St.Andrews, St. Andrews, Fife, Scotland. December 1978 CRITICAL EVALUATION: Three investigations of the solubility of CaCl, in formamide have been reported. As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (1) is required to lower the water content to below 0.01 mol dm^{-3} and the conductance to below 2 x 10^{-5} S m⁻¹. Since preferential solvation of the ions by water is probable, use of wet solvent might give rise to elevated estimates of solubility. The solubility reported by Colton and Brooker (2), a value of 222 g/dm^{-3} of saturated solution at 298 K, cannot be compared with the molal solubilities given by the other workers since densities were not measured. However this result is likely to be high since the solvent was not purified and may have contained significant quantities of water. The solubility at 298 K was measured by Berardelli et al. (3) and at five temperatures between 308 and 328 K by Gopal and Husain (4). To check the consistency of the two investigations, all six values were subjected to a regression analysis where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was found (correlation coefficient -0.993). Recommended values 298 K

202 [±] 3 g/kg of solvent <u>308 K</u> 224 [±] 3 g/kg of solvent <u>318 K</u> 249 [±] 3 g/kg of solvent <u>328 K</u> 271 [±] 3 g/kg of solvent. <u>References</u> 1. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), <u>1966</u>, 362-366 2. Colton, E.; Brooker, R.E. J. Phys. Chem. <u>1958</u>, 62, 1595-1596. 3. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., <u>1968</u>, 38, 814-819

4. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963, 40, 272-274.

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Formamide

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Formamide /5		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Calcium chloride; CaCl ₂ ;[10043-52-4] (2) Formamide; CH ₃ NO; [75-12-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819	
VARIABLES:	PREPARED BY:	
One temperature: 25 °C	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of calcium chloride ir	formamide at 25 ^o C.	
20.0 g/100 g solvent.		
AUXILIARY	INFORMATION	
METHOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:	
Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated calcium chloride solutions were prepared by dissolving excess of the halide in about 3 20-25 cm ³ of solvent contained in a 50 cm Pyrex flask with a standard taper joint. The solution was then stored for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and	Calcium chloride, reagent grade, was dried under high vacuum before use. Commercial formamide (C. Erba, RP Product) was purified by storing over CaO for 2 days before twice distilling under reduced pressure. The conductance of the formamide varied from 0.9 to 1.40 x 10 ⁻⁴ S m ⁻¹ .	
the amount of dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT 1) and, presumably, standard aqueous silver nitrate. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of formamide to aqueous solution (1:5). The equivalence point remained the same (1).	ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.05 °C (author) REFERENCES: 1. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172	

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76 Forma	amide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Calcium chloride; CaCl ₂ ; [10043-52-4]	Gopal, R.; Husain, M.M.
(2) Formamide; CH ₃ NO; [75-12-7]	J. Ind. Chem. Soc., <u>1963</u> , 40, 272-274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o c	g/100 g
35	22.9
40	22.9 23.6
45	24.5
50	25.9
55	27.2
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of CaCl, were prepared at $10-15^{\circ}$ above the temperature at which the	A.R. grade calcium chloride.
solubility was to be measured before	Commercial formamide was redistilled and stored out of contact with light in
thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a	amber-coloured bottles until used.
pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ .	
Two more samples were taken for each	
temperature and the amount of solute present determined by one of the following methods.	
(1) Evaporation Method - Weighed samples were	
samples were then cooled and weighed in a	ESTIMATED ERROR:
to obtain a constant weight. All three	Solubility ± 1%
samples taken were treated in a similar	Temperature ± 0.1 °C (compiler)
(2) Precipitation Method - Weighed samples	
of saturated solutions were diluted with water and the halide content was determined	ALF LIGHTED;
gravimetrically. Reliability of procedure	ļ
formamide and estimating gravimetrically.	
When experimental and theoretical values agreed within ±0.5% the procedure was	
applied to the samples of unknown solubility.	
temperature and the amount of solute present determined by one of the following methods. (1) Evaporation Method - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure was tested using known amounts of KCl in formamide and estimating gravimetrically. When experimental and theoretical values agreed within ±0.5% the procedure was	ESTIMATED ERROR: Solubility ± 1%

Formanide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Calcium chloride; CaCl ₂ ; [10043-52-4] (2) Formamide; CH ₃ NO; [75-12-7]	Colton, E.; Brooker, R.E. J. Phys. Chem., <u>1958</u> , 62, 1595-1596
VARIABLES:	PREPARED BY:
One temperature: 25 ⁰ C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of calcium chloride in formamide at 25 $^{\circ}$ C 222 g/1000 cm ³ of saturated solution	
	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS;
Saturated solutions of calcium chloride in formamide were prepared in glass containers so that an excess of solid was always present After tumbling for a minimum of 48 hours in a thermostatted water bath the solutions were filtered quickly and aliquots taken for analysis. Duplicate samples were run. The calcium was determined photometrically after decomposing the formamide. A Beckman DU spectrophotometer was used.	Reagent grade calcium chloride, Commercial formamide (Fisher Scientific Co) with a freezing point of 2.50 [°] C was used without further purification.
	ESTIMATED ERROR: Solubility ⁺ 5% (compiler)
	Temperature $\stackrel{+}{=} 1 {}^{\circ}C$ (author)
	REFERENCES :

COMPONENTS:	EVALUATOR:
(1) Calcium bromide; CaBr ₂ ; [7789-41-5] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland.

rews, Fife, d. December 1979

CRITICAL EVALUATION:

One investigation of the solubility of CaBr, in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm^{-3} and the conductance to below 2 x 10^{-5} S m⁻¹. In this study, the conductance of the formamide was in the range 0.9 to $1.4 \times 10^{-4} \text{ S m}^{-1}$.

While the result of only this one investigation is available, it would appear that the measurement has been undertaken with sufficient care for it to be taken as a recommended value. Good agreement has been shown between the results for CaCl, obtained by the above workers and those of Gopal and Husain (3).

Recommended value at 298 K

 $434 \stackrel{+}{=} 4 \text{ g/kg of solvent.}$

References

1. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819.

2. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.

3. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963,40, 272-274.

COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Calcium bromide; CaBr₂; [7789-41-5] (2) Formamide; CH₃NO; [75-12-7] 	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 ⁰ C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of calcium bromide in formamide at 25 °C

43.4 g/ 100 g solvent.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Considerable effort was made to obtain water free solutions and all the solutions were Prepared in a dry box. Saturated calcium bromide solutions were prepared by dissolving excess of the halide in about 3 20-25 cm³ of solvent contained in a 50 cm³ Pyrex flask with a standard taper joint. The solution was then stored for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT 1) and, Dresumeter

presumably, standard aqueous silver nitrate. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of formamide to aqueous solution (1:5). The equivalence point remained the same (1) SOURCE AND PURITY OF MATERIALS:

Calcium bromide, reagent grade, was dried under high vacuum before use. Commercial formamide (C. Erba, RP Product) was purified by storing over CaO for 2 days before twice distilling under reduced pressure. The conductance of the formamide varied from 0.9 to $1.40 \times 10^{-4} \text{ Sm}^{-1}$.

ESTIMATED ERROR:

Solubility

Temperature

REFERENCES:

 Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, 1967, 37, 1167-1172

± 1%

 \pm 0.05 °C (author)

10111	annas
COMPONENTS :	EVALUATOR:
(1) Strontium chloride; SrCl ₂ ; [10476-85-4] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

CRITICAL EVALUATION:

One investigation of the solubility of SrCl, in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm^{-3} and the conductance to below $2 \times 10^{-5} \text{ sm}^{-1}$. In this study, commercial formamide was redistilled, but no further drying operations were carried out. Since preferential solvation of the ions by water is likely, somewhat elevated estimates of solubility may be given. On the other hand, where solubilities obtained by Gopal and Husain (1) can be compared with the results of other workers using very dry formamide, reasonable agreement is usually found.

To check the internal consistency of the results, a regression analysis was undertaken where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was obtained with a correlation coefficient of -0.994. On the basis of this analysis the following solubility values are recommended.

Recommended values

т/к	Solubility (g/kg of solvent)
293	149.7 [±] 1.5
298	156.9 [±] 1.5
303	164.1 [±] 1.5
308	171.4 + 1.5
313	178.8 + 2.0
318	186.3 [±] 2.0
323	194.0 [±] 2.0

References

Gopal, R.; Husain, M.M. J. Indian Chem. Soc., <u>1963</u>, 40, 272-274
 Notley, J.M.; Spiro, M. J. Chem. Soc. (B), <u>1966</u>, 362-366.

Forma	amide
COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Strontium chloride; SrCl₂; [10476-85-4]</pre>	Gopal, R.; Husain, M.M.
(2) Formamide; CH ₃ NO; [75-12-7]	J. Ind. Chem. Soc., 1963, 40, 272
5	
ARIABLES:	PREPARED BY:
Cemperature	J.S. McKechnie
XPERIMENTAL VALUES:	
t/ [°] C	(100
·	g/100 g solvent
20 25	15.0
30	15.9 16.2
30	
35 40	17.0
40	18.1
-	18.8
50	19.3
	INFORMATION
ETHOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS;
Saturated solutions of SrC1 were prepared	
at 10-15 shows the temperature of which '	A.R. grade strontium chloride.
^{che} solubility was to be measured before	A.R. grade strontium chloride. Commercial formamide was redistilled
the solubility was to be measured before thermostatting at the required temperature.	Commercial formamide was redistilled and stored out of contact with light
the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica	Commercial formamide was redistilled
the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P.O	Commercial formamide was redistilled and stored out of contact with light
pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each	Commercial formamide was redistilled and stored out of contact with light
the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods.	Commercial formamide was redistilled and stored out of contact with light
the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) Evaporation Method - Weighed samples	Commercial formamide was redistilled and stored out of contact with light
the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in	Commercial formamide was redistilled and stored out of contact with light
the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR:
the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1%
The solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner.	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR:
The solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1%
The solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 °C (compiler)
The solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure was tested using known amounts of KCl in	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 °C (compiler)
The solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure Was tested using known amounts of KC1 in formamide and estimating gravimetrically.	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 °C (compiler)
The solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with	Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 °C (compiler)

COMPONENTS: (1) Strontium bromide; SrBr₂;[10476-81-0] (2) Formamide; CH₃NO; [75-12-7] Scotland. December 1978

CRITICAL EVALUATION:

One investigation of the solubility of SrBr_2 in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. In this study, commercial formamide was redistilled, but no further drying operations were carried out. Since preferential solvation of the ions by water is likely, somewhat elevated estimates of solubility may be given. On the other hand, when solubilities obtained by Gopal and Husain (1) can be compared with the results of other workers using very dry formamide, reasonable agreement is usually found.

To check the internal consistency of the results, a regression analysis was undertaken where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was obtained with a correlation coefficient of -0.999. On the basis of this analysis the following solubility values are recommended.

Recommended values

т/к	Solubility (g/kg of solvent)
298	190.5 ⁺ 2.0
303	212.6 ± 2.0
308	234.6 [±] 2.0
313	263.2 + 2.0
318	285.9 + 2.0
323	321.5 + 2.0

References

1. Gopal, R.; Husain, M.M. J. Indian Chem. Soc., 1963, 40, 272-274.

2. Notley, J.M.; Spiro, M. J. Chem. Soc. (B), 1966, 362-366.

Formamide

Forma	mide 83
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Strontium bromide; SrBr ₂ ; [10476-81-0] (2) Formamide; CH ₃ NO; [75-12-7]	Gopal, R.; Husain, M.M. J. Ind. Chem. Soc., <u>1963</u> , 40, 272-274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ [°] C	g/100 g solvent
25	19.1
30	21.3
35	23.5
40	26.3
45	28.6
50	32.2
	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of SrBr ₂ were prepared	SOURCE AND PURITY OF MATERIALS: A.R. grade strontium bromide.
at 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) Evaporation Method - Weighed samples were carefully heated to expel the solvent.	A.K. grade strontium bromide. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar fashion. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure was tested using known amounts of KC1 in formamide and estimating gravimetrically. When experimental and theoretical values agreed within ±0.5% the procedure was applied to the samples of unknown solubility.	ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 ^O C (compiler) REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Barium chloride; BaC1 ₂ ;[10361-37-2] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of $BaCl_2$ in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. In this study, commercial formamide was redistilled, but no further drying operations were carried out. Since preferential solvation of the ions by water is likely, somewhat elevated estimates of solubility may be given. On the other hand, where solubilities obtained by Gopal and Husain (1) can be compared with the results of other workers using very dry formamide, reasonable agreement is usually found.

To check the internal consistency of the results, a regression analysis was undertaken where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was obtained with a correlation coefficient of -0.999. On the basis of this analysis the following solubility values are recommended.

Recommended values

T/K	Solubility (g/kg of solvent)
298	117.6 [±] 1.5
303	124.6 ± 1.5
308	131.7 [±] 1.5
313	139.0 [±] 1.5
318	146.4 [±] 1.5
323	154.2 [±] 1.5

References

Gopal, R.; Husain, M.M. J. Indian Chem. Soc., <u>1963</u>, 40, 272-274.
 Notley, J.M.; Spiro, M. J. Chem Soc.(B), <u>1966</u>, 362-366.

Form	amide
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Barium chloride; BaCl ₂ ; [10361-37-2]	Gopal, R.; Husain, M.M.
(2) Formamide; CH ₃ NO; [75-12-17]	J. Ind. Chem. Soc., <u>1963</u> , 40, 272-274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	11.8
30	12.5
35	13.1
40	13.0
45	14.6
50	15.4
	INFORMATION
ETHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
ETHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl were prepared a	SOURCE AND PURITY OF MATERIALS:
ETHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl ₂ were prepared a 10-15° above the temperature at which the solubility was to be measured before	SOURCE AND PURITY OF MATERIALS: t A.R. grade barium chloride. Commercial formamide was redistilled
ETHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl ₂ were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature.	SOURCE AND PURITY OF MATERIALS: t A.R. grade barium chloride.
ETHOD/APPARATUS/PROCEDURE: Saturated solutions of BaC1, were prepared a lo-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica	SOURCE AND PURITY OF MATERIALS: t A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light
ETHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl, were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P 0.	SOURCE AND PURITY OF MATERIALS: t A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light
ETHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl, were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
ETHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl, were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods.	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
ETHOD /APPARATUS/PROCEDURE: Saturated solutions of BaCl, were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each 5 temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent.	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
THOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl, were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR:
ETHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl, were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 12
METHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl, were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) Evaporation Method - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR:
METHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl, were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) Evaporation Method - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) Precipitation Method - Weighed samples	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 °C (compiler)
METHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl ₂ were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 12
METHOD/APPARATUS/PROCEDURE: Saturated solutions of BaC1_ were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each ⁻ temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 °C (compiler)
METHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl ₂ were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute present determined by one of the following methods. (1) Evaporation Method - Weighed samples were carefully heated to expel the solvent. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All three samples taken were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined gravimetrically. Reliability of procedure was tested using known amounts of KCl in	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 °C (compiler)
METHOD/APPARATUS/PROCEDURE: Saturated solutions of BaCl, were prepared a 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each ⁵ temperature and the amount of solute present determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent. The samples were treated in a similar manner. (2) <u>Precipitation Method</u> - Weighed samples of saturated solutions were diluted with water and the halide content was determined	SOURCE AND PURITY OF MATERIALS: A.R. grade barium chloride. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used. ESTIMATED ERROR: Solubility ± 1% Temperature ± 0.1 °C (compiler) REFERENCES:

COMPONENTS :	EVALUATOR:
(1) Barium bromide; BaBr ₂ ;[10553-31-8] (2) Formamide; CH ₃ NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

CRITICAL EVALUATION:

One investigation of the solubility of $BaBr_2$ in formamide has been reported (1). As in other measurements of salt solubility in this solvent, the accuracy of the data is likely to be determined primarily by the purity of the solvent used, rather than by the precision of the analytical methods. Formamide is a thermally unstable, photosensitive and hygroscopic liquid, and a very elaborate purification procedure (2) is required to lower the water content to below 0.01 mol dm⁻³ and the conductance to below 2×10^{-5} S m⁻¹. In this study, commercial formamide was redistilled, but no further drying operations were carried out. Since preferential solvation of the ions by water is likely, somewhat elevated estimates of solubility may be given. On the other hand, when solubilities obtained by Gopal and Husain (1) can be compared with the results of other workers using very dry formamide, reasonable agreement is usually found.

To check the internal consistency of the results, a regression analysis was undertaken where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was obtained with a correlation coefficient of -0.946. On the basis of this analysis the following solubility values are recommended.

Recommended values

T/K	Solubility (g/kg of solvent)
298	305.0 [±] 3.0
303	318.2 [±] 3.0
308	331.5 [±] 3.0
313	344.9 [±] 3.0
318	358.4 [±] 3.0
323	372.3 [±] 3.0

References

Gopal, R.; Husain, M.M. J. Indian Chem. Soc., <u>1963</u>, 40, 272-274
 Notley, J.M.; Spiro, M. J. Chem. Soc. (B), <u>1966</u>, 362-366.

	namide
OMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Barium bromide; BaBr ₂ ; [10553-31-8] (2) Formamide; CH ₃ NO; [75-12-7]	Gopal, R.; Husain, M.M. J. Ind. Chem. Soc., <u>1963</u> , 40,272-274
ARIABLES: Temperature	PREPARED BY: J.S. McKechnie
XPERIMENTAL VALUES:	
a DATENTAL VALUES:	
τ/°c	g/1Q0 g solvent
25	29.9
30	31.5
35	34.5
40	35.4
45	35.5
50	36.5
AUXILIAR	(INFORMATION
ETHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
ÆTHOD/APPARATUS/PROCEDURE: Saturated solutions of BaBr, were prepared	SOURCE AND PURITY OF MATERIALS:
G THOD/APPARATUS/PROCEDURE: Saturated solutions of BaBr ₂ were prepared 10-15 ^o above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-5 cm ³) were withdrawn with a pro-pipette and weighed in a closed silica dish protected from moisture with P ₂ O ₅ . Two more samples were taken for each temperature and the amount of solute presen determined by one of the following methods. (1) <u>Evaporation Method</u> - Weighed samples were carefully heated to expel the solvent.	SOURCE AND PURITY OF MATERIALS: at A.R. grade barium bromide. Commercial formamide was redistilled and stored out of contact with light in amber-coloured bottles until used.
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COMPONENTS:	EVALUATOR:
 (1) Lithium chloride; LiC1; [7447-41-8] (2) N-methylformamide; C₂H₅NO; [123-39-7] 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Three investigations of the solubility of LiCl in N-methylformamide have been reported, all within the last fifteen years (1-3). N-methylformamide is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (4). Later, Feakins and Lawrence (5) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls, and recommended baking out containers, etc., where possible, before use.

It should be noted that in two of the current solubility studies (those by Strack et al. (1) and by Paul et al. (3)), the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent.

A major problem in the determination of the solubility of LiCl in N-methylformamide has, however, been detected by Strack et al. (1). These workers have shown that the equilibrium state of the solid phase is almost certainly a solvate, although its composition was not determined. Further, not only was the normal solubility equilibrium impossible to measure with any certainty, but no equilibrium of any kind had been established at the end of three weeks at 298 K.

An additional problem is that none of the investigators followed the recommended procedure for preparing anhydrous LiC1 (by passing dry HC1 over the salt) so that the purity of the solute is unknown.

A mean value for the solubility of LiCl of 230 \pm 20 g/kg of solvent is obtained if results from all three investigations are considered. However in view of the likelihood of solvate formation, no recommended value is given for this salt.

References

1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, <u>1964</u>, 9, 416-418.

- 2. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973,11, 1024-1026.
- 4. French, C.M.; Glover, K.H.; Trans. Farad, Soc., 1955, 51, 1418-1427.
- 5. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

ONEGENAL MEASUREMENTS: (1) Lithium chloride; LiCl; [7447-41-8] (2) N=methylformamide; C_2H_NO; [123-39-7] (2) N=methylformamide; C_2H_NO; [123-39-7] VARLARLES: Temperature J.S. McKechnie 20.6 35 20.6 35 45 AUXILIARY INFORMATION METHOD/APPARATUS/FPROCEDURE: SILVERATUS/FPROCEDURE: SILVERATUS/FPROCEDURE: SILVERATUS/FPROCEDURE: SOUNCE AND FURIT OF MATERIALS: AUXILIARY INFORMATION METHOD/APPARATUS/FPROCEDURE: SOUNCE AND FURIT OF MATERIALS: SUBJOR AND FURT OF MATERIALS:	N-Methyl	formamide 8
(2) N-methylformæmide; C ₂ H ₃ NO; [123-39-7] Gill,D.S.; Narula, S.P. Indian J. Chem., 1973, 12, 1024-1026 VARLABLES: PREPARED BY: Temperature J.S. McKechnie EXPERIMENTAL VALUES: ¢/°C ¢/°C g/100 g solvent 25 20.6 35 15.1 45 12.9 KTHOD/APPARATUS/PROCEDURE: Saturated solutions of lithium chloride were prepared by adding excess of the powdered st to N-enthylformænide (15-20 g) in Prex these (15 x 2.5 cm) and preheating to 10 °C the content of the equilabilithic water intergen and profiler to final thermostating. The the content intergen and malyyed filteria malyses. The solutions ere the filteria malyses. The solutions of material, as far as graves how as carried out in a mitrogen filled dy box. Kansurements were made in triplicate. Source AND PURITY OF MATERIALS: A.R. grade lithium chloride BMD was crystallised and dried under vacuum at tion of the solvent was purified by fraction- tices of the solvent was purified by fraction- tices of the solvent was torred in dark coloured bottles and kept in a dry box. Physical constants were rehecked before use. Source AND PURITY DERROR: Solubility 112	COMPONENTS:	ORIGINAL MEASUREMENTS:
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COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium chloride; LiCl; [7447-41-8]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M.
	Ric. Sci., 1 <u>968,</u> 38, 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of lithium chloride in N	-methylformamide at 25 °C
23.9 g/100 g solvent	
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AUXILIARY	INFORMATION
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: Considerable effort was made to obtain water	SOURCE AND PURITY OF MATERIALS: Lithium chloride, reagent grade, was dried
free solutions and all the solutions were	under high vacuum before use.
prepared in a dry box. Saturated lithium chloride solutions were prepared by dissoly-	N-methylformamide (Fluka product) was purified by storing over CaO for two days
ing excess of the halide in about 20-25 cm ³	before twice distilling under reduced
of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution	pressure. The conductance of the N-methyl-
with a standard taper joint. The solution was then stirred for three or more days in a	formamide varied from 3.0 to 6.0 x 10^{-4} s m ⁻¹ .
thermostatted bath. With the aid of a	
syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was	
diluted with water (1:20) and the amount of	
dissolved halide determined by a potentio- metric method using a titrimeter (type	ESTIMATED ERROR:
Radiometer TTT1) and, presumably, standard	Solubility = 1%
aqueous silver nitrate. Equilibrium between solid and solution	Temperature ⁺ 0.05 ^o C (author)
phases was considered to be attained when	REFERENCES :
analysis of the solution, made at progressive periods of time, gave the same value for	1. Pistoia, G.; Pecci, G.; Scrosati, B.
dissolved halide. The applicability of the	Ric. Sci., <u>1967</u> , 37, 1167-1172
method was checked by increasing the ratio of N-methylformamide to aqueous solution	<u> </u>
(1:5). The equivalence point remained	
the same(1).	

COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Lithium chloride; LiCl; [7447-41-8]	Strack, G.A.; Swanda, K.S.; Bahe, L.W.	
(2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	J. Chem. Eng. Data, <u>1964</u> , 9, 416-418	
2 5		
VARIABLES:		
VOLTABLES :	PREPARED BY:	
One temperature: 25 °C	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of lithium chloride in N-methylfor	mamide at 25 C	
24.2 g/100 g solvent.		
	INFORMATION	
METHOD/APPARATUS/PROCEDURE:		
An excess of lithium chloride and 10 cm 3 of	SOURCE AND PURITY OF MATERIALS; Anhydrous A.R. grade lithium chloride was	
N-methylformamide in a carefully stoppered	used without further purification after	
test tube were placed in a constant temper- ature bath for 2-4 days. The test tubes	drying at 105 ^{-O} C for 24 hours. N-methylformamide was prepared by either	
were shaken several times each day. Satur- ation was assumed when samples taken at	of two methods: the addition of methylamine to formic acid(2) or by the classical method	
least one day apart gave identical concentr-	of the addition of methylamine to ethyl	
ations. 5 cm ³ samples were withdrawn with a pipette equipped with a glass wool filter	formate:(3). The N-methylformamide was fractionally recrystallised until the	
over the tip, and transferred to a glass	conductance reached a value of between 10 ⁻¹	
stoppered weighing bottle. The contents of the weighing bottle were diluted to 100 cm ³	(continued)	
with water and the halide titrated by Fajan's	ESTIMATED ERROR:	
method, known samples of halide salts mixed	Solubility ⁺ 1%	
with N-methylformamide in water were analysed and the results reported as	Temperature ⁺ 0.1 ^o C (author)	
excellent. Berger and Dawson(1) also	REFERENCES:	
report successful analyses of halides in the presence of formamide by Fajan's method.	1. Berger, C.; Dawson, L.R. Anal. Chem.,	
Densities were not as accurate as the Corresponding weight data since they relied	<u>1952</u> , 24, 994-996.	
On temperature dependent pipette volumes.		
As the densities were used to calculate	(continued)	
(continued)		

COMPONENTS: (1) Lithium chloride; LiCl; [7447-41-8] (2) N-methylformamide; C₂H₅NO; [123-39-7] ORIGINAL MEASUREMENTS: Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, <u>1964</u>, 9, 416-418

METHOD/APPARATUS/PROCEDURE:

(continuation)

concentrations on the molar scale the results are estimated to be accurate to within 2% on that scale, and to within 1% on the molal and mole ratio scales.

SOURCE AND FURITY OF MATERIALS: (continuation)

and 10^{-2} S m⁻¹. It was stored in tightly-stoppered brown glass bottles at room temperature.

The physical constants of the N-methylformamide were (literature values in parentheses): $n_D^{25} = 1.4305 [1.4300(4), 1.4310(2)]; \rho_4^{21} = 1.01 [1.008 at 15 °C(5)]; B.Pt. = 62 °C at$ 6 mm of Hg [51 °C at 1 mm of Hg(5)].

In all cases, salts and solvent were stored and transferred in such a manner as to minimise the absorption of atmospheric moisture, presumably by the use of a dry box.

REFERENCES: (continuation)

2. Leader, G.R.; Gormley, J.F. J. Am. Chem. Soc., 1952, 73, 5731-5733.

3. Bordwell, F.G. "Organic Chemistry", p446, Macmillan, N.Y., 1963.

4. D'alelio, G.F.; Reid, E.E. J. Am. Chem. Soc., 1937, 59, 109-111.

5. French, C.M.; Glover, K.H. Trans. Faraday Soc., 1955, 51, 1418-1427.

COMPONENTS:	EVALUATOR:
(1) Lithium bromide; LiBr; [7550-35-8] .(2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	C.A. Vincent Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland. December 1978

Two investigations of the solubility of LiBr in N-methylformamide have been reported (1-2) N-methylformamide is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (3). Later Feakins and Lawrence (4) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use.

In the second of the current solubility studies (that by Paul et al. (2)), the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent. The solvent used by Berardelli et al. (1) had a mean conductance of 5×10^{-4} S m⁻¹.

In a parallel study of the solubility of LiCl in N-methylformamide, Strack et al.(5) showed that the equilibrium state of the solid phase was not pure LiCl, but a solvate of unidentified composition. The formation of such a phase made the determination of the solubility of LiCl virtually impossible. While no reference to solvate formation by LiBr in N-methylformamide is made by either group, the disparate results obtained (540 g/kg of solvent (1) and 352 g/kg of solvent (2) at 298 K) suggest that such a phenomenon may also occur with this salt. It might be noted that equilibrium had still not been established with LiCl after three weeks. No recommended value is therefore given for LiBr.

- 1. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. *Ric. Sci.*, <u>1968</u>, *38*, 814-819.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 3. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 4. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.
- 5. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416;418.

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Lithium bromide; LiBr; [7550-35-8]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem.,
(2) N-methylformamide; C ₂ H ₅ NO;[123-39-7]	1973, <i>11</i> , 1024–1026.
	<u>1979</u> , 11, 1024 10201
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	35.2
35	22.0
45	11.7
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS:	
Saturated solutions of lithium bromide were	A.R. grade lithium bromide (BDH) was
prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyrex	recrystallised and dried under vacuum at 110-125 °C.
tubes (15 x 2.5 cm) and preheating to 10 $^{\circ}$ C	N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry
above the required temperature of the bath prior to final thermostatting. The	nitrogen atmosphere and had the following
attainment of the equilibrium was checked by intermittent analyses. The solutions were	physical constants: B.Pt. 52-53 ^o C/1 mm; specific conductance 3-4 x 10 ⁻⁵ S m ⁻¹ ;density
then filtered under nitrogen and analysed	0.9986 g/cm ³ ; refractive index 1.4312 at
for halide. No details of analytical methods were given. Transference of material, as	25 °C. The solvent was stored in dark coloured bottles and kept in a dry box.
far as possible, was carried out in a	Physical constants were rechecked before use.
nitrogen filled dry box. Measurements were made in triplicate.	ESTIMATED ERROR: Solubility ⁺ 1Z
•	Solubility - 1% Temperature - 0.05 [°] C (author)
	REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Lithium bromide; LiBr; [7550-35-8] (2) N-methylformamide; C₂H₅NO; [123-39-7] 	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES: One temperature: 25 ^O C	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	

Solubility of lithium bromide in N-methylformamide at 25 °C

54.0 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated lithium bromide solutions were prepared by dissolv-ing excess of the halide in about 20-25 cm³ of solvent contained in a 50 cm³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT1) and, presumably, standard aqueous silver nitrate.

Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of N-methylformamide to aqueous solution (1:5). The equivalence point remained the same(1).

SOURCE AND PURITY OF MATERIALS:

Lithium bromide, reagent grade, was dried under high vacuum before use. N-methylformamide (Fluka product) was

purified by storing over CaO for two days before twice distilling under reduced pressure. The conductance of the N-methyl formamide varied from 3.0 to 6.0 x 10⁻⁴S m⁻¹.

ESTIMATED ERROR:

Solubility $\stackrel{+}{-}$ 1% Temperature $\stackrel{+}{-}$ 0.05 °C (author)

REFERENCES:

 Pisţoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, <u>1967</u>, 37, 1167-1172

COMPONENTS :	EVALUATOR:
(1) Sodium chloride; NaCl; [7647-14-5] (2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Three investigations of the solubility of NaCl in N-methylformamide have been reported, all within the last fifteen years (1-3). N-methylformamide is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (4). Later, Feakins and Lawrence (5) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls, and recommended baking out containers, etc., where possible, before use.

It should be noted that in two of the current solubility studies (those by Strack et al. (1) and by Paul et al. (3)), the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent. The other major difficulty which may arise in solubility measurements in N-methylformamide, namely solvate formation, has not been detected in any of the three studies of NaCl.

The solubilities reported at 298 K by Strack et al. (1) (32.2 g/kg of solvent) and by Berardelli et al. (2) (32.9 g/kg of solvent) are in excellent agreement. The value given by Paul et al. (3) (56.0 g/kg of solvent) is almost a factor of two higher and is rejected. There is no obvious reason for such a discrepancy, but it might be noted that solubilities quoted by Paul et al. for chlorides in N-methylformamide are generally higher than those given by other groups. No details of analytical methods were given by Paul et al. (3).

Recommended value at 298 K

 $32.6 \stackrel{+}{=} 1.0 \text{ g/kg of solvent.}$

Tentative values at other temperatures

Strack et al. (1), who measured solubilities over the temperature range 273-308 K, do not quote values at temperatures other than 298 K, but report a linear relationship between molal solubility and temperature, with a slowly increasing solubility with decreasing temperature. Paul et al. (3) who give values at 308 K and 318 K show a very sharp rise in solubility with decreasing temperature. Consideration of these results leads to, at best, an estimate of a minimum value of the solubility of 30.6 g/kg of solvent at 308 K.

References

1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416-418.

2. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819.

- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 4. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 5. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

N-Methylformamide		9
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Sodium chloride; NaCl;[7647-14-5]	Paul, R.C.; Singla, J.P.; Lamba,. M.S.;	
 (2) N-methylformamide; C₂H₅NO; [123-39-7] 	Gill, D.S.; Narula, S.P. Indian J. Chem.,	
	<u>1973, 11, 1024–1026</u>	
	<u>, , , , , , , , , , , , , , , , , , , </u>	
VARIABLES:		_
	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:	l	
t/ ^o C	g/100 g solvent	
25	5.60	
35	4.75	
45	3.32	
AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium chloride were prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyrex	SOURCE AND PURITY OF MATERIALS: A.R. grade sodium chloride (BDH) was recrystallised and dried under vacuum at 110-125 ^O C.	
tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of	N-methylformamide was purified by fraction ation of the solvent over sodamide in a dry nitrogen atmosphere and had the following physical constants: B.Pt. $52-53$ °C/1 mm; specific conductance $3-4 \times 10^{-5}$ S m ⁻¹ ; dens: 0.9986 g/cm ³ ; refractive index 1.4312 at 25 °C. The solvent was stored in dark coloured bottles and kept in a dry box.	y
material, as far as possible, was carried	Physical constants were rechecked before us	51
out in a nitrogen filled dry box. ^{Measurements} were made in triplicate.	ESTIMATED ERROR:	
	Solubility [±] 1%	
	Temperature ⁺ 0.05 [°] C (author)	
	REFERENCES :	

98 <i>N</i> -Methylformamide		
COMPONENTS: (1) Sodium chloride; NaCl; [7647-14-5] (2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	ORIGINAL MEASUREMENTS: Berardelli, M.L.; Pistoia, G.; Polcaro, A.M] Ric. Sci., <u>1968</u> , 38, 814-819	
VARIABLES: One temperature: 25 ^O C	PREPARED BY: J.S. McKechnie	
Solubility of sodium chloride in 3.29 g/100 g solvent	N-methylformamide at 25 ^o C	
AUXILIA	ARY INFORMATION	

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water	Sodium chloride, reagent grade, was dried
free solutions and all the solutions were	under high vacuum before use.
prepared in a dry box. Saturated sodium	N-methylformamide (Fluka product) was
chloride solutions were prepared by dissolv-	purified by storing over CaO for two days
ing excess of the halide in about $20-25$ cm ³	before twice distilling under reduced
of solvent contained in a 50 cm ³ Pyrex flask	pressure. The conductance of the N-methyl-
with a standard taper joint. The solution	formamide varied from 3.0 to 6.0 x 10^{-4} S m ⁻¹
was then stirred for three or more days in a	
thermostatted bath. With the aid of a	
syphon provided with a terminal G-3 gooch,	
an aliquot of the saturated solution was	
diluted with water (1:20) and the amount of	
dissolved halide determined by a potentio-	ESTIMATED ERROR:
metric method using a titrimeter (type	
Radiometer TTT1) and, presumably, standard	Solubility ⁺ 1%
aqueous silver nitrate.	Temperature $\stackrel{+}{=}$ 0.05 °C (author)
Equilibrium between solid and solution	Temperature 0.05 0 (author)
phases was considered to be attained when	
analysis of the solution, made at progressive	REFERENCES:
periods of time, gave the same value for	1. Pistoia, G.; Pecci, G.; Scrosati, B.
dissolved halide. The applicability of the	
method was checked by increasing the ratio	<i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1167–1172
of N-methylformamide to aqueous solution	
(1:5). The equivalence point remained	
the same(1).	

V-Meth	vlform	amide
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N-Methyltormamide 99		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
 (1) Sodium chloride; NaCl; [7647-14-5] (2) N-methylformamide; C₂H₅NO; [123-39-7] 	Strack. G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, <u>1964</u> , 9, 416-418	
VARIABLES: Temperature	PREPARED BY: J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of sodium chloride in N-methylform 3.22 g/100 g solvent Within the temperature rante O-35 °C the solu of solvent, S, are given by: S = -0.011 T + 6.419 Where T is the absolute temperature.		
METHOD/APPARATUS/PROCEDURE: An excess of sodium chloride and 10 cm ³ of N-methylformamide in a carefully stoppered test tube were placed in a constant temper- ature bath for 2-4 days. The test tubes were shaken several times each day. Satur- ation was assumed when samples taken at least one day apart gave identical concentr- ations. 5 cm ³ samples were withdrawn with a pipette equipped with a glass wool filter over the tip, and transferred to a glass stoppered weighing bottle. The contents of the weighing bottle were diluted to loo cm ³ with water and the halide titrated by Fajan's method. To check the reliability of the method, known samples of halide salts mixed with N-methylformamide in water were analysed and the results reported as	INFORMATION SOURCE AND PURITY OF MATERIALS: Anhydrous A.R. grade sodium chloride was used without further purification after drying at 105 °C for 24 hours. N-methylformamide was prepared by either of two methods: the addition of methyl- amine to formic acid (2) or by the classical method of the addition of methylamine to ethyl formate (3). The N-methylformamide was fractionally recrystallised until the conductance reached a value of between (continued) ESTIMATED ERROR: Solubility ⁺ / ₋ 1% Temperature ⁺ / ₋ 0.1 °C (author)	
excellent. Berger and Dawson (1) also report successful analyses of halides in the presence of formamide by Fajan's method. Densities were not as accurate as the corresponding weight data since they relied On temperature dependent pipette volumes. (continued)	REFERENCES: 1. Berger, C.; Dawson, L.R. Anal. Chem., <u>1952</u> , 24, 994-996.	

COMPONENTS:

Sodium chloride; NaCl; [7647-14-5]
 N-methylformamide; C₂H₅NO; [123-39-7]

ORIGINAL MEASUREMENTS:

Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416-418

METHOD/APPARATUS/PROCEDURE: (continuation)

As the densities were used to calculate concentrations on the molar scale the results are estimated to be accurate to within 2% on that scale, and to within 1% on the molal and mole ratio scales.

The solubilities in molal units were found to be linear functions of temperature over the range O-35 $^{\circ}$ C. The constants A and B in the equation m = AT + B were determined for each salt by linear regression analysis. No information is given concerning the standard errors of the regression coefficients. However by comparing the solubilities quoted for direct measurements at 25 $^{\circ}$ C with those derived from the linear equations, the calculated solubilities are within 0.6 - 2.5% of those derived by direct measurements.

SOURCE AND PURITY OF MATERIALS: (continuation)

 10^{-1} and 10^{-2} S m⁻¹. It was stored in tightly-stoppered brown glass bottles at room temperature. The physical constants of the N-methylformamide were (literature values in parentheses): $n_D^{25} = 1.4305 [1.4300(4), 1.4310(2)]; \rho_4^{21} = 1.01 [1.008 at 15 °C(5)]; B.Pt. = 62 °C$ at 6 mm. of Hg [51 °C at 1 mm of Hg(5)]. In all cases, salts and solvent were stored and transferred in such a manner as

to minimise the absorption of atmospheric moisture, presumably by the use of a dry box.

REFERENCES: (continuation)

2. Leader, G.R.; Gormley, J.F. J. Am. Chem. Soc., <u>1952</u>, 73, 5731-5733.

3. Bordwell, F.G. "Organic Chemistry", p.446, Macmillan, N.Y., 1963,

4. D'alelio, G.F.; Reid, E.E. J. Am. Chem. Soc., 1937, 59, 109-111

5. French, C.M.; Glover, K.H. Trans. Faraday Soc., 1955, 51, 1418-1427.

COMPONENTS:	EVALUATOR:
(1) Sodium bromide; NaBr; [7647-15-6] (2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

Three investigations of the solubility of NaBr in N-methylformamide have been reported, all within the last fifteen years (1-3). N-methylformamide is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (4). Later, Feakins and Lawrence (5) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls, and recommended baking out containers, etc., where possible, before use.

It should be noted that in two of the current solubility studies (those by Strack et al. (1) and by Paul et al. (3)), the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent. The other major difficulty which may arise in solubility measurements in N-methylformamide, namely solvate formation, has not been detected in any of the three studies of NaBr.

The solubilities reported at 298 K by all three groups show good agreement, having a mean value of 298.1 g/kg of solvent and a standard deviation of 9.8 g/kg of solvent, or 3%. There are no compelling grounds for rejecting the highest value (that of Berardelli et al. (2)) although the solvent used had a higher conductance than in the other studies. The mean of the three investigations is therefore taken to be the recommended value.

Recommended value at 298 K

 $298 \stackrel{+}{=} 10 \text{ g/kg of solvent}$

Tentative values at other temperatures.

Strack et al. (1), who measured solubilities over the temperature range 273-308 K, do not quote values at temperatures other than 298 K, but report a linear relationship between molal sulubility and temperature. Paul et al. (3) give values at 308 K and 318 K. At 308 K, the calculated value from (1), namely 283 g/kg of solvent is in reasonable agreement with that given in (3), 289.9 g/kg of solvent. Thus despite the fact that Paul et al. gave no details of their analytical methods, the following tentative values are probably acceptable:

<u>308 K</u>

287 [±] 10 g/kg of solvent

<u>318 K</u>

277 - 10 g/kg of solvent.

- 1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416-418
- ². Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. *Ric. Sci.*, <u>1968</u>, 38, 814-819.
- 3. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 4. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- ⁵. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), <u>1966</u>, 212-219.

102 N-Methy	/Iformamide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; [7647-15-6] (2) N-methylformamide; C ₂ H ₅ NO;[123-39-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026
VARIABLES:	PREPARED BY:
	J.S. McKechnie
Temperature	J.J. Ackelinite
EXPERIMENTAL VALUES:	
t/°c	g/100 g solvent
25	29.8
35	28.0
45	27.7
AUXILIAR	Y INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of sodium bromide were	A.R. grade sodium bromide (BDH) was recrystallised and dried under vacuum at
prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyres	110-125 °C.
tubes (15 x 2.5 cm) and preheating to 10° C above the required temperature of the bath	N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry
prior to final thermostatting. The	nitrogen atmosphere and had the following physical constants: B.Pt. 52-53 ^O C/1 mm;
attainment of the equilibrium was checked by intermittent analyses. The solutions were	specific conductance 3-4 x 10-5 S m-1; density
then filtered under nitrogen and analysed	0.9986 g/cm ³ ; refractive index 1.4312 at 25 °C. The solvent was stored in dark
for halide. No details of analytical method were given. Transference of material, as	coloured bottles and kept in a dry box.
far as possible, was carried out in a nitrogen filled dry box. Measurements were	Physical constants were rechecked before use. ESTIMATED ERROR:
made in triplicate.	Solubility ± 1%
	Temperature $\stackrel{+}{=}$ 0.05 °C (author)
	REFERENCES :
]	

N-Methylformamide 103		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Sodium bromide; NaBr; [7647-15-6]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M.	
(2) N-methylformamide; C ₂ H ₅ NO;[123-39-7]	<i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819	
VARIABLES:	PREPARED BY:	
One temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of sodium bromide in N-methylformamide at 25 °C		
30.8 g/100 g solvent		
<u>-</u>		
•		
	INFORMATION	
METHOD/APPARATUS/PROCEDURE: Considerable effort was made to obtain water	SOURCE AND PURITY OF MATERIALS: Sodium bromide, reagent grade, was dried	
free solutions and all the solutions were	under high vacuum before use.	
prepared in a dry box. Saturated sodium bromide solutions were prepared by dissolv-	N-methylformamide (Fluka product) was purified by storing over CaO for two days	
ing excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask	before twice distilling under reduced pressure. The conductance of the N-methyl-	
With a standard taper joint. The solution Was then stirred for three or more days in a	formamide varied from 3.0 to 6.0 x 10^{-4} S m ⁻¹ .	
thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch,		
an aliquot of the saturated solution was diluted with water (1:20) and the amount of		
dissolved halide determined by a potentio-	ESTIMATED ERROR:	
metric method using a titrimeter (type Radiometer TTT1) and, presumably, standard	Solubility ⁺ 1%	
aqueous silver nitrate. Equilibrium between solid and solution	Temperature $\stackrel{+}{-}$ 0.05 °C (author)	
Phases was considered to be attained when ^{analysis} of the solution, made at progressive	REFERENCES:	
Periods of time, gave the same value for dissolved halide. The applicability of the	1. Pistoia, G.; Pecci, G.; Scrosati, B.	
method was checked by increasing the ratio of N-methylformamide to aqueous solution (1:5).	<i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1167-1172	
The equivalence point remained the same (1).		

io n // ///		
COMPONENTS :	ORIGINAL MEASUREMENTS:	
 (1) Sodium bromide; NaBr; [7647-15-6] (2) N-methylformamide; C₂H₅NO; [123-39-7] 	Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, <u>1964</u> , 9, 416-418	
- 25		
VARIABLES:	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of sodium bromide in N-methylformamide at 25 $^{\circ}$ C 28.6 g/100 g solvent		
Within the temperature range O-35 $^{\circ}$ C the solution are given by:	bilities in g/100 g of solvent, S,	
S = -0.124 T + 66.59		
Where T is the absolute temperature.		
AUXILIARY	INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS;	
An excess of sodium bromide and 10 cm of N-methylformamide in a carefully stoppered	Anhydrous A.R. grade sodium bromide was used without_further purification after drying	
test tube were placed in a constant temper-	at 105 $^{\circ}$ C for 24 hours. N-methylformamide was prepared by either of	
ature bath for 2-4 days. The test tubes were shaken several times each day. Satur-	two methods: the addition of methylamine to formic acid (2) or by the classical	
ation was assumed when samples taken at least one day, apart gave identical concentr-	method of the addition of methylamine to	
ations. 5 cm ² samples were withdrawn with a pipette equipped with a glass wool filter	ethyl formate (3). The N-methylformamide was fractionally recrystallised until the	
over the tip, and transferred to a glass	conductance reached a value of between 10 ⁻¹ and 10 ⁻² S m ⁻¹ . It was stored in tightly	
stoppered weighing bottle. The contents of the weighing bottle were diluted to 100 cm ³	(continued)	
with water and the halide titrated by Fajan's method. To check the reliability	ESTIMATED ERROR: Solubility + 1%	
of the method, known samples of halide salts mixed with N-methylformamide in water were	Temperature $\stackrel{+}{=} 0.1 ^{\circ}C$ (author)	
analysed and the results reported as		
excellent. Berger and Dawson (1) also report successful analyses of halides in	REFERENCES :	
the presence of formamide by Fajan's method. Densities were not as accurate as the	 Berger, C.; Dawson, L.R. Anal. Chem., 1952, 24, 994-996. 	
corresponding weight data since they relied on temperature dependent pipette volumes.	<u>1777</u> 279 774-770.	
As the densities were used to calculate	(continued)	
concentrations on the molar scale the (continued)		

N-Methylformamide		105
COMPONENTS: (1) Sodium bromide; NaBr; [7647-15-6] (2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	ORIGINAL MEASUREMENTS: Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, <u>1964</u> , 9, 416-418	
METHOD/APPARATUS/PROCEDURE: (continuation) results are estimated to be accurate to within 2% on that scale, and to within 1% on the molal and mole ratio scales. The solubilities in molal units were found to be linear functions of temperature over the range O-35 °C. The constants A and B in the equation m = AT + B were determined for each salt by linear regression analysis. No information is given Concerning the standard errors of the regression coefficients. However by comparing the solubilities quoted for direct measurements at 25 °C with those derived from the linear equations, the calculated solubilities are within O.6 - 2.5% of those derived by direct measurement.		
SOURCE AND PURITY OF MATERIALS: (continuation) stoppered brown glass bottles at room temperature. The physical constants of the N-methylformamide were (literature values in parentheses): $n_D^{25} = 1.4305 [1.4300(4), 1.4310(2)]; \rho_4^{21} = 1.01 [1.008 at 15 °C(5)];$ B.Pt. = 62 °C at 6 mm of Hg [51 °C at 1 mm of Hg(5)]. In all cases, salts and solvent were stored and transferred in such a manner as to minimise the absorption of atmospheric moisture, presumably by the use of a dry box.		
 REFERENCES: (continuation) 2. Leader, G.R.; Gormley, J.F. J. Am. Chem. Soc., <u>1952</u>, 73, 5731-5733. 3. Bordwell, F.G. "Organic Chemistry", p.446, Macmillan, N.Y., 1963. 4. D'alelio, G.F.; Reid, E.E. J. Am. Chem. Soc., <u>1937</u>, 59, 109-111 5. French, C.M.; Glover, K.H. Trans. Faraday Soc., <u>1955</u>, 51, 1418-1427. 		

COMPONENTS :	EVALUATOR:
(2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife. Scotland.
	December 1978.

CRITICAL EVALUATION:

Three investigations of the solubility of NaI in N-methylformamide have been reported, all within the last fifteen years (1-3). N-methylformamide is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (4). Later, Feakins and Lawrence (5) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use.

It should be noted that in two of the current solubility studies (those by Strack et al. (1) and by Paul et al. (3)), the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent. The other major difficulty which may arise in solubility measurements in N-methylformamide, namely solvate formation, has not been detected in any of the three studies of NaI. However Strack et al. noted that the solution phase showed a brownish-yellow tinge after 24 hours at 298 K which became darker with the passage of time. Free iodine was identified, and it was not found possible to prevent its formation at the high iodide concentrations involved. The effect of free iodine on the solubility of NaI is difficult to judge, but some modification of the solvent is certain and the possibility of tri-iodide formation cannot be ruled out. As it seems probable that all the solutions were contaminated to some extent with free iodine, only tentative values for NaI solubility in N-methylformamide are given. The solubilities at 298 K suggested by Strack et al. (1) (779 g/kg of solvent) and by Paul et al. (3) (795 g/kg of solvent) are in good agreement, being within 2% of each other. The value given by Berardelli et al. (2) (575 g/kg of solvent) is considerably lower than the others and is rejected on statistical grounds. Tentative value at 298 K.

787 ⁺ 25 g/kg of solvent.

Tentative values at 308 K and 318 K

Paul et al. (3) report solubilities at 308 K and 318 K. Since the results for solubility at 298 K given by this group have been authenticated by comparison (1), their values at higher temperature have been taken to be reliable. To check the internal consistency of these results a regression analysis was undertaken where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Almost perfect linearity was obtained.

308 K

 $870 \stackrel{+}{=} 30 \text{ g/kg of solvent.}$

318 K

946 [±] 30 g/kg of solvent.

- 1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416-418.
- 2. Berardelli, M.L; Pistoia, G.; Polcaro, A.M. Ric. Sci., <u>1968</u>, 38, 814-819.
- 3. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 4. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 5. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

N-Methylformamide 10	
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Sodium iodide; NaI;[7681-82-5] N-methylformamide; C₂H₅NO;[123-39-7] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	L
t/ ^o C 25 35 45	g/100 g solvent 79.5 87.0 94.6
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	SOURCE AND PURITY OF MATERIALS: A.R. grade sodium iodide (BDH) was recrystallised and dried under vacuum at 110-125 °C. N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry nitrogen atmosphere and had the following physical constants: B.Pt. 52-53 °C/1 mm; specific conductance 3-4 x 10 ⁻⁵ S m ⁻¹ ;density 0.9986 g/ cm ³ ; refractive index 1.4312 at 25 °C. The solvent was stored in dark coloured bottles and kept in a dry box. Physical constants were rechecked before use ESTIMATED ERROR: Solubility [±] 1% Temperature [±] 0.05 °C (author) REFERENCES:

ORIGINAL MEASUREMENTS:
Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
PREPARED BY: J.S. McKechnie thylformamide at 25 ^O C
INFORMATION
<pre>SOURCE AND PURITY OF MATERIALS: Sodium iodide, reagent grade, was dried under high vacuum before use. N-methylformamide (Fluka product) was purified by storing over CaO for two days before twice distilling under reduced pressure. The conductance of the N-methyl- formamide varied from 3.0 to 6.0 x 10⁻⁴S m⁻¹.</pre> ESTIMATED ERROR: Solubility [±] 1% Temperature [±] 0.05 °C (author) REFERENCES: 1. Pistoia, G.; Pecci, G.; Scrosati, B. <i>Rio. Soi.</i> , <u>1967</u> ,37, 1167-1172

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium iodide; NaI; [7681-82-5]	Strack, G.A.; Swanda, K.S.; Bahe, L.W.
(2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	J. Chem. Eng. Data, <u>1964</u> , 9, 416-418
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium iodide in N-methylforma	mide at 25 °C
77.9 g/100 g solvent	

	. <u></u>
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: An excess of sodium iodide and 10 cm ³ of	SOURCE AND PURITY OF MATERIALS: Anhydrous A.R. grade sodium iodide was used
N-methylformamide in a carefully stoppered test tube were placed in a constant tempera- ture bath for 2-4 days. The test tubes were shaken several times each day. Satur- ation was assumed when samples taken at least	without further purification after drying at 105 ^O C for 24 hours. N-methylformamide was prepared by either of two methods: the addition of methylamine to formic acid(2) or by the classical method
one day apart gave identical concentrations. 5 cm samples were withdrawn with a pipette equipped with a glass wool filter over the tip, and transferred to a glass stoppered weighing bottle. The contents of the weighing bottle were diluted to 100 cm with water and the halide titrated by Fajan's method. To check the reliability of the	of the addition of methylamine to ethyl formate(3). The N-methylformamide was fractionally recrystallised until the conductance reached a value of between 10 ⁻¹ (continued)
	ESTIMATED ERROR:
method, known samples of halide salts mixed	Solubility ⁺ _1%
with N-methylformamide in water were analysed and the results reported as excellent. Berger and Dawson(1) also report successful	Temperature $\stackrel{+}{-}$ 0.1 $^{\circ}$ C (author)
analyses of halides in the presence of formamide by Fajan's method. Densities were not as accurate as the Corresponding weight data since they relied	REFERENCES: 1. Berger, C.; Dawson, L.R. Anal. Chem., <u>1952</u> , 24, 994-996.
on temperature dependent pipette volumes. As the densities were used to calculate (continued)	(continued)

COMPONENTS: ORIGINAL MEASUREMENTS Strack, G.A.; Swanda, K.S.; Bahe, L.W. (1) Sodium iodide; NaI; [7681-82-5] J. Chem. Eng. Data, 1964, 9, 416-418 (2) N-methylformamide; C₂H₅NO; [123-39-7] METHOD/APPARATUS/PROCEDURE: (continuation) concentrations on the molar scale the results are estimated to be accurate to within 2% on that scale, and to within 1% on the molal and mole ratio scales. SOURCE AND PURITY OF MATERIALS: (continuation) and 10^{-2} sm^{-1} . It was stored in tightly-stoppered brown glass bottles at room temperature. The physical constants of the N-methylformamide were (literature values in parentheses): $N_{D}^{25} = 1.4305 [1.4300(4), 1.4310(2)]; \rho_{d}^{21} = 1.01 [1.008 at 15 °C(5)]; B.Pt. = 62 °C at$ 6 mm of Hg [51 °C at 1 mm of Hg(5)]. In all cases, salts and solvent were stored and transferred in such a manner as to minimise the absorption of atmospheric moisture, presumably by the use of a dry box. REFERENCES: (continuation) 2. Leader, G.R.; Gormley, J.F. J. Am. Chem. Soc., 1951, 73, 5731-5733. 3. Bordwell, F.G. "Organic Chemistry", p446, Macmillan, N.Y., 1963. 4. D'alelio, G.F.; Reid, E.E. J. Am. Chem. Soc., 1937, 59, 109-111. 5. French, C.M.; Glover, K.H. Trans. Faraday Soc., 1955, 52, 1418-1427.

COMPONENTS:	EVALUATOR:
<pre>(1) Sodium thiocyanate; NaCNS; [540-72-7] (2) N-methylformamide; C₂H₅NO; [123-39-7]</pre>	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

One investigation of the solubility of NaCNS in N-methylformamide has been reported (1). This solvent is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (2). Later, Feakins and Lawrence (3) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls, and recommended baking out containers, etc., where possible, before use. In the current study, the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent.

The solubility was measured at 298 K, 308 K and 318 K. To check the internal consistency of the results, a regression analysis was undertaken where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was obtained with a correlation coefficient of 0.993. On the basis of this analysis the following solubility values are recommended.

Recommended values

<u>298 K</u>

503 [±] 10 g/kg of solvent

<u>308 k</u>

356 [±] 8 g/kg of solvent

<u>318 K</u>

288 - 6 g/kg of solvent.

- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 2. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 3. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

112. /¥-1416(11)1	lormaniae
COMPONENTS: (1) Sodium thiocyanate; NaCNS; [540-72-7] (2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	ORIGINAL MEASUREMENTS: Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES: Temperature	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	
t/°C 25 35 45	g/100 g solvent 50.3 35.6 28.8
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium thiocyanate were prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box.	SOURCE AND PURITY OF MATERIALS: A.R.grade sodium thiocyanate (BDH) was recrystallised and dried under vacuum at 110-125 °C. N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry nitrogen atmosphere and had the following physical constants: B.Pt. 52-53 °C/1 mm; specific conductance 3-4 x 10 ⁻⁵ S m ⁻¹ ; density 0.9986 g/ cm ³ ; refractive index 1.4312 at 25 °C. The solvent was stored in dark coloured bottles and kept in a dry box. Physical constants were rechecked before use. ESTIMATED ERROR:
Measurements were made in triplicate.	Solubility [±] 1% Temperature [±] 0.05 ^o C (author) REFERENCES:

COMPONENTS:	EVALUATOR:
 Potassium chloride; KC1; [7447-40-7] N-methylformamide; C₂H₅N0; [123-39-7] 	C.A. Vincent. Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland.
	December 1978.

Three investigations of the solubility of KCl in N-methylformamide have been reported, all within the last fifteen years (1-3). N-methylformamide is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (4). Later, Feakins and Lawrence (5) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use.

It should be noted that in two of the current solubility studies (those by Strack et al. (1) and by Paul et al. (3)), the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent. The other major difficulty which may arise in solubility measurements in N-methylformamide, namely solvate formation, has not been detected in any of the three studies of KC1.

The solubilities reported at 298 K by Strack et al. (1) (20.4 g/kg of solvent) and by Berardelli et al. (2) (21.4 g/kg of solvent) are in excellent agreement. The value given by Paul et al. (3) (25.4 g/kg of solvent) is relatively high, but there seems no reason, either statistical or methodological, to reject it. (It might however be noted that Paul et al. give no details of their analytical methods). The mean of all three experimental values is therefore taken as the recommended value. <u>Recommended</u> value at 298 K

-onincided value at 200 K

22.4 - 2.7 g/kg of solvent.

Values at other temperatures

Strack et al. (1), who measured solubilities over the temperature range 273-308 K, do not quote values at temperatures other than 298 K, but report a zero temperature coefficient of solubility. Paul et al. (3), on the other hand, who give values at 308 K and 318 K suggest that the solubility increases markedly with temperature. No tentative values for solubility are therefore recommended for temperatures other than 298 K.

- Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, <u>1964</u>, 9, 416-418.
- ². Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. *Ric. Sci.*, <u>1968</u>, 38, 814-819.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 4. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 5. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

14 <i>N</i> -Methylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Potassium chloride; KC1; [7447-40-7] (2) N-methylformamide; C₂H₅NO;[123-39-7]</pre>	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	I
t/°C 25 35 45	g/100 g solvent 2.54 2.64 2.96
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium chloride were prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	INFORMATION SOURCE AND PURITY OF MATERIALS: A.R. grade potassium chloride (BDH) was recrystallised and dried under vacuum at 110-125 °C. N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry nitrogen atmosphere and had the following physical constants: B.Pt. 52-53 °C/1 mm; specific conductance 3-4 x 10 ⁻⁵ S m ⁻ ;density 0.9986 g/cm ³ ; refractive index 1.4312 at 25 °C. The solvent was stored in dark coloured bottles and kept in a dry box. Physical constants were rechecked before use. ESTIMATED ERROR: Solubility ⁺ 1% Temperature ⁺ 0.05 °C (author) REFERENCES:

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N-Methylformamide		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
 Potassium chloride; KC1; [7447-40-7] N-methylformamide; C₂H₅NO; [123-39-7] 	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819	
VARIABLES: One temperature: 25 ^O C	PREPARED BY: J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of potassium chloride i	n N-methylformamide at 25 ^O C	
2.14 g/100 g solven	t.	
AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
	r Potassium chloride, reagent grade, was dried under high vacuum before use. M-methylformamide (Fluka product) was purified by storing over CaO for two days before twice distilling under reduced pressure. The conductance of the N-methyl-	
syphon provided with a terminal G-3 gooch,		

ESTIMATED ERROR:

± 1% Solubility Temperature $\stackrel{+}{=}$ 0.05 °C (author)

REFERENCES:

1. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.

dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT1) and, presumably, standard Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the

an aliquot of the saturated solution was diluted with water (1:20) and the amount of

ratio of N-methylformamide to aqueous solution (1:5). The equivalence point

aqueous silver nitrate.

remained the same (1).

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KC1; [7447-40-7]	Strack, G.A.; Swanda, K.S.; Bahe, L.W.
(2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	J. Chem. Eng. Data, <u>1964</u> , 9, 416-418
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of potassium chlorid	de in N-methylformamide at 25 ^o C
3.0% 5/100 5.5	- 1
2.04 g/100 g so	Sivent
Within the temperature range O	-35 $^{\circ}$ C the solubilities in g/100 g
of solvent, S, are given by:	
S = 0.000 T + 2.00	36
where T is the absolute tempera	11016
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: An excess of potassium chloride and 10 cm of	SOURCE AND PURITY OF MATERIALS;
N-methylformamide in a carefully stoppered	used without further purification after
test tube were placed in a constant	drying at 105 °C for 24 hours.
temperature bath for 2-4 days. The test	N-methylformamide was prepared by either
tubes were shaken several times each day. Saturation was assumed when samples taken at	of two methods: the addition of methylamine to formic acid (2) or by the classical method
least one day apart gave identical concentra-	of the addition of methylamine to ethyl
tions. 5 cm samples were withdrawn with a	formate (3). The N-methylformamide was
pipette equipped with a glass wool filter over the tip, and transferred to a glass	fractionally recrystallised until the
stoppered weighing bottle. The contents	conductance reached a value of between 10^{-1} and 10^{-2} S m ⁻¹ . It was stored in tightly-
of the weighing bottle were diluted to	(continued)
100 cm with water and the halide titrated by	ESTIMATED ERROR:
Fajan's method. To check the reliability of the method, known samples of halide salts	
mixed with N-methylformamide in water were	Solubility ± 1%
analysed and the results reported as excellent	Temperature ⁺ 0.1 ^o C (author)
Berger and Dawson (1) also report successful	
analyses of halides in the presence of formamide by Fajan's method.	REFERENCES :
Densities were not as accurate as the	 Berger, C.; Dawson, L.R. Anal. Chem., 1952, 24, 994-996.
corresponding weight data since they relied	
on temperature dependent pipette volumes. As the densities were used to calculate	(continued)
concentrations on the molar scale the results	
are estimated to be accurate to within 2% on	
(continued)	

COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Potassium chloride; KC1; [7447-40-7]	Strack, G.A.; Swanda, K.S.; Bahe, L.W.	
(2) N-methylformamide; $C_{2}H_{c}NO;$ [123-39-7]	J. Chem. Eng. Data, <u>1964</u> , 9, 416-418	
METHOD/APPARATUS PROCEDURE. (continuation)		
that scale, and to within 1% on the molal and	mole ratio scales.	
The solubilities in molal units were found to be linear functions of temperature over the range 0-35 °C. The constants A and B in the equation m = At + B were determined for each salt by linear regression analysis. No information is given concerning the standard errors of the regression coefficients. However by comparing the solubilities quoted for direct measurements at 25 °C with those derived from the		
linear equations, the calculated solubilities derived by direct measurement.	are within 0.6 to 2.5% of those	
SOURCE AND PURITY OF MATERIALS (continuatio	n)	
stoppered brown glass bottles at room tempera	ture.	
The physical constants of the N-methylform 2^{5} 1 (205 [1 (200(1) 1 (210(2))] 21	amide were (literature values in parentheses);	
$n_D^{25} = 1.4305 [1.4300(4), 1.4310(2)]; \rho_4^{21} = 6 \text{ mm. of Hg} [51 ^{\circ}C \text{ at } 1 \text{ mm. of Hg}(5)].$	1.01 [1.008 at 15 $C(5)$]; B.Pt. = 62 C at	
	d and transformed in such a menner as to	
In all cases, salts and solvent were stored and transferred in such a manner as to minimise the absorption of atmospheric moisture, presumably by the use of a dry box.		
REFERENCES (continuation)		
2. Leader, G.R.; Gormley, J.F. J. Am. Chem.	Soc. 1051 77 5731-5733	
3. Bordwell, F.G.: "Organic Chemistry", p44		
4. D'alelio, G.F.; Reid, E.E. J. Am. Chem.		
5. French, C.M.; Glover, K.H. Trans. Faraday Soc., <u>1955</u> , 51, 1418-1427.		

COMPONENTS: (1) Potassium bromide; (2) N-methylformamide;	• • •	EVALUATOR: C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland. December 1978.

Three investigations of the solubility of KBr in N-methylformamide have been reported, all within the last fifteen years (1-3). N-methylformamide is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (4). Later, Feakins and Lawrence (5) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use.

It should be noted that in two of the current solubility studies (those by Strack et al. (1) and by Paul et al. (3)), the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent. The other major difficulty which may arise in solubility measurements in N-methylformamide, namely solvate formation, has not been detected in any of the three studies of KBr.

The solubilities reported at 298 K by all three groups show excellent agreement, having a mean value of 101.7 g/kg of solvent, and a standard deviation of 1.0 g/kg of solvent, or 1%. This mean is taken as the recommended value.

Recommended value at 298 K

101.7 ⁺ 1.0 g/kg of solvent.

Values at other temperatures

Strack et al. (1), who measured solubilities over the temperature range 273-308 K, do not quote values at temperatures other than 298 K, but report a linear relationship between molal solubility and temperature, with a decreasing solubility with increasing temperature. Paul et al. (3), on the other hand, who give values at 308 K and 318 K, show a slowly increasing solubility with increasing temperature. It is likely that the temperature coefficient of solubility is small, but because of these discrepancies no tentative values can be given.

- 1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416-418.
- 2. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819.
- 3. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Naruula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 4. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 5. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Potassium bromide; KBr; [7758-02-3] (2) N-methylformamide; C₂H₅NO;[123-39-7]</pre>	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
.0_	
t/°c	g/100 g solvent
25	10.3
35	10.4
45	11.1
,	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium bromide were	SOURCE AND PURITY OF MATERIALS:
prepared by adding excess of the powdered	recrystallised and dried under vacuum at
salt to N-methylformamide (15-20 g) in Pyrex	110-125 °C.
tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath	N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry
prior to final thermostatting. The	nitrogen atmosphere and had the following
attainment of the equilibrium was checked by intermittent analyses. The solutions were	physical constants: B.Pt. 52-53 ^o C/1 mm; specific conductance 3-4 x 10 ⁻⁵ S m ⁻¹ ;density
then filtered under nitrogen and analysed	0.9986 g/cm ³ ; refractive index 1.4312 at
for halide. No details of analytical methods were given. Transference of material, as	25 °C. The solvent was stored in dark
far as possible, was carried out in a	coloured bottles and kept in a dry box. Physical constants were rechecked before use.
nitrogen filled dry box. Measurements were	ESTIMATED ERROR:
made in triplicate.	Solubility ± 1%
	Temperature ⁺ 0.05 ^o C (author)
Ì · · · · · · · · · · · · · · · · · · ·	REFERENCES:

20 <i>N</i> -Methylformamide	
COMPONENTS :	ORIGINAL MEASUREMENTS:
 Potassium bromide; KBr; [7758-02-3] N-methylformamide; C₂H₅NO; [123-39-7] 	Berardelli, M.L.; Pistoia, G.; Polcaro,A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	L
Solubility of potassium bromide in N- 10.2 g /100 g solvent.	-methylformamide at 25 ^o C
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated potassium bromide solutions were prepared by dissolv ing excess of the halide in about 20-25 cm of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentio- metric method using a titrimeter (type Radiometer TTTl) and, presumably, standard aqueous silver nitrate. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of N-methylformamide to aqueous solution	Potassium bromide, reagent grade, was dried under high vacuum before use. N-methylformamide (Fluka product) was purified by storing over CaO for two days before twice distilling under reduced pressure. The conductance of the N-methyl_1 formamide varied from 3.0 to 6.0 x 10 ⁻⁴ S m ⁻¹ ESTIMATED ERROR: Solubility [±] 1% Temperature [±] 0.05 °C (author) REFERENCES: 1. Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1167-1172

N-Methylforma	mide
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N-Methylforr	namide 12
COMPONENTS: (1) Potassium bromide; KBr; [7758-02-3] (2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	ORIGINAL MEASUREMENTS: Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416-418
METHOD/APPARATUS/PROCEDURE: (continuation) As the densities were used to calculate concent are estimated to be accurate to within 2% on the and mole ratio scales. The solubilities in molal units were found over the range O-35 °C. The constants A and B determined for each salt by linear regression a concerning the standard errors of the regression the solubilities quoted for direct measurements linear equations, the calculated solubilities a derived by direct measurement.	That scale, and to within 1% on the molal to be linear functions of temperature 3 in the equation $m = AT + B$ were unalysis. No information is given on coefficients. However by comparing at 25 °C with those derived from the are within 0.6 to 2.5% of those
SOURCE AND PURITY OF MATERIALS: (continuation in tightly-stoppered brown glass bottles at root ${}_{25}$ The physical constants of the N-methylformam $n_D^{}$ = 1.4305 [1.4300(4), 1.4310(2)]; ρ^{21}_{4} = 1.66 mm. of Hg [51 °C at 1 mm. of Hg (5)]. In all cases, salts and solvent were stored minimise the absorption of atmospheric moisture	om temperature. iide were (literature values in parentheses): Ol [1.008 at 15 ^O C(5)]; B.Pt. = 62 ^O C at and transferred in such a manner as to
REFERENCES (continuation) 2. Leader, G.R.; Gormley, J.F. J. Am. Chem. 3. Bordwell, F.G.; "Organic Chemistry", p446, 4. D'alelio, G.F.; Reid, E.E. J. Am. Chem. 5. French, C.M.; Glover, K.H. Trans. Faraday	Macmillan, N.Y., 1963. Soc., <u>1937</u> , 59, 109-111.

•		
COMPONENTS :	ORIGINAL MEASUREMENTS:	
1) Potassium bromide; KBr; [7758-02-3]	Strack, G.A.; Swanda, K.S.; Bahe, L.W.	
(2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	J. Chem. Eng. Data, <u>1964</u> , 9, 416-418	
	v. onom. Dry. Dava, <u>1904,</u> v, 410 410	
VARIABLES:	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of potassium bromide in N-methylfc		
(rmanide at 25 C	
9.95 g/100 g solvent Within the temperature range O-35 $^{\circ}$ C the solubilities in g/100 g of solvent, S,		
are given by:	of additioned in grido g of solvent, S,	
S = -0.012 T + 13.79		
where T is the absolute temperature	•	
AUXILIARY	INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
An excess of potassium bromide and 10 cm ³ of	Anhydrous A.R. grade potassium bromide was used without further purification after	
N-methylformamide in a carefully stoppered test tube were placed in a constant temper-	drying at 105 ^O C for 24 hours.	
ature bath for 2-4 days. The test tubes	N-methylformamide was prepared by either of two methods: the addition of	
were shaken several times each day. Satur- ation was assumed when samples taken at least	methylamine to formic acid (2) or by the	
one day apart gave identical concentrations.	classical method of the addition of methyl- amine to ethyl formate (3). The N-methyl-	
5 cm ³ samples were withdrawn with a pipette equipped with a glass wool filter over the	formamide was fractionally recrystallised	
tip, and transferred to a glass stoppered	until the conductance reached a value of between 10^{-1} and 10^{-2} S m ⁻¹ . It was stored	
weighing bottle. The contents of the weighing bottle were diluted to 100 cm ³ with	(continued)	
water and the halide titrated by Fajan's	ESTIMATED ERROR:	
method. To check the reliability of the method, known samples of halide salts mixed		
with N-methylformamide in water were	Solubility $+$ 1%	
analysed and the results reported as excellent. Berger and Dawson (1) also	Temperature ⁺ 0.1 ^o C (author)	
report successful analyses of halides in the	REFERENCES:	
presence of formamide by Fajan's method. Densities were not as accurate as the	1. Berger, C.; Dawson, L.R. Anal. Chem.,	
corresponding weight data since they relied	<u>1952</u> , 24, 994-996.	
on temperature dependent pipette volumes.	(continued)	
(continued)	(continued)	

COMPONENTS:	EVALUATOR:
<pre>(1) Potassium iodide; KI; [7681-11-0] (2) N-methylformamide; C₂H₅NO; [123-39-7]</pre>	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

Three investigations of the solubility of KI in N-methylformamide have been reported, all within the last fifteen years (1-3). N-methylformamide is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (4). Later, Feakins and Lawrence (5) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls, and recommended baking out containers, etc., where possible before use.

It should be noted that in two of the current solubility studies (those by Strack et al. (1) and by Paul et al. (3)), the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent. The other major difficulty which may arise in solubility measurements in N-methylformamide, namely solvate formation, has not been detected in any of the three studies of KI. However Strack et al. noted that the solution phases showed a brownish-yellow tinge after 24 hours at 298 K which became darker with the passage of time. Free iodine was identified, and it was not found possible to prevent its formation at the high iodide concentrations involved. The effect of free iodine on the solubility of KI is difficult to judge, but some modification of the solvent is certain and the possibility of tri-iodide formation cannot be ruled out. As it seems probable that all the solutions were contaminated to some extent with free iodine, only tentative values for KI solubility in N-methylformamide are given. The solubilities at 298 K suggested by Strack et al. (1) (486 g/kg of solvent) and by Berardelli et al. (2) (496 g/kg of solvent) are in good agreement, being within 1% of each other. The value given by Paul et al. (3) (442.1 g/kg of solvent) is considerably lower than the others and is rejected on statistical grounds. Tentative value at 298 K.

491 ⁺ 15 g/kg of solvent.

- 1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416-418.
- 2. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 4. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- ⁵. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), <u>1966</u>, 212-219.

4 N-Methylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Potassium iodide; KI; [7681-11-0] (2) N-methylformamide; C₂H₅NO;[123-39-7]</pre>	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C 25 35 45	g/100 g solvent 44.2 45.9 46.8
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium iodide were prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	SOURCE AND PURITY OF MATERIALS: A.R. grade potassium iodide (BDH) was recrystallised and dried under vacuum at 110-125 °C. N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry nitrogen atmosphere and had the following physical constants: B.Pt. 52-53 °C/1 mm; specific conductance 3-4 x 10 ⁻⁵ S m ⁻¹ ; density 0.9986 g/cm ³ ; refractive index 1.4312 at 25 °C. The solvent was stored in dark coloured bottles and kept in a dry box. Physical constants were rechecked before use. ESTIMATED ERROR: Solubility [±] 1% Temperature [±] 0.05 °C (author) REFERENCES:

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0]	Berardelli, M.L.; Pistoia, G.; Polcaro,A.M.
(2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	<i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie
	· · · · · · · · · · · · · · · · · · ·

EXPERIMENTAL VALUES:

Solubility of potassium iodide in N-methylformamide at 25 °C

49.6 g/100 g solvent

AUXILIARY	INFORMATION

METHOD/APPARATUS/PROCEDURE: Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated potassium iodide solutions were prepared by dissolv-ing excess of the halide in about 20-25 cm of solvent contained in a 50 cm Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT1) and, presumably, standard aqueous silver nitrate.

Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of N-methylformamide to aqueous solution (1:5). The equivalence point remained the same(1).

SOURCE AND PURITY OF MATERIALS: Potassium iodide, reagent grade, was dried under high vacuum before use. N-methylformamide (Fluka product) was purified by storing over CaO for two days before twice distilling under reduced pressure. The conductance of the N-methylformamide varied from 3.0 to 6.0 x 10⁻⁴ S m⁻¹.

ESTIMATED ERROR:

Solubility ⁺/₋ 1% Temperature ⁺/₋ 0.05 ^oC (author)

REFERENCES:

 Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., <u>1967</u>, 37, 1167-1172

COMPONENTS:	ORIGINAL MEASUREMENTS:
•	Strack, G.A.; Swanda, K.S.; Bahe, L.W. <i>J.Chem. Eng. Data</i> , <u>1964</u> , <i>9</i> , 416-418
VARIABLES: One temperature: 25 ^O C EXPERIMENTAL VALUES:	PREPARED BY: J.S. McKechnie
Solubility of potassium iodide in N-methylformamide at 25 °C 48.6 g/100 g solvent	
METHOD/APPARATUS/PROCEDURE: An excess of potassium iodide and lo cm ³ of N-methylformamide in a carefully stoppered test tube were placed in a constant temper- ature bath for 2-4 days. The test tubes were shaken several times each day. Satur- ation was assumed when samples taken at least one day apart gave identical concentr- ations. 5 cm ³ samples were withdrawn with a pipette equipped with a glass wool filter over the tip, and transferred to a glass stoppered weighing bottle. The contents of the weighing bottle were diluted to loo cm ³ with water and the halide titrated by Fajan's method. To check the reliabil- ity of the method, known samples of halide salts mixed with N-methylformamide in water were analysed and the results reported as excellent. Berger and Dawson (1) also report successful analyses of halides in the presence of formamide by Fajan's method. Densities were not as accurate as the corresponding weight data since they relied on temperature dependent pipette volumes.	to formic acid (2) or by the classical
(continued)	

ORIGINAL MEASUREMENTS:

Strack, G.A.; Swanda, K.S.; Bahe, L.W.

J. Chem. Eng. Data, 1964, 9, 416-418

COMPONENTS:	
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Potassium iodide; KI; [7681-11-0]
 N-methylformamide; C₂H₅NO; [23-39-7]

METHOD/APPARATUS/PROCEDURE: (continuation)

As the densities were used to calculate concentrations on the molar scale the results Were estimated to be accurate to within 2% on that scale, and to within 1% on the molal and mole ratio scales.

SOURCE AND PURITY OF MATERIALS: (continuation)

stoppered brown glass bottles at room temperature. The physical constants of the N-methylformamide were (literature values in parentheses): $n_D^2 = 1.4305 [1.4300(4), 1.4310(2)]; \rho_4^2 = 1.01 [1.008 at 15 C(5)];$ B.Pt. = 62 °C at 6 mm. of Hg [51 °C at 1 mm. of Hg (5)].

In all cases, salts and solvent were stored and transferred in such a manner as to minimise the absorption of atmospheric moisture, presumably by the use of a dry box.

REFERENCES: (continuation)

2. Leader, G.R.; Gormley, J.F. J. Am. Chem. Soc., 1951, 73, 5731-5733.

3. Bordwell, F.G.; "Organic Chemistry", p446, Macmillan, N.Y., 1963.

4. D'alelio, G.F.; Reid, E.E. J. Am. Chem. Soc., <u>1937</u>, 59, 109-111

5. French, C.M.; Glover, K.H. Trans. Faraday Soc., 1955, 51, 1418-1427.

COMPONENTS: EV	EVALUATOR:
(1) Potassium thiocyanate; KCNS; [333-20-0] D (2) N-methylformamide; C ₂ H ₅ NO; [123-39-7] U S S	C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of KCNS in N-methylformamide has been reported (1). This solvent is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (2). Later, Feakins and Lawrence (3) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers etc., where possible, before use. In the current study, the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent.

The solubility was measured at 298 K, 308 K and 318 K. To check the internal consistency of the results, a regression analysis was undertaken where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was obtained with a correlation coefficient of 0.997. On the basis of this analysis the following solubility values are recommended.

Recommended values

298 K

809[±] 8 g/kg of solvent

308 K

773 [±] 8 g/kg of solvent 318 K

731 $\stackrel{+}{-}$ 7 g/kg of solvent.

- 1. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 2. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 3. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

/v-iviethylformanide 129		
COMPONENTS :	ORIGINAL MEASUREMENTS:	
(2) N-methylformamide; C ₂ H ₅ NO;[123-39-7]	Paul, R.C.; Singla, J.P.; Lamba M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026	
VARIABLES: Temperature	PREPARED BY: J.S. McKechnie	
EXPERIMENTAL VALUES:		
t/ ^o C 25 35 45	g/100 g solvent 80.9 77.3 73.1	
AUXILIARY	INFORMATION	
AUXILIARY METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium thiocyanate were prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	INFORMATION SOURCE AND PURITY OF MATERIALS: A.R. grade potassium thiocyanate (BDH) was recrystallised and dried under vacuum at 110-125 °C. N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry nitrogen atmosphere and had the following physical constants: B.Pt. 52-53 °C/1 mm; specific conductance 3-4 x 10 ⁻⁵ S m ⁻¹ ; density 0.9986 g/cm ³ ; refractive index 1.4312 at 25 °C. The solvent was stored in dark coloured bottles and kept in a dry box. Physical constants were rechecked before use. ESTIMATED ERROR: Solubility [±] 1% Temperature [±] 0.05 °C (author) REFERENCES:	
L		

COMPONENTS :	EVALUATOR:
<pre>(1) Ammonium chloride; NH₄C1; [12125-02-9] (2) N-methylformamide; C₂H₅NO; [123-39-7]</pre>	C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Two investigations of the solubility of NH₄Cl in N-methylformamide have been reported (1,2). This solvent is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (3). Later, Feakins and Lawrence (4) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use. In both of the current studies, the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent. No suggestion of solvate formation is made by either group of workers.

The solubility was measured at 298 K, 308 K and 318 K by Paul et al. (2). The internal consistency of their results was good, as shown by a regression analysis taking the logarithm of the solubility as a linear function of the reciprocal of the absolute temperature. However agreement between the two sets of results at 298K is rather poor. Tentatively, one might suggest the mean value at 298 K as representing a reasonable estimate of the solubility.

Tentative value at 298 K

58 - 8 g/kg of solvent.

- 1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, <u>1964</u>, 9, 416-418.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 3. French, C.M.; Glover, K.H. Trans. Farad. Soc., <u>1955</u>, 51, 1418-1427.
- 4. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

COMPONENTS :	ORIGINAL MEASUREMENTS:
 (1) Ammonium chloride; NH₄Cl; [12125-02-9] (2) N-methylformamide; C₂H₅NO; [123-39-7] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	6.59
35	5.57
45	3.81
· · · · · · · · · · · · · · · · · · ·	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of ammonium chloride were	A.R. grade ammonium chloride (BDH) was
prepared by adding excess of the powdered	recrystallised and dried under vacuum at
salt to N-methylformamide (15-20 g) in Pyrex	110-125 ^o C. N-methylformamide was purified by fraction-
tubes (15 x 2.5 cm) and preheating to 10 $^{\circ}$ C above the required temperature of the bath	ation of the solvent over sodamide in a dry
Prior to final thermostatting. The	nitrogen atmosphere and had the following
attainment of the equilibrium was checked by	physical constants: B.Pt. 52-53 °C/1 mm;
intermittent analyses. The solutions were	specific conductance $3-4 \times 10^{-5}$ S m ⁻¹ ; density 0,9986 g/cm ³ ; refractive index 1.4312 at
then filtered under nitrogen and analysed for halide. No details of analytical methods	25 ^o C. The solvent was stored in dark
were given. Transference of material, as	coloured bottles and kept in a dry box.
far as possible, was carried out in a	Physical constants were rechecked before use.
nitrogen filled dry box. Measurements were made in triplicate.	ESTIMATED ERROR:
	Solubility - 1%
	Temperature ⁺ 0.05 ^o C (author)
	REFERENCES :

COMPONENTS:	ORIGINAL MEASUREMENTS:	
<pre>(1) Ammonium chloride; NH₄Cl; [12125-02-9] (2) N-methylformamide; C₂H₅NO; [123-39-7]</pre>	Strack, G.A.; Swanda, K.S.; Bahe, L.W. <i>J. Chem. Eng. Data</i> , <u>1964</u> , 9, 416-418	
VARIABLES:	PREPARED BY:	
	J.S. McKechnie	
Temperature	U.S. MCRECIMITE	
EXPERIMENTAL VALUES:		
Solubility of ammonium chloride in N-methylformamide at 25 ^O C 5.03 g/lOO g solvent		
Within the temperature range O-35 $^{\circ}$ C the solubilities in g/lOO g of solvent, S, are given by:		
S = 0.020 T -0.977		
where T is the absolute temperature.		
AUXILIARY	INFORMATION	
METHOD/APPARATUS/PROCEDURE: 3	SOURCE AND PURITY OF MATERIALS;	
An excess of ammonium chloride and 10 cm of N-methylformamide in a carefully stoppered	Anhydrous A.R. grade ammonium chloride was used without further purification after	
test tube were placed in a constant temper-	drying at 105 °C for 24 hours.	
ature bath for 2-4 days. The test tubes were shaken several times each day. Satur-	N-methylformamide was prepared by either of two methods: the addition of methylamine	
ation was assumed when samples taken at least one day apart gave identical concentr-	to formic acid (2) or by the classical method of the addition of methylamine to ethyl	
ations. 5 cm ³ samples were withdrawn with a pipette equipped with a glass wool filter	formate (3). The N-methylformamide was	
over the tip, and transferred to a glass	fractionally recrystallised until the _1 conductance reached a value of between 10 1	
stoppered weighing bottle. The contents of the weighing bottle were diluted to 100 cm ³	and 10 Sm ⁻ . It was stored in tightly- (continued)	
with water and the halide titrated by Fajan's method. To check the reliability of the	ESTIMATED ERROR:	
method, known samples of halide salts mixed	Solubility - 1%	
with N-methylformamide in water were analysed and the results reported as	Temperature $\stackrel{+}{=}$ 0.1 $^{\circ}$ C (author)	
excellent. Berger and Dawson(1) also report successful analyses of halides in the	REFERENCES :	
presence of formamide by Fajan's method. Densities were not as accurate as the corresponding weight data since they relied	 Berger, C.; Dawson, L.R. Anal. Chem., <u>1952</u>, 24, 994-996. 	
on temperature dependent pipette volumes. As the densities were used to calculate		
concentrations on the molar scale the (continued)	(continued)	
(continued)	1	

COMPONENTS:	ORIGINAL MEASUREMENTS:
	Strack, G.A.; Swanda, K.S.; Bahe, L.W.
(1) Ammonium chloride; NH ₄ Cl; [12125-02-9]	J. Chem. Eng. Data, <u>1964</u> , 9, 416-418
(2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	

METHOD/APPARATUS/PROCEDURE: (continuation)

results are estimated to be accurate to within 2% on that scale, and to within 1% on the molal and mole ratio scales.

The solubilities in molal units were found to be linear functions of temperature over the range O-35 $^{\circ}$ C. The constants A and B in the equation m = AT + B were determined for each salt by linear regression analysis. No information is given concerning the standard errors of the regression coefficients. However by comparing the solubilities quoted for direct measurements at 25 $^{\circ}$ C with those derived from the linear equations, the calculated solubilities are within O.6 - 2.5% of those derived by direct measurement.

SOURCE AND PURITY OF MATERIALS: (continuation)

stoppered brown glass bottles at room temperature.

The physical constants of the N-methylformamide were (literature values in parentheses) $n_D^{25} = 1.4305 [1.4300(4), 1.4310(2)]; \rho_4^{21} = 1.01 [1.008 at 15 °C(5)]; B.Pt. = 62 °C at$ 6 mm of Hg [51 °C at 1 mm of Hg(5)].

In all cases, salts and solvent were stored and transferred in such a manner as to minimise the absorption of atmospheric moisture, presumably by the use of a dry box.

REFERENCES

Leader, G.R.; Gormley, J.F. J. Am. Chem. Soc., <u>1951</u>, 73, 5731-5733.

3. Bordwell, F.G. "Organic Chemistry", p446, Macmillan, N.Y., 1963.

4. D'alelio, G.F.; Reid, E.E. J. Am. Chem. Soc., 1937, 59, 109-111.

5. French, C.M.; Glover, K.H. Trans. Faraday Soc., 1955, 51, 1418-1427.

COMPONENTS :	EVALUATOR:
<pre>(1) Ammonium bromide; NH₄Br; [12124-97-9] (2) N-methylformamide; C₂H₅NO; [123-39-7]</pre>	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Two investigations of the solubility of NH₄Br in N-methylformamide have been reported (1,2). This solvent is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (3). Later, Feakins and Lawrence (4) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use. In both of the current studies, the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent. No suggestion of solvate formation is made by either group of workers.

The solubility was measured at 298 K, 308 K and 318 K by Paul et al. (2). The internal consistency of their results was good, as shown by a regression analysis taking the logarithm of the solubility as a linear function of the reciprocal of the absolute temperature. However agreement between the two sets of results at 298 K is rather poor.

The mean value of 281 g/kg of solvent would require an error bar of $\frac{1}{2}$ 44 g/kg or $\frac{1}{2}$ 15% to encompass the two experimental values. The only reasonable conclusion that may be drawn from these results is that the minimum solubility of NH₄Br at 298 K is likely to be \geq 237 g/kg of solvent.

References

1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416-418.

 Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.

3. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.

4. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

N-Methylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Ammonium bromide; NH4Br;[12124-97-9] (2) N-methylformamide; C2H5N0;[123-39-7]</pre>	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026.
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	32.6
35	32.9
45	33.3
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium bromide were Prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	110-125 °C. N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry nitrogen atmosphere and had the following physical constants: B.Pt. 52-53 °C/1 mm; specific conductance 3-4 x 10 ⁻ S m ⁻¹ ; density 0.9986 g/cm ³ ; refractive index 1.4312 at 25 °C. The solvent was stored in dark coloured bottles and kept in a dry box. Physical constants were rechecked before use.

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N-Methylformamide

W-Wettytotmanide		
COMPONENTS :	ORIGINAL MEASUREMENTS:	
<pre>(1) Ammonium bromide; NH₄Br; [12124-97-9] (2) N-methylformamide; C₂H₅NO; [123-39-7]</pre>	ſ	
VARIABLES:	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:	2	
Solubility of ammonium bromide in N-methylf	ormamide at 25 °C	
23.7 g/100 g solvent		
Within the temperature range O-35 $^{\circ}$ C the solubilities in g/100 g of solvent, S, are given by:		
S = 0.000 T + 23.99		
where T is the absolute temperate	ire.	
1		
1		
Mar		
**************************************	INFORMATION	
METHOD/APPARATUS/PROCEDURE: An excess of ammonium bromide and 10 cm ³ of	SOURCE AND PURITY OF MATERIALS; Anhydrous A.R. grade ammonium bromide was	
N-methylformamide in a carefully stoppered	used without further purification after	
test tube were placed in a constant temper- ature bath for 2-4 days. The test tubes	drying at 105 ^o C for 24 hours. N-methylformamide was prepared by either	
were shaken several times each day. Satur-	of two methods: the addition of methylamine to formic acid(2) or by the classical method	
ation was assumed when samples taken at least one day apart gave identical concentr-	of the addition of methylamine to ethyl	
ations. 5 cm samples were withdrawn with a pipette equipped with a glass wool filter	formate(3). The N-methylformamide was fractionally recrystallised until the	
over the tip, and transferred to a glass	conductance reached a value of between 10 ⁻¹	
stoppered weighing bottle. The contents of the weighing bottle were diluted to 100 cm^3	(continued)	
with water and the halide titrated by	ESTIMATED ERROR:	
Fajan's method. To check the reliability of the method, known samples of halide salts	Solubility - 1%	
mixed with N-methylformamide in water were analysed and the results reported as	Temperature ⁺ 0.1 ^o C (author)	
excellent. Berger and Dawson (1) also	DEFEDENCUS	
report successful analyses of halides in the presence of formamide by Fajan's method.	REFERENCES:	
Densities were not as accurate as the corresponding weight data since they relied	1. Berger, C.; Dawson, L.R. Anal. Chem., <u>1952</u> , 24, 994-996.	
on temperature dependent pipette volumes.		
to the deposition years used to coloulate		
As the densities were used to calculate (continued)	(continued)	

N-Methylformamide 13	
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Ammonium bromide;NH4Br; [12124-97-9] N-methylformamide; C2H5NO; [123-39-7] 	Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, <u>1964</u> , 9, 416-418
METHOD/APPARATUS/PROCEDURE: (continuati	
Concentrations on the molar scale the result 2% on that scale, and to within 1% on the mo The solubilities in molal units were fou over the range O-35 °C. The constants A an	lal and mole ratio scales. Ind to be linear functions of temperature
determined for each salt by linear regression analysis. No information is given concerning the standard errors of the regression coefficients. However by comparing the solubilities quoted for direct measurements at 25 °C with those derived from the	

by direct measurement.

SOURCE AND PURITY OF MATERIALS: (continuation)

and 10^{-2} sm^{-1} . It was stored in tightly-stoppered brown glass bottles at room temperature.

linear equations, the calculated solubilities are within 0.6 - 2.5% of those derived

The physical constants of the N-methylformamide were (literature values in parentheses): $n_D^{25} = 1.4305 [1.4300(4), 1.4310(2)]; \rho_4^{21} = 1.01 [1.008 at 15 °C(5)]; B.Pt. = 62 °C at$ $6 \text{ mm of Hg} [51 \circ C \text{ at } 1 \text{ mm. of Hg} (5)].$

In all cases, salts and solvent were stored and transferred in such a manner as to minimise the absorption of atmospheric moisture, presumably by the use of a dry box.

REFERENCES: (continuation)

2. Leader, G.R.; Gormley, J.F. J. Am. Chem. Soc., 1951, 73, 5731-5733

3. Bordwell, F.G. "Organic Chemistry", p446, Macmillan, N.Y., 1963.

4. D'alelio, G.F.; Reid, E.E. J. Am. Chem. Soc., 1937, 59, 109-111.

5. French, C.M.; Glover, K.H. Trans. Faraday Soc., 1955, 51, 1418-1427

COMPONENTS :	EVALUATOR:
(1) Ammonium iodide; NH ₄ I; [12027-06-4] (2) N~methylformamide; C ₂ H ₅ NO; [123-39~7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife. Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of NH_4I in N-methylformamide has been reported (1). This solvent is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (2). Later, Feakins and Lawrence (3) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use. In the current study, the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent.

The solubility was measured at 298 K, 308 K and 318 K. To check the internal consistency of the results, a regression analysis was undertaken where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was obtained with a correlation coefficient of 0.999. However in a parallel investigation of NaI and KI, Strack et al.(4) pointed out that iodide ion in high concentration in this solvent was liable to form free iodine in solution No reference to this penomenon is made by Paul et al., but because of the possibility of iodine contamination, the following values should be taken as tentative.

Tentative values

298 K 380 [±] 12 g/kg of solvent 308 K 585 [±] 18 g/kg of solvent. <u>318 K</u> 821 [±] 25 g/kg of solvent.

References

 Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.

- 2. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 3. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.
- 4. Strack, G.A.; Swanda, K.S.; Bahe, L.W. J. Chem. Eng. Data, 1964, 9, 416-418.

N-Methylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Ammonium iodide; NH₄I; [12027-06-4] (2) N-methylformamide; C₂H₅N0; [123-39-7]</pre>	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , <i>11</i> , 1024–1026
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ [°] C	g/100 g solvent
25	38.0
35	58.5
45	82.1
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of ammonium iodide were prepared by adding excess of the powdered salt to N-methylformamide $(15-20 \text{ g})$ in Pyrex tubes $(15 \times 2.5 \text{ cm})$ and preheating to $10 ^{\circ}\text{C}$ above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	Physical constants were rechecked before use. ESTIMATED ERROR:
	Solubility ⁺ 1% Temperature ⁺ 0.05 [°] C (author)
	REFERENCES :

COMPONENTS:	EVALUATOR:
(1) Ammonium thiocyanate; NH ₄ CNS; [1762-95-4] (2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of NH_4CNS in N-methylformamide has been reported (1). This solvent is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (2). Later, Feakins and Lawrence (3) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use. In the current study, the solvent conductance was 5×10^{-5} S m⁻¹, which is as low as that reported in any investigation of this solvent.

The solubility was measured at 298 K, 308 K and 318 K. To check the internal consistency of the results, a regression analysis was undertaken where the logarithm of the solubility was assumed to be a linear function of the reciprocal of the absolute temperature. Excellent linearity was obtained with a correlation coefficient of 0.963. On the basis of this analysis the following solubility values are recommended.

Recommended values

298 К

744 ⁺ 15 g/kg of solvent <u>308 K</u> 717 ⁺ 14 g/kg of solvent

<u>318 K</u>

652 [±] 13 g/kg of solvent.

- 1. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 2. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 3. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

74-1416(1191)	formamide
COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Ammonium thiocyanate; NH₄CNS; [1762-95-4] (2) N-methylformamide; C₂H₅NO; [123-39-7] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026
VARIABLES: Temperature	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	I
t/ [°] C	g/100 g solvent
25	74.4
35	71.7
	65.2

AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to N-methylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	SOURCE AND PURITY OF MATERIALS: A.R. grade ammonium thiocyanate (BDH) was recrystallised and dried under vacuum at 110-125 °C. N-methylformamide was purified by fraction- ation of the solvent over sodamide in a dry nitrogen atmosphere and had the following physical constants: B.Pt. 52-53 °C/1 mm; specific conductance 3-4 x 10 ⁻⁵ S m ⁻¹ ; density 0.9986 g/cm ³ ; refractive index 1.4312 at 25 °C. The solvent was stored in dark coloured bottles and kept in a dry box. Physical constants were rechecked before use. ESTIMATED ERROR: Solubility ⁺ 1% Temperature ⁺ 0.05 °C (author) REFERENCES:	

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<pre>(1) Magnesium chloride; MgCl₂; [7786-30-3] (2) N-methylformamide; C₂H₅NO; [123-39-7] US s</pre>	EVALUATOR: C.A. Vincent, Department of Chemistry, University of St. Andrewş, St. Andrews, Fife, Scotland. December 1978.
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CRITICAL EVALUATION:

One investigation of the solubility of MgCl₂ in N-methylformamide has been reported (1). This solvent is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (2). Later, Feakins and Lawrence (3) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use. In the current study, the solvent conductance had a mean value of 5×10^{-4} S m⁻¹, which is an order of magnitude greater than that of solvent used by other workers, studying electrolyte solubility. An additional problem is that the investigators did not follow the recommended method for preparing anhydrous MgCl₂ (by passing dry HCl over the hydrated double salt, MgCl₂. NH₄Cl), and so the purity of the solute is not known. The reported value of the solubility at 298 K is therefore proposed only as a tentative value.

Tentative value at 298 K

 $88 \stackrel{+}{=} 2 \text{ g/kg of solvent.}$

References

1. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819.

2. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.

3. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

N-Methylformamide	
COMPONENTS :	ORIGINAL MEASUREMENTS:
 Magnesium chloride; MgCl₂; [7786-30-3] N-methylformamide; C₂H₅NO; [123-39-7] 	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
111 N	
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie
EXPERIMENTAL VALUES: Solubility of magnesium chloride in 8.80 g/100 g solvent	n N-methylformamide at 25 ^o C

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated magnesium chloride solutions were prepared by dissolving excess of the halide in about 20-25 cm³ of solvent contained in a 50 cm³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT1) and, presumably, standard ^{aqueous} silver nitrate.

Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of N-methylformamide to aqueous solution (1:5). The equivalence point remained the same (1).

SOURCE AND PURITY OF MATERIALS:

Magnesium chloride, reagent grade, was dried under high vacuum before use. N-methylformamide (Fluka product) was purified by storing over CaO for two days before twice distilling under reduced pressure. The conductance of the N-methyl formamide varied from 3.0 to 6.0 x 10⁻⁴S m⁻¹.

ESTIMATED ERROR:

Solubility [±] 1% Temperature [±] 0.05 ^oC (author)

REFERENCES:

 Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, <u>1967</u>, 37, 1167-1172

COMPONENTS:	EVALUATOR:
<pre>(1) Calcium chloride; CaCl₂; [10043-52-4] (2) N-methylformamide; C₂H₅NO;[123-39-7]</pre>	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION: One investigation of the solubility of $CaCl_2$ in N-methylformamide has been reported (1). This solvent is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (2). Later, Feakins and Lawrence (3) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use. In the current study, the solvent conductance had a mean value of 5×10^{-4} S m⁻¹, which is an order of magnitude greater than that of solvent used by other workers studying electrolyte solubility. The reported value of the solubility at 298 K is therefore proposed only as a tentative value.

Tentative value at 298 K

186 - 5 g/kg of solvent.

- 1. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., <u>1968</u>, 38, 814-819.
- 2. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 3. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

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COMPONENTS:	ORIGINAL MEASUREMENTS:
2.	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38,</i> 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of calcium chloride	in N-methylformamide at 25 °C
18.6 g/100 g sol	
AUXILIARY	INFORMATION
METHOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water free solutions and all the solutions were	Calcium chloride, reagent grade, was dried under high vacuum before use.
prepared in a dry box. Saturated calcium	N-methylformamide (Fluka product) was
chloride solutions were prepared by dissolu- ing excess of the halide in about 20-25 cm ³	purified by storing over CaO for two days before twice distilling under reduced
of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution	pressure. The conductance of the N-methyl ₁ formamide varied from 3.0 to 6.0×10^{-4} S m ⁻¹ .
was then stirred for three or more days in	
^a thermostatted bath. With the aid of a ^{syphon} provided with a terminal G-3 gooch,	
an aliquot of the saturated solution was diluted with water (1:20) and the amount of	
dissolved halide determined by a potentio- metric method using a titrimeter (type	ESTIMATED ERROR:
Radiometer TTT1) and, presumably, standard	Solubility + 1%
^{aqueous} silver nitrate. Equilibrium between solid and solution	Temperature $\stackrel{+}{=}$ 0.05 °C (author)
phases was considered to be attained when analysis of the solution, made at progressive	REFERENCES :
periods of time, gave the same value for	1. Pístoia, G.; Pecci, G.; Scrosati, B.
dissolved halide. The applicability of the method was checked by increasing the ratio	Ric. Sci., <u>1967</u> , 37, 1167-1172
of N-methylformamide to aqueous solution (1:5). The equivalence point remained the	
same(1).	

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COMPONENTS:	EVALUATOR:
(1) Calcium bromide; CaBr ₂ ; [7789-41-5] (2) N-methylformamide; C ₂ H ₅ NO;[123-39-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland.
	December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of CaBr_2 in N-methylformamide has been reported (1). This solvent is perhaps the most difficult of all the amide solvents to make and retain in a pure condition. A continuous rise in the conductance of the solvent with time was first reported by French and Glover (2). Later, Feakins and Lawrence (3) suggested that this might be due to the formation of methyl ammonium formate by a hydrolysis reaction at the vessel walls and recommended baking out containers, etc., where possible, before use. In the current study, the solvent conductance had a mean value of 5×10^{-4} S m⁻¹, which is an order of magnitude greater than that of solvent used by other workers studying electrolyte solubility. The reported value of the solubility at 298 K is therefore proposed only as a tentative value.

Tentative value at 298 K

 $303 \stackrel{+}{=} 9 \text{ g/kg of solvent.}$

- 1. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. Ric. Sci., 1968, 38, 814-819.
- 2. French, C.M.; Glover, K.H. Trans. Farad. Soc., 1955, 51, 1418-1427.
- 3. Feakins, D.; Lawrence, K.G. J. Chem. Soc. (A), 1966, 212-219.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Calcium bromide; CaBr ₂ ; [7789-41-5] (2) N-methylformamide; C ₂ H ₅ NO; [123-39-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. <i>Ric. Sci.</i> , <u>1968</u> , <i>38</i> , 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	

Solubility of calcium bromide in N-methylformamide at 25 °C

30.3 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated calcium bromide solutions were prepared by dissolving excess of the halide in about 20-25 cm³ of solvent contained in a 50 cm³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was diluted with water (1:20) and the amount of dissolved halide determined by a potentiometric method using a titrimeter (type Radiometer TTT1) and, presumably, standard aqueous silver nitrate.

Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide. The applicability of the method was checked by increasing the ratio of N-methylformamide to aqueous solution (1:5). The equivalence point remained the same (1).

SOURCE AND PURITY OF MATERIALS: Calcium bromide, reagent grade, was dried under high vacuum before use. N-methylformamide (Fluka product) was purified by storing over CaO for two days before twice distilling under reduced pressure. The conductance of the N-methylformamide varied from 3.0 to 6.0 x 10⁻⁴S m⁻¹.

ESTIMATED ERROR:

Solubility + 1%

Temperature $\stackrel{+}{=}$ 0.05 °C (author)

REFERENCES:

 Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, <u>1967</u>, 37, 1167-1172 N.N-Dimethylformamide

COMPONENTS: (1) Lithium fluoride; LiF; [7789-24-4] (2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	EVALUATOR: C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of LiF in N,N-dimethylformamide (DMF) has been reported (1). This solvent is readily prepared free from ionic impurities by vacuum distillation and the conductance found in this study, $0.32 - 1.50 \times 10^{-5}$ S m⁻¹ is as low as has ever been reported. However, as Thomas and Rochow (2) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF. $2H_20$. These authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. Again, the solvent used in this solubility study by Criss and Luksha had one of the lowest water contents so far reported ($\leq 0.002\%$ as determined by Karl Fischer titration). Solvate formation was shown to be absent in this system.

Recommended value at 298 K

 $(1.39 - 0.18) \times 10^{-3}$ g/kg of solvent

References

1. Criss, C.M.; Luksha, E. J. Phys. Chem., 1968, 72, 2966-2970.

2. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Lithium fluoride; LiF; [7789-24-4]	Criss, C.M.; Luksha, E.
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	J. Phys. Chem., <u>1968</u> , 72, 2966-2970.
5,1	
VARIABLES:	DEDADED DV.
LIDIES.	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of lithium fluoride in N,N-	-
1.39×10^{-4} g/100 g solvent	
Value calculated from solubility data given as:	
$5.34 \div 0.69 \times 10^{-5}$	mol/kg of N,N-dimethylformamide.
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of lithium fluoride were prepared in special Pyrex glass cells, 18 cm	Lithium fluoride was prepared by the dropwise
^{10ng} and 4.5 cm in diameter. The cells were	addition of aqueous HF to an aqueous solution of lithium chloride in a polyethylene beaker.
drawn out at the top in order that they could be fitted with rubber serum caps. They were	
supported in a water bath by a rocker which	ivity water, before drying at 150 °C under
oscillated 200 times/minute through an arc of about 30°.	vacuum for 24 hours and storing in sealed vials until ready for use.
Solubilities were obtained by approaching	viais until leady for use.
equilibrium from both under- and supersatur- ation. In both approaches solutions were	(continued)
analysed daily until no further concentration	
changes could be detected. The solutions were analysed either by a flame photometer	ESTIMATED ERROR:
lor by carefully evaporating to dryness and	Solubility + 13%
weighing the residue. In cases where both techniques were employed, the agreement was	Temperature $\stackrel{+}{=}$ 0.05 °C (author)
within 3%.	REFERENCES :
	REFERENCES;
	(1) Prue, J.E.; Sherrington, P.J.
	Trans. Faraday Soc., <u>1961</u> , 57. 1795-1808.
	(continued)
	,

COMPONENTS:	ORIGINAL MEASUREMENTS:
	Criss, C.M.; Luksha, E.
(1) Lithium fluoride; LiF; [7789-24-4]	J. Phys. Chem., <u>1968</u> , 72, 2966-2970.
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	

SOURCE AND PURITY OF MATERIALS: (continuation)

N,N-dimethylformamide was purified by treatment with calcium hydride accompanied by vigorous stirring and then distilling under reduced pressure through a 35 cm Vigreux column. The boiling point was 68 °C at 53 mm pressure. The reported value is 79 °C at 61-62 mm (1). The specific conductance was in the range $0.32 - 1.5 \times 10^{-5} \text{ S m}^{-1}$, which is in good agreement with the lowest values reported in the literature $(0.6 - 18.3 \times 10^{-5} \text{ S m}^{-1} (1), (2))$. There was no noticeable increase in conductivity with time.

Karl Fischer titrations indicated the water content to be less than 0.002%.

REFERENCES: (continuation)

(2) Ames, D.P.; Sears, P.G. J. Phys. Chem., 1955, 59, 16-19.

COMPONENTS:	EVALUATOR:
<pre>(1) Lithium chloride; LiCl; [7447-41-8] (2) N,N-dimethylformamide; C₃H₇NO;[68-12-2]</pre>	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

CRITICAL EVALUATION:

Three investigations of the solubility of LiCl in N,N-dimethylformamide (DMF) have been reported (1-3). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (4). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (5) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂0. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. It should be noted that none of the three groups who studied this system report that they followed the recommended method for preparing anhydrous LiCl (by passing dry HCl over the salt). The purity of the solute is unknown.

A major problem in the determination of the solubility of LiCl in this solvent is that the equilibrium state of the solid phase is almost certainly a solvate. Paul and Sreenathan (1) isolated a white solid of composition LiCl.DMF (Li: found 6.17%, required 6.06%; Cl: found 31.03%, required 30.74%). However it is not clear from their results whether the solution was in equilibrium with this solvate, or, as seems more likely, more than one solid phase was present and no equilibrium was established. None of the other authors considers the possibility of solvate formation. The wide range of solubilities reported for LiCl (114-275 g/kg of solvent) suggests that a normal solubility equilibrium cannot be measured and no solubility value is therefore recommended.

- 1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.
- ². Pistoia, G.; Pecci, G.; Scorsati, B. *Ric. Sci.*, <u>1967</u>, 37, 1167-1172.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 4. "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.) Wilmington, Delaware, U.S.A.
- ⁵. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Lithium chloride; LiC1;[7447-41-8]	Properties and uses of DIMETHYLFORMAMIDE	
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	(DMF), Du Pont Information Booklet, 1976.	
37		
VARIABLES :	PREPARED BY:	
One temperature: 25 ^O C	J.S. McKechnie	
one competituee. 25 0	J.J. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of lithium chloride in N,N	-dimethylformamide at 25 ^O C	
11.40 g/100 g solvent		
Data taken from table of semi-quantitative solubilities of inorganic materials in DMF at 25 ^O C.		
AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Not stated	Not stated	
	ESTIMATED ERROR:	
	LITTATED ERROR.	
	DEFEDENCES	
	REFERENCES:	

N, N-Dimethylformamide 15		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Lithium chloride; LiCl; [7447-41-8]	Paul, R.C.; Singla, J.P.; Lamba, M.S.;	
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	Gill, D.S.; Narula, S.P. Indian J. Chem.,	
37.00	1973, 11, 1024–1026.	
VARIABLES:	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:		
t/ ^o C	g/100 g solvent	
25	13.8	
35	12.3	
45	11.6	
	ΙΝΕΩΡΜΑΤΙΩΝ	
AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE: Saturated solutions of lithium chloride were	SOURCE AND PURITY OF MATERIALS: A.R. grade lithium chloride (BDH) was	
repared by adding excess of the powdered	recrystallised and dried under vacuum at	
alt to N,N-dimethylformamide (15-20 g) in Yrex tubes (15 x 2.5 cm) and preheating to	110-125 °C.	
¹⁰ ^o C above the required temperature of the	N,N-dimethylformamide (Riedel Pure) was dried over A.R. anhydrous sodium carbonate	
Path prior to final thermostatting. The attainment of the equilibrium was checked by	for 24 hours before distilling under reduce	
Intermittent analyses. The solutions were	pressure. The fraction boiling at 73 °C/ 57 mm was collected and had the following	
hen filtered under nitrogen and analysed or halide. No details of analytical methods	physical constants: density 0,9442 g/cm ³ ;	
^{ere} given. Transference of material, as	specific conductance 2.5 x 10^{-5} S m ^{-I} at 25 °C. The solvent was freshly distilled	
ar as possible, was carried out in a nitrogen illed dry box. Measurements were made in	for each experiment.	
riplicate.	ESTIMATED ERROR:	
	Solubility + 1%	
	Temperature $\stackrel{+}{=}$ 0.05 °C (author)	
	REFERENCES:	

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	in of final made
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Lithium chloride; LiCl; [7447-41-8]	Pistoia, G.; Pecci, G.; Scrosati, B.
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	
37	
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
·····	
EXPERIMENTAL VALUES:	
Solubility of lithium chloride in N,N-o	limethylformamide at 25 ^o C
11.3 g/100 g solvent	
1115 5,100 5 5010000	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS:	
Considerable effort was made to obtain water	Lithium chloride, reagent grade, was dried
free solutions and all the solutions were prepared in the dry box. Saturated lithium	under vacuum before use. Reagent grade N.N-dimethylformamide
chloride solutions were prepared by dissolv-	(C. Erba RP) was purified by fractional
ing excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask	distillation under reduced pressure (2 mm Hg). The final product had a specific conductance
with a standard taper joint. The solution	of 3×10^{-5} S m ⁻¹ .
was then stirred for three or more days in a thermostatted bath. With the aid of a	
syphon provided with a terminal G-3 gooch,	
an aliquot of the saturated solution was collected and weighed. This solution was	
then diluted with water (1:10) and the	ESTIMATED ERROR:
amount of dissolved halide determined volumetrically by titration with standard	Solubility + 1%
silver nitrate using potassium chromate as	Temperature ⁺ 0.05 [°] C (author)
indicator. Equilibrium between solid and solution	
phases was considered to be attained when analysis of the solution, made at progressive	REFERENCES :
periods of time, gave the same value for	
dissolved halide.	

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium chloride; LiCl; [7447-41-8]	Paul, R.C.; Sreenathan, B.R.
(2) N,N-dimethylformamide; $C_{3}H_{7}N0; [68-12-2]$	Indian J. Chem., 1 <u>966,</u> 4, 382–386.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of lithium chloride in N	N-dimethylformamide at 25 ^o C
27.5 g/100 g solvent	
27.5 g/100 g sol	vent
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of lithium chloride were	Purity of lithium chloride was not stated. N,M-dimethylformamide (J.T. Baker analysed)
prepared by adding the salt in small quantities to 10 cm of N.N-dimethylformamide	was stored over A.R. anhydrous sodium carbon-
Contained in Pyrex test tubes, until an	ate for 48 hours with occasional shaking.
appreciable amount remained undissolved. The tubes were then sealed and fixed to a shaft	The solvent was then decanted and distilled through a short column. The middle fraction
immersed and arranged to rotate in a thermo-	through a short column. The middle fraction distilling at 149-151 C/745 mm was collected
Statted water-bath at 25 °C. The temperature	and redistilled under a reduced pressure of 35 mm in an atmosphere of dry nitrogen. The
was maintained using a toluene regulator. After 24-30 hours of thorough mixing the seal	specific conductance of this N,N-dimethy1-
was broken in a dry box and the slurry	formamide measured 3 x 10^{-5} S m ⁻¹ at 25 °C.
rapidly filtered under dry conditions. From a known quantity of the solution the	ESTIMATED ERROR:
^{ruecal} was estimated and the solubility of	Solubility $+$ 1%
the salt determined. No details of the analytical methods used were given.	Temperature $\stackrel{+}{=}$ 0.1 °C (author)
	REFERENCES:
]

N, N-Dimethylformamide

COMPONENTS :	EVALUATOR:
<pre>(1) Lithium bromide; LiBr; [7550-35-8] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2]</pre>	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Two investigations of the solubility of LiBr in N,N-dimethylformamide (DMF) have been reported (1-2). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (3) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

Neither of the groups studying the solubility of LiBr report examining the solid in equilibrium with the solution with a view to determining whether solvate formation was occurring. The discrepant nature of the results (166 g/kg of solvent (1) and 251 g/kg of solvent (2)) suggests that a proper equilibrium might not have been established. No recommendation is made for a value of the solubility.

References

1. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.

- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 3. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

N, N-Dimethylformami	de
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N, N-Dimet	hylformamide 15
COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Lithium bromide; LiBr; [7550-35-8] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
VARIABLES: Temperature	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o c 25	g/100 g solvent 25.1
35	18.7
45	13.3
,	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: Saturated solutions of lithium bromide were prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in	SOURCE AND PURITY OF MATERIALS: A.R. grade lithium bromide (BDH) was recrystallised and dried under vacuum at 110-125 °C.
Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions wer	pressure. The fraction boiling at 73 °C/
then filtered under nitrogen and analysed for halide. No details of analytical method were given. Transference of material, as far as possible, was carried out in a	hbysical constants: density 0.9442 g/cm ³
nitrogen filled dry box. Measurements were made in triplicate.	
•	Solubility $\frac{1}{2}$ 1%
	Temperature [±] 0.05 [°] C (author)
	REFERENCES :

158	N, N-Dimeth	ylformamide	
COMI	PONENTS:	ORIGINAL MEASUREMENTS:	
		Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci</i> ., <u>1967</u> , <i>37</i> , 1167-1172	
VAR.	IABLES:	PREPARED BY: J.S. McKechnie	
	One temperature: 25 [°] C	J.S. MCKechnie	
EXP	ERIMENTAL VALUES:		
	Solubility of lithium bromide in N,N	-dimethylformamide at 25 ^O C	
	16.6 g/100 g solvent		
	AUXILIARY INFORMATION		
MET	HOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:	
free precession bree solution with was a the syppan colution of by usin pha ana per	ee solutions and all the solutions were epared in the dry box. Saturated lithium omide solutions were prepared by dissolving cess of the halide in about 20-25 cm ³ of lvent contained in a 50 cm ³ Pyrex flask	distillation under reduced pressure (2 mm Hg). The final product had a specific conductance of 3 x 10 ⁻⁵ S m ⁻¹ . ESTIMATED ERROR:	
1			

COMPONENTS:	EVALUATOR:
 (1) Sodium fluoride; NaF; [7681-49-4] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of NaF in N,N-dimethylformamide (DMF) has been reported (1). This solvent is readily prepared free from ionic impurities by vacuum distillation and the conductance found in this study, $0.32-1.50 \times 10^{-5}$ S m⁻¹ is as low as has ever been reported. However, as Thomas and Rochow (2) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. These authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. Again, the solvent used in this solubility study by Criss and Luksha had one of the lowest water contents so far reported (≤ 0.0027 as determined by Karl Fischer titration). Solvate formation was shown to be absent in this system.

Recommended value at 298 K

 $(1.96 \pm 0.14) \times 10^{-3}$ g/kg of solvent.

- 1. Criss, C.M.; Luksha, E. J. Phys. Chem., 1968, 72, 2966-2970.
- 2. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

100 /v,/v-Dimethylformanide	
COMPONENTS :	ORIGINAL MEASUREMENTS:
 (1) Sodium fluoride; NaF; [7681-49-4] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Criss, C.M.; Luksha, E. <i>J. Phys. Chem.</i> , <u>1968</u> , 72, 2966-2920
VARIABLES:	PREPARED BY:
One temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium fluoride in N,N-dimethylformamide at 25 ^O C	
1.96 x 10 ⁻⁴ g/100 g solvent	
Value calculated from solubility data given as:	
4.68 \div 0.34 x 10 ⁻⁵ mol/kg N,N-dimethylformamide	
	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium fluoride were prepared in special Pyrex glass cells, 18 cm long and 4.5 cm in diameter. The cells were drawn out at the top in order that they could be fitted with rubber serum caps. They were supported in a water bath by a rocker which oscillated 200 times/minute through an arc of about 30°. Solubilities were obtained by approaching equilibrium from both under- and supersatur- ation. In both approaches solutions were analysed daily until no further concentration changes could be detected. The solutions were analysed either by a flame photometer or by carefully evaporating to dryness and weighing the residue. In cases where both techniques were employed, the agreement was within 3%.	filtered and washed repeatedly with conduct- ivity water, before drying at 150 °C under
	(continued)
	ESTIMATED ERROR:
	Solubility ± 7%
	Temperature $\stackrel{+}{=}$ 0.05 $^{\circ}$ C (author)
	REFERENCES:
	(1) Prue, J.E.; Sherrington, P.J. Trans. Faraday Soc., <u>1961</u> , 57, 1795-180
	(continued)

COMPONENTS:

Sodium fluoride; NaF; [7681-49-4]
 N,N-dimethylformamide; C₃H₇NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Criss, C.M.; Luksha, E. J. Phys. Chem 1968, 72, 2966-2920

SOURCE AND PURITY OF MATERIALS: (continuation)

N,N-dimethylformamide was purified by treatment with calcium hydride accompanied by vigorous stirring and then distilling under reduced pressure through a 35 cm Vigreux column. The boiling point was 68 °C at 53 mm pressure. The reported value is 79 °C at 61-62 mm (1). The specific conductance was in the range $0.32 - 1.5 \times 10^{-5} \text{ S m}^{-1}$, which is in good agreement with the lowest values reported in the literature $(0.6 - 18.3 \times 10^{-5} \text{ S m}^{-1} (1), (2))$. There was no noticeable increase in conductivity with time.

Karl Fischer titrations indicated the water content to be less than 0.002 %.

REFERENCES: (continuation)

(2) Ames, D.P.; Sears, P.G. J. Phys. Chem., 1955, 59, 16-19.

COMPONENTS: (1) Sodium chloride; NaCl; [7647-14-5] (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	EVALUATOR: C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland.
	December 1978.

CRITICAL EVALUATION:

Three investigations of the solubility of NaCl in N,N-dimethylformamide (DMF) have been reported (1-3). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (4). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (5) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. The most anhydrous solvent used in these studies of NaCl solubility is likely to have been that of Criss and Luksha (3) who found a water content of $\leq 0.002\%$ by Karl Fischer titration. The other major difficulty which may arise in solubility measurements in DMF, namely solvate formation, was shown to be absent in this particular system.

The results of the four measurements (all at a single temperature, 298 K) are in good agreement, falling in the range 0.36 - 0.50 g/kg of solvent. The lowest value, which was obtained with the driest solvent and very careful analytical techniques is taken as the recommended value.

Recommended value at 298 K

0.36 - 0.01 g/kg of solvent.

- 1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.
- 2. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.
- 3. Criss, C.M.; Luksha, E. J. Phys. Chem., 1968, 72, 2966-2970.
- "Properties and Uses of Dimethylformamide", E.J. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.
- 5. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

COMPONENTS:	OBLOTINAL MELCURENTE
SOUT UNENID:	ORIGINAL MEASUREMENTS:
(1)Sodium chloride; NaCl:[7647-14-5]	Proportion and uses of promotive converse
	Properties and uses of DIMETHYLFORMAMIDE
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	(DMF) Du Pont Information Booklet, 1976.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium chloride in	N.N-dimethylformamide at 25 ^O C
0.05 g/100 g solvent	
Data taken from table of semi-quantitative solubilities of inorganic	
materials in DMF at 25 °C	
materials in DMF at 25 C	
	TURODULETOU
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Not stated	Not stated
	ESTIMATED ERROR:
	REFERENCES:

64 N, N-Dimethylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) 'Sodium chloride; NaCl; [7647-14-5] (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	Criss, C.M.; Luksha, E. J. Phys. Chem., <u>1968</u> , 72, 2966-2970.
WARTARY PC.	
VARIABLES: One temperature: 25 ^o C	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium chloride in N,N- 0.036 g/100 g solvent	•dimethylformamide at 25 ^o C
Value calculated from solubility data 6.08 ⁺ 0.06 x 10 ⁻³ mol/	a given as: /kg N,N-dimethylformamide
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium chloride were prepared in special Pyrex glass cells 18 cm long and 4.5 cm in diameter. The cells were drawn out at the top in order that they could be fitted with rubber serum caps. They were supported in a water bath by a rocker which oscillated 200 times/minute through an arc of about 30° Solubilities were obtained by approaching equilibrium from both under- and supersatur- ation. In both approaches solutions were analysed daily until no further concentration	SOURCE AND PURITY OF MATERIALS: Reagent grade sodium chloride was dissolved in conductivity water, treated with chlorine and reprecipitated accprding to the method of Ives and Janz (1). The salt was stored at 400 °C until ready for use. (continued)
changes could be detected. The solutions were analysed either by a flame photometer or by carefully evaporating to dryness and weighing the residue. In cases where both techniques were employed, the agreement was	ESTIMATED ERROR: Solubility ⁺ 1% Temperature ⁺ 0.05 ^o C (author)
within 3%.	REFERENCES: (1) Ives, D.J.G.; Janz, G.J. Reference Electrodes- Theory and Practice Academic Press, Inc., New York, N.Y. 1961

COMPONENTS:

(1) Sodium chloride; NaCl; [7647-14-5]
 (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Criss, C.M.; Luksha, E. J. Phys. Chem., 1968, 72, 2966-2970.

SOURCE AND PURITY OF MATERIALS; (continuation)

N,N-dimethylformamide was purified by treatment with calcium hydride accompanied by vigorous stirring and then distilling under reduced pressure through a 35 cm Vigreux column. The boiling point was 68 °C at 53 mm pressure. The reported value is 79 °C at 61-62 mm (2). The specific conductance was in the range $0.32 - 1.5 \times 10^{-5}$ S m⁻¹, which is in good agreement with the lowest values reported in the literature. $(0.6 - 18.3 \times 10^{-5}$ S m⁻¹ (2), (3)). There was no noticeable increase in conductivity with time.

Karl Fischer titrations indicated the water content to be less than 0.002 %.

REFERENCES: (continuation)

(2) Prue, J.E.; Sherrington, P.J. Trans. Faraday Soc., 1961, 57, 1795-1808.

(3) Ames, D.P.; Sears, P.G. J. Phys. Chem., 1955, 59, 16-19.

bo /v,v-Dimethylformanide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Sodium chloride; NaCl; [7647-14-5] (2) N,N-dimethylformamide; C₃H₇NO;[68-12-2] 	Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> ,1167-1172.
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium chloride in N,N-dimethylformamide at 25 $^{\circ}$ C	
0.042 g/100 g solvent	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water free solutions and all the solutions were prepared in the dry box. Saturated sodium chloride solutions were prepared by dissolv- ing excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was collected and weighed. This solution was	Sodium chloride, reagent grade, was dried under vacuum before use. Reagent grade N,N-dimethylformamide (C. Erba RP) was purified by fractional distillation under reduced pressure (2 mm Hg). The final product had a specific conductance of 3 x 10^{-5} S m ⁻¹ .
then diluted with water (1:10) and the amount of dissolved halide determined volumetrically	
by titration with standard silver nitrate using potassium chromate as indicator. Equilibrium between solid and solution	Solubility ⁺ 1% Temperature ⁺ 0.05 [°] C (author)
phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide.	REFERENCES:

N, N-Dimethylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Sodium chloride; NaCl; [7647-14-5] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2]</pre>	Paul, R.C.; Sreenathan, B.R. Indian J. Chem., <u>1966</u> , 4, 382-386.
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	L
Solubility of sodium chloride in M	N,N-dimethylformamide at 25 ⁰ C
0.05 g/100 g solvent	
	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium chloride were prepared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamide Contained in Pyrex test tubes, until an appreciable amount remained undissolved. The tubes were then sealed and fixed to a shaft immersed and arranged to rotate in a thermo- statted water-bath at 25 °C. The temperature was maintained using a toluene regulator. After 24-30 hours of thorough mixing the seal was broken in a dry box and the slurry rapidly filtered under dry conditions. From a known quantity of the solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.	carbonate for 48 hours with occasional shaking. The solvent was then decanted and distilled through a short column. The middle fraction distilling at 149-151 °C/745 mm was
	REFERENCES:

COMPONENTS :	EVALUATOR:
 (1) Sodium bromide; NaBr; [7647-15-6] (2) N,N-dimethylformamide; C₃H₇NO;[68-12-2] 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife, Scotland. December 1978.

Two investigations of the solubility of NaBr in N,N-dimethylformamide (DMF) have been reported (1-2). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (3) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

Neither of the groups studying the solubility of NaBr report examining the solid in equilibrium with the solution with a view to determining whether solvation formation was occurring. However the results of the two investigations at 298 K are in reasonable agreement and the mean is recommended as a tentative value.

Tentative value at 298 K

118 - 15 g/kg of solvent.

Valuesat other temperatures

Paul et al. (2) also studied the NaBr system at 308 K and 318 K and found that the solubility had a large negative temperature coefficient.

- 1. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 3. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

N, N-Dimethylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Sodium bromide; NaBr; [7647-15-6] N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026.
VARIABLES: Temperature	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	
t/°C 25 35 45	g/100 g solvent 13.3 10.9 9.3
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium bromide were prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	SOURCE AND PURITY OF MATERIALS: A.R. grade sodium bromide (BDH) was recrystallised and dried under vacuum at 110-125 °C. N,N-dimethylformamide (Riedel Pure) was dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced pressure. The fraction boiling at 73°C/ 57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ; specific conductance 2.5 x 10 ⁻⁵ S m ⁻¹ at 25 °C. The solvent was freshly distilled for each experiment. ESTIMATED ERROR: Solubility ⁺ 1% Temperature ⁺ 0.05 °C (author) REFERENCES:

70 /v, /v-Dimethylformanide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
	Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci</i> ., <u>1967</u> , <i>37</i> , 1167-1172.
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium bromide in N,N-dimethylformamide at 25 $^{\circ}$ C	
10.3 g/100 g solvent	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water free solutions and all the solutions were	Sodium bromide, reagent grade, was dried under vacuum before use.
prepared in the dry box. Saturated sodium bromide solutions were prepared by dissolv-	Reagent grade N,N-dimethylformamide (C.Erba RP) was purified by fractional
ing excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask	distillation under reduced pressure (2 mm Hg). The final product had a specific conductance
with a standard taper joint. The solution was then stirred for three or more days in a	of 3 x 10 ⁻⁵ S m ⁻¹ .
thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch.	
an aliquot of the saturated solution was collected and weighed. This solution was	
then diluted with water (1:10) and the	ESTIMATED ERROR:
amount of dissolved halide determined volumetrically by titration with standard	Solubility ⁺ 1%
silver nitrate using potassium chromate as indicator.	Temperature $\stackrel{+}{=}$ 0.05 °C (author)
Equilibrium between solid and solution phases was considered to be attained when	REFERENCES:
analysis of the solution made at progressive periods of time, gave the same value for	
dissolved halide.	

COMPONENTS:	EVALUATOR:
(1) Sodium iodide; NaI; [7681-82-5] (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2	C.A. Vincent.

Three investigations of the solubility of NaI in N,N-dimethylformamide (DMF) have been reported (1-3). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (4). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (5) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

In the case of NaI, the possible complication of solvate formation was excluded by Paul and Sreenathan (1) who analysed the solid phase in equilibrium with the solution. However the results of these solubility studies show little agreement, even if the very high value suggested by the duPont report is excluded. No convincing explanation of the wide range of values from the remaining three groups (37.2 - 80.5 g/kg of solvent) can be given, but it might be noted that iodide ion has been found to react with DMF, albeit to a limited extent, producing a yellow tinge in solutions of KI (6). No tentative value is recommended.

References

- 1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.
- 2. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.
- 3. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P.

Indian J. Chem., <u>1973</u>, 11, 1024-1026.

- "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.
- 5. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.
- Alexander, R.; Ko, E.C.F.; Mac. Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u>, 89, 3703-3712.

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium iodide; NaI; [7681-82-5]	Properties and uses of DIMETHYLFORMAMIDE
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	(DMF), Du Pont Information Booklet, 1976.
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
	2
Solubility of sodium iodide in N,N-dimethylformamide at 25 $^{\circ}$ C	
14.4 g/100 g solvent	
Data taken from table of semi-quantitative solubilities of inorganic	
materials in DMF at 25 °C	
AUXILIARY INFORMATION	
METHOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Not stated	Not stated
	ESTIMATED ERROR:
	REFERENCES :

N, N-Dimethylformamide 17	
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Sodium iodide; NaI; [7681-11-0] N,N-dimethylformamide; C₃H₇NO;[68-12-2] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Marula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
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VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	<u>1</u>
t/ ^o c	g/100 g solvent
25	8.1
35	9.1
45	10.4
·	
	······
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10° C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions Were then filtered under nitrogen and analysed for halide. No details of	SOURCE AND PURITY OF MATERIALS: A.R. grade sodium iodide (BDH) was recrystallised and dried under vacuum at 110-125 °C. N,N-dimethylformamide (Riedel Pure) was dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced pressure. The fraction boiling at 73 °C/ 57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ; specific conductance 2.5 x 10 ⁻⁵ S m ⁻¹ at
analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	25 °C. The solvent was freshly distilled

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium iodide; NaI; [7681-82-5]	Pistoia, G.; Pecci, G.; Scrosati, B.
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	· · · · · · · · · · · · · · · · · · ·
3, 11	
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium iodide in N,N-di	methylformamide at 25 [°] C
6.35 g/100 g solvent	
	INFORMATION
METHOD/APPARATUS/PROCEDURE: Considerable effort was made to obtain water	SOURCE AND PURITY OF MATERIALS: Sodium iodide, reagent grade, was dried
free solutions and all the solutions were prepared in the dry box. Saturated sodium	under vacuum before use. Reagent grade N,N-dimethylformamide
iodide solutions were prepared by dissolving	(C. Erba RP) was purified by fractional distillation under reduced pressure (2 mm Hg).
excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask	The final product had a specific conductance
with a standard taper joint. The solution was then stirred for three or more days in a	of $3 \times 10^{-5} \text{ S m}^{-1}$.
thermostatted bath. With the aid of a	
syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was	
collected and weighed. This solution was then diluted with water (1:10) and the amount	
of dissolved halide determined volumetrically	ESTIMATED ERROR:
by adding an excess of standard silver nitrate and back titrating against standard	Solupility ⁺ 1%
ammonium thiocyanate using ferric alum as indicator.	Temperature $\stackrel{+}{=}$ 0.05 °C (author)
Equilibrium between solid and solution	REFERENCES :
phases was considered to be attained when analysis of the solution, made at progressive	
periods of time, gave the same value for dissolved halide.	
dissince matric.	
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COMPONENTS: ORIGINAL MEASUREMENTS: (1) Sodium iodide; NaI; [0681-82-5] Paul, R.C.; Sreenathan, B.R. (2) N,N-dimethylformamide; C ₃ H ₂ MO; [80-12-2] Dadian J. Cham., 1966, 4, 382-386. VARLABLES: PREPARED BY: One temperature: 25 °C J.S. McKechnie EXPERIMENTAL VALUES: Solubility of sodium iodide in N,N-dimethylformamide at 25°C 3.72 g/100 g solvent 3.72 g/100 g solvent METHOD/APPARATUS/PROCEDURE: Source and the solution of sodium iodide vere the seale the small methylformamide is contained unisolved. MeTHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: Staturated solutions of sodium iodide vere the seale and fixed to a shaft immersed and arranged to rotate in a theremeatated vater-back is 25 °C. The tubes were then seale and aftaked to a shaft immersed and arranged to rotate in a futer mainted using a toluene tigeliator. After 24-30 hours of thorough a short colume, The indide fracture doed pressure of the soluted result of asolited metar is 25 °C. The solutions. From sknoom quantity of the salt determined. No details of the analytical methods used vere slubelity 2 12 ENTIMATED ERROR: SUTIMATED ERROR: Solubility of the salt determined. No details of the analytical methods used vere slubelity 2 12 Temperature 2 0.1 °C (author)	F	,
(2) N,N-dimethylformamide; C,JI,NO; [58-12-2] Indian J. Cham., 1966, 4, 382-386. VARIABLES: PREPARED BY: One temperature: 25 °C J.S. McKechnie EXPERIMENTAL VALUES: Solubility of sodium iodide in N,N-dimethylformamide at 25°C Solubility of sodium iodide were 3.72 g/100 g solvent MUTHON/APPARATUS/PROCEDURE: Source AND FURITY OF MATERIALS: Saturated solutions of sodium iodide were Prepared by adding the salt in small Ome temperatures dand arranged to rocke in a thermsmeed and using to tolent in dry rocket was broken in a dry rocket in a thermsmeed and arranged to rocke and the soluty ripidly filtered under the rocke and the solution the metal was estimated and the solution the metal was estimated and the solution the salt determined. No details of the analytical methods used were Siven. Solubility ± 1% Solubility ± 10 C (author)	COMPONENTS:	ORIGINAL MEASUREMENTS:
VARIABLES: PREFARED BY: One temperature: 25 °C J.S. McKechnie EXPERIMENTAL VALUES: Solubility of sodium iodide in N,N-dimethylformamide at 25°C Solubility of sodium iodide was not stated. 3.72 g/100 g solvent MUNILIANY INFORMATION MUNICAL AND PURITY OF MATERIALS: Prescrible to 10 cm 30 RN-dimethylformamide Mantifies to 10 cm 30 RN-dimethylformamide Mattifies to 10 cm 30 RN-dimethylformamide Mattifies to 10 cm 30 RN-dimethylformamide Mattifies amount remained undissolved. Mather interest and arranged to roctar in a thermised and arranged to roctar in a thermiset and aradisto to a solution the soluty roctar arrange	(1) Sodium iodide; NaI; [7681-82-5]	Paul, R.C.; Sreenathan, B.R.
One temperature: 25 °C J.S. McKechnie EXPERIMENTAL VALUES: Solubility of sodium iodide in N,N-dimethylformamide at 25°C 3.72 g/100 g solvent METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were propared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamide to 70 for MATERIALS: Surret a solutions of sodium iodide were fimmersed and arranged to rotate in a htmostated vater-bath at 25 °C. The temperature vas maintained using a toluene through and redistiled under a reduced pressure of and arranged to rotate in a htmostated vater-bath at 25 °C. The temperature vas maintained using a toluene through at 10°-5 m ⁻¹ at 25 °C. Missing the seal vas broken in a dry box and the solution. From a known quantity of the solution te metal was estimated and the solutionity of the sait determined. No distility ± 112 Solubility ± 10.1 °C (author)	(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	Indian J. Chem. <u>,1966</u> , 4, 382–386.
One temperature: 25 °C J.S. McKechnie EXPERIMENTAL VALUES: Solubility of sodium iodide in N,N-dimethylformamide at 25°C Solubility of sodium iodide in N,N-dimethylformamide at 25°C 3.72 g/100 g solvent METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were prepared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamide to 25°C. The times were then seeled and fixed to a shaft immersed and arranged to rotate in a htmostated vater-bah at 25°C. The temperature vas maintained using a toluene regulator. After 24-30 hours of thorough Mixing the seal vas broken in a dry box and the solution. Fine atled extirmed and the solutionity of the salt determined. No distilled under a reduced pressure of 35 m ⁻¹ at 25°C. ESTIMATED ERROR: Solubility 2 file analytical methods used were fiven.		
One temperature: 25 °C J.S. McKechnie EXPERIMENTAL VALUES: Solubility of sodium iodide in N,N-dimethylformamide at 25°C Solubility of sodium iodide in N,N-dimethylformamide at 25°C 3.72 g/100 g solvent METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were prepared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamide to 25°C. The times were then seeled and fixed to a shaft immersed and arranged to rotate in a htmostated vater-bah at 25°C. The temperature vas maintained using a toluene regulator. After 24-30 hours of thorough Mixing the seal vas broken in a dry box and the solution. Fine atled extirmed and the solutionity of the salt determined. No distilled under a reduced pressure of 35 m ⁻¹ at 25°C. ESTIMATED ERROR: Solubility 2 file analytical methods used were fiven.		
AUXILIARY INFORMATION AUXILIARY INFORMATION AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were Prepared by adding the salt in small Sumities to 10 cm ³ of N ₃ -dimethylformamide Sumities to 10 cm ³ of N ₃ -dimethylformamide Subility ± 112 Temperature ± 0.1 °C (author)	VARIABLES:	PREPARED BY:
AUXILIARY INFORMATION AUXILIARY INFORMATION AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were Prepared by adding the salt in small Sumities to 10 cm ³ of N ₃ -dimethylformamide Sumities to 10 cm ³ of N ₃ -dimethylformamide Subility ± 112 Temperature ± 0.1 °C (author)		
AUXILIARY INFORMATION AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were propared by adding the salt in small quanticies to 10 cm ³ of NN-dimethylformamide (J.T.Baker analysed) quanticies to 10 cm ³ of NN-dimethylformamide (J.T.Baker analysed) NN-dimethylformamide (J.T.Baker analysed) Soluting a 140-13 (J.T.Baker analysed) Soluting ta short a stimated and the Solubility j f 12 Temperature [±] 0.1 °C (author)	One temperature: 25 C	J.S. McKechnie
AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide were Prepared by adding the salt in small Quantities to 10 cm ³ of N,N-dimethylformamide (J.T.Baker analysed) The tubes were then sealed and fixed to a shaft immered and arranged to rotate in a thermostatted water-bath at 25 °C. The temperature was maintained using a toluene tempulator. After 24-30 hours of thorough mixing the seal was broken in a dry box and the sluty rapidly filtered under dry Conditions. From a known quantity of the solution the metal was estimated and the solutionity of the salt determined. No details of the analytical methods used were given. AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium iodide vare prepared by adding the salt in small quantities amount remained undissolved. The tubes were then sealed and fixed to a shaft immered and arranged to rotate in a thermostatted water-bath at 25 °C. The temperature vas mainted and the solubility of the salt determined. No details of the analytical methods used were given.	EXPERIMENTAL VALUES:	
$eq:approx_appr$	Solubility of sodium iodide in N,N-d	imethylformamide at 25 [°] C
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the slurry rapidly filtered under dry conditions. From a known quantity of the solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given. ESTIMATED ERROR: Solubility [±] 1% Temperature [±] 0.1 °C (author)	^{regulator} . After 24-30 hours of thorough	specific conductance of this N,N-dimethyl- formamide measured 3 x 10 ⁻⁵ S m ⁻¹ at 25 °C.
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Solubility of the salt determined. No details of the analytical methods used were given. Solubility - 1% Temperature - 0.1 °C (author)	^{solution} the metal was estimated and the	
Temperature - 0.1 °C (author)	^{Solub} ility of the salt determined. No ^{details} of the analytical methods used were	
REFERENCES :	given.	Temperature $\stackrel{+}{=}$ 0.1 ^o C (author)
	1	REFERENCES :
1 1		{
		1

COMPONENTS:	EVALUATOR:
(1) Sodium cyanide; NaCN; [143-33-9] (2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of NaCN in N,N-dimethylformamide (DMF) has been reported (1). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (2). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (3) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

The solubilities reported in the duPont study (2) (7.6 g/kg of solvent) and by Paul and Sreenathan (1) (187.6 g/kg of solvent) are so far apart that it is difficult to suggest a reason. One factor which may be of importance is the reactivity of N,N-dimethylformamide with cyanide ions which, at least at higher temperatures, gives rise to the formation of HCN, CO and the metal dimethylamine complex (1). No solvate is formed by NaCN with this solvent (1).

It is therefore not possible to recommend a value for the solubility of this salt.

References

1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.

2. "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.

3. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium cyanide; NaCN; [143-33-9]	Properties and uses of DIMETHYLFORMAMIDE
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	(DMF), Du Pont Information Booklet, 1976.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
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EXPERIMENTAL VALUES:	
Solubility of sodium cyanide in N,N-o	limethylformamide at 25 [°] C
0.76 g/100 g solvent	
	antine allebilities of instrumin
Data taken from table of semi-quantit materials in DMF at 25 ^O C	ative solubilities of inorganic
materials in DMF at 25 C	
	INFORMATION
METHOD / AP PARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Not stated	Not stated
	1
	ESTIMATED ERROR:
	REFERENCES:
	ł

78 N, N-Dimethylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium cyanide; NaCN;[143-33-9]	Paul, R.C.; Sreenathan, B.R.
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	Indian J. Chem., <u>1966</u> 4, 382-386.
5,	
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium cyanide in N,N	-dimethylformamide at 25 [°] C
18.8 g/100 g solven	t
·····	<u></u>
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of sodium cyanide were	Purity of sodium cyanide was not stated. N,N-dimethylformamide (J.T.Baker analysed
prepared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamid	was stored over A.R. anhydrous sodium carbo
contained in Pyrex test tubes, until an	ate for 48 nours with occasional snaking.
appreciable amount remained undissolved.	The solvent was then decanted and distilled through a short column. The middle fraction
The tubes were then sealed and fixed to a shaft immersed and arranged to rotate in a	distilling at 149-151 °C/745 mm was collect
thermostatted water-bath at 25 °C. The	and redistilled under a reduced pressure of 35 mm in an atmosphere of dry nitrogen. The
temperature was maintained using a toluene	specific conductance of this N,N-dimethyl-
regulator. After 24-30 hours of thorough	formamide measured 3 x 10 ⁻⁵ S m ⁻¹ at 25 °C

ESTIMATED ERROR:

REFERENCES:

Solubility [±] 1%

Temperature [±] 0.1 ^oC (author)

temperature was maintained using a toluene regulator. After 24-30 hours of thorough mixing the seal was broken in a dry box and the slurry rapidly filtered under dry

conditions. From a known quantity of the solution the metal was estimated and the

solubility of the salt determined. No details of the analytical methods used were

given.

COMPONENTS: (1) Sodium cyanate; NaCNO; (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2	EVALUATOR: C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland.
	December 1978.

The only investigation of the solubility of NaCNO in N,N-dimethylformamide (DMF) so far reported has been a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) (1). No details are given of either the analytical methods used nor of the purity of the solvent. At best the duPont value can be recommended as a tentative value. Note should be taken, however, of the very large discrepancy between the duPont value for the solubility of NaCN in this solvent and that reported by Paul and Sreenathan (2).

Tentative value at 298 K

0.5 [±] 0.1 g/kg of solvent.

- "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.
- 2. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium cyanate; NaCNO	Properties and uses of DIMETHYLFORMAMIDE
 (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	(DMF), Du Pont Information Booklet, 1976.
5 /	
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium cyanate in N,N-d	imethylformamide at 25 ^o C
0.05 g/100 g solven	
Data taken from table of semi-quantit	
	active solubilities of inorganic
materials in DMF at 25 °C	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Not stated	Not stated
	ESTIMATED ERROR:
	REFERENCES :

COMPONENTS: EVALUATOR:	
 (1) Sodium thiocyanate; NaCNS; [540-72-7] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] C.A. Vincen Department University St. Andrews Scotland. December 19 	of Chemistry, of St.Andrews, , Fife,

Three investigations of the solubility of NaCNS in N,N-dimethylformamide (DMF) have been reported (1-3). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (4). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (5) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

A major problem in the determination of the solubility of NaCNS in this solvent is that the true equilibrium state of the solid phase is almost certainly a solvate. Paul and Sreenathan (1) isolated a colourless solid of composition NaCNS. DMF (Na: found 15.17%, required 14.93%; S: found 21.03%, required 20.77%). However it is not clear from their results whether the solution was in equilibrium with the solvate or whether both solvate and salt solid phases were present. None of the other authors considered the possibility of solvate formation. However the solubilities reported by three of the groups show good consistency, all falling within the range 292-299 g/kg of solvent. The mean of these results is therefore recommended as a tentative value.

Tentative value at 298 K

295 - 4 g/kg of solvent.

- 1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.
- 2. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 4. "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.
- 5. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

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COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium thiocyanate; NaCNS;[540-72-7]	Properties and uses of DIMETHYLFORMAMIDE
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	(DMF), Du Pont Information Booklet, 1976.
57	
VARIABLES:	
	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of soidum thiocyanate in N	,N-dimethylformamide at 25 ^O C
29.2 g/100 g solvent	
Data taken from table of semi-quantit	ative solubilities of inorganic
materials in DMF at 25 °C	Č
	INFORMATION
METHOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Not stated.	Not stated.
	ESTIMATED ERROR:
	REFERENCES :

	hylformamide 183
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium thiocyanate; NaCNS; [540-72-7]	Paul, R.C.; Singla, J.P.; Lamba, M.S.;
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	Gill, D.S.; Narula, S.P. Indian J. Chem.,
37.00	1973, 11, 1024-1026.
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	29.5
35	20.9
45	14.9
AUXILIARY	INFORMATION
ME THOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of sodium thiocyanate	A.R. grade sodium thiocyanate (BDH) was
were prepared by adding excess of the powdered salt to N,N-dimethylformamide	recrystallised and dried under vacuum at 110-125 °C.
(15-20 g) in Pyrex tubes (15 x 2.5 cm) and	N,N-dimethylformamide (Riedel Pure) was
preheating to 10 °C above the required temperature of the bath prior to final	dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced
thermostatting. The attainment of the	pressure. The fraction boiling at 73 °C/
^{eq} uilibrium was checked by intermittent ^{analyses} . The solutions were then filtered	57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ;
under nitrogen and analysed for halide. No	specific conductance 2.5 x 10 ⁻⁵ S m ⁻¹ at
details of analytical methods were given. ^{Transference} of material, as far as possible,	25 °C. The solvent was freshly distilled for each experiment.
was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	ESTIMATED ERROR:
	Solubility ± 1%
	Temperature $\stackrel{+}{=}$ 0.05 °C (author)
	REFERENCES:
	1

	monnanao
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium thiocyanate; NaCNS; [540-72-7]	Pistoia, G.; Pecci, G.; Scrosati, B.
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	Ric. Sci., <u>1967</u> , 37, 1167-1172
5,	
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium thiocyanate	in N.N-dimethylformamide at 25 ^O C
29.9 g/100 g solv	ent
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water free solutions and all the solutions were	Sodium thiocyanate, reagent grade, was dried under vacuum before use.
prepared in the dry box. Saturated sodium thiocyanate solutions were prepared by	Reagent grade N,N-dimethylformamide
dissolving excess of the halide in about	(C. Erba RP) was purified by fractional distillation under reduced pressure (2 mm Hg).
$20-25 \text{ cm}^3$ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint.	The final product had a specific conductance of 3×10^{-5} S m ⁻¹ .
The solution was then stirred for three or more days in a thermostatted bath. With the	
aid of a syphon provided with a terminal G-3	
gooch, an aliquot of the saturated solution was collected and weighed. This solution	
was then diluted with water (1:10) and the amount of dissolved halide determined	ESTIMATED ERROR:
volumetrically presumably by Mohr titration using dichlorofluorescein as indicator.	Solubility + 1%
Equilibrium between solid and solution	Temperature $\stackrel{+}{=}$ 0.05 °C (author)
phases was considered to be attained when analysis of the solution made at progressive	REFERENCES :
periods of time, gave the same value for dissolved halide.	
1	

COMPONENTS:	ORIGINAL MEASUREMENTS:
 Sodium thiocyanate; NaCNS; [540-72-7] N,N-dimethylformamide; C₃H₇NO; [68-12] 	Paul, R.C.; Sreenathan, B.R. 2-2] Indian J. Chem., 1966, 4, 382-386
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of sodium thiocyanat	e in N,N-dimethylformamide at 25 ^O C
21.3 g/100 g solvent	

AUXILIARY	INFORMATION

METHOD/APPARATUS/PROCEDURE: Saturated solutions of sodium thiocyanate were prepared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamide contained in Pyrex test tubes, until an appreciable amount remained undissolved. The tubes were then sealed and fixed to a shaft immersed and arranged to rotate in a thermo- statted water-bath at 25 °C. The temperature was maintained using a toluene regulator. After 24-30 hours of thorough mixing the seal was broken in a dry box and the slurry rapidly filtered under dry conditions. From a known quantity of the solution the metal was estimated and the solubility of the salt determined. No details of the analytical nethods used were given.	through a short column. The middle fraction distilling at 149-151 °C/745 mm was collected and redistilled under a reduced pressure of 35 mm in an atmosphere of dry nitrogen. The specific conductance of this N,N-dimethyl-
	REFERENCES :

COMPONENTS :	EVALUATOR:
(1) Potassium chloride; KC1; [7447-40-7] (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Five investigations of the solubility of KC1 in N,N-dimethylformamide (DMF) have been reported (1-5). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (6). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (7) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. The most anhydrous solvent used in these studies of KCl solubility is likely to have been that of Criss and Luksha (4) who found a water content of $\leq 0.002\%$ by Karl Fischer titration. The other major difficulty which may arise in solubility measurements in DMF, namely solvate formation, was shown to be absent in this particular system.

The six solubilities reported at 298 K fall into two groups. The value of 0.50 g/kg of solvent found by Paul and Sreenathan (1) and by Paul et al. (5) is supported by the duPont study. A considerably lower value was determined by the three other groups. Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by Alexander et al. (3) with the molal solubilities of the other two groups. However the latter result is certainly consistent with those of Criss and Luksha (4) and Pistoia et al. (2). Taking into account the known low water content of the solvent used by Criss and Luksha, the recommended value is taken as the mean of the solubilities reported by Criss and Luksha and by Pistoia et al.

Recommended value at 298 K

0.185 ⁺ 0.020 g/kg of solvent.

Values at other temperatures

Paul et al. (5) also measured the solubility of KCl at 308 K and 318 K. Since their value at 298 K has been rejected, no recommended or tentative value is given at these temperatures. However it might be noted that the temperature coefficient of solubility appears to be small and negative.

- 1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.
- 2. Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, <u>1967</u>, 37, 1167-1172.
- Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u>, 89, 3703-3712.
- 4. Criss, C.M.; Luksha, E. J. Phys. Chem., 1968, 72, 2966-2970.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.
- 7. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., <u>1957</u>, 79, 1843-1848.

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COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KC1; [7447-40-7]	Properties and uses of DIMETHYLFORMAMIDE
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	
3/11	
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubilition of notassium chloride	e in N,N-dimethylformamide at 25 ^o C
0.05 g/100 g s	olvent
	titative solubilities of inorganic
materials in DMF at 25 ^O C	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Not stated	Not stated
	ESTIMATED ERROR:
	REFERENCES :
1	

B8 N, N-Dimeth	ylformamide
COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Potassium chloride; KC1; [7447-40-7] (2) N,N-dimethylformamide; C₃H₇N0; [68-12-2] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	0.05
35 45	0.04 0.04
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium chloride were prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	

/v,/v-Dimethy	ynormamide 185
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Potassium chloride; KCl; [7447-40-7] N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Criss, C.M.; Luksha, E. J. Phys. Chem., 1968, 72, 2966-2970.
VARIABLES:	
One temperature: 25 ^o C	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of potassium chloride in N 0.017 g/100 g solve	
Value calculated from solubility dat	a given as:
$2.28 \stackrel{+}{=} 0.07 \times 10^{-3}$	mol/kg N,N-dimethylformamide
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of potassium chloride were prepared in special Pyrex glass cells, 18 cm long and 4.5 cm in diameter. The cells were drawn out at the top in order that they Could be fitted with rubber serum caps. They were supported in a water bath by a rocker	Reagent grade potassium chloride was further purified by double recrystallisation from conductivity water. The salt was stored at 400 °C until ready for use.
which oscillated 200 times/minute through an arc of about 30°. Solubilities were obtained by approaching equilibrium from both under- and supersatur- ation. In both approaches solutions were analysed daily until no further concentration	(continued)
Changes could be detected. The solutions Were analysed either by a flame photometer	ESTIMATED ERROR:
or by carefully evaporating to dryness and Weighing the residue. In cases where both techniques were employed, the agreement was	Solubility ⁺ 3% Temperature ⁺ 0.05 [°] C (author)
Within 3%.	REFERENCES:
	(1)Prue, J.E.; Sherrington, P.J. Trans. Faraday Soc., <u>1961</u> , 57, 1795-1808
	(continued)

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KC1; [7447-40-7]	Criss, C.M.; Luksha, E. J. Phys. Chem.
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	<u>1968</u> , 72, 2966-2970.
SOURCE AND PURITY OF MATERIALS; (continuation N,N-dimethylformamide was purified by treatment vigorous stirring and then distilling under reduc column. The boiling point was 68 °C at 53 mm at 61-62 mm (1). The specific conductance was which is in good agreement with the lowest value ($0.6 - 18.3 \times 10^{-5}$ S m ⁻¹ (1), (2)). There was with time. Karl Fischer titrations indicated the water of	with calcium hydride accompanied by used pressure through a 35 cm Vigreux pressure. The reported value is 79 $^{\circ}$ C in the range 0.32 - 1.5 x 10 ⁻⁵ S m ⁻¹ , es reported in the literature no noticeable increase in conductivity

REFERENCES: (continuation)

(2) Ames, D.P.; Sears, P.G. J. Phys. Chem., 1955, 59, 16-19.

N,N-Dimetrynormaniae		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
 Potassium chloride; KCl; [7447-40-7] N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1167-1172	
VARIABLES:	PREPARED BY:	
One temperature: 25 ^O C	J.S. McKechnie	
EXPERIMENTAL VALUES: Solubility of potassium chloride in N,N-dimethylformamide at 25 °C 0.02 g/100 g solvent		
AUXILIARY	INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Considerable effort was made to obtain water free solutions and all the solutions were prepared in the dry box. Saturated potassium chloride solutions were prepared by dissolving excess of the halide in about $20-25 \text{ cm}^3$ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated	Botassium chloride, reagent grade, was dried under vacuum before use. Reagent grade N,N-dimethylformamide (C. Erba RP) was purified by fractional distillation under reduced pressure (2 mm Hg). The final product had a specific conductance of 3 x 10 ⁻⁵ S m ⁻¹ .	
solution was collected and weighed. This solution was then diluted with water (1:10) and the amount of dissolved halide determined volumetrically by titration with standard silver nitrate using potassium chromate as indicator. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide.	Solubility - 1% Temperature + 0.05 °C (author) REFERENCES:	

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192 W, N-Dimethylformamide	
ORIGINAL MEASUREMENTS:	
Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. <i>J. Am. Chem. Soc.</i> , <u>1967</u> , 89, 3703-3712.	
PREPARED BY:	
J.S. McKechnie	
n N,N-dimethylformamide at 25 ^O C	
saturated solution.	
coduct data given as:	
-5.4	
INFORMATION	
SOURCE AND PURITY OF MATERIALS: A.R. grade potassium chloride was used without further preparation. N.N-dimethylformamide was dried with Type 4A molecular sieves and fractionated twice under a reduced pressure of dry nitrogen. It was stored over molecular sieves in dark bottles and was used within 7 days of purification. The stock solution of 0.01 mol dm ⁻³ AgNO ₃ for the potentiometric titrations was also stored in a light proof container. ESTIMATED ERROR: Solubility [±] 2% Temperature [±] 0.1 ^o C (compiler) REFERENCES:	

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloríde; KCl; [7447-40-7] (2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	Paul, R.C.; Sreenathan, B.R. Indian J.Chem., <u>1966</u> , 4, 382-386
VARIABLES:	PREPARED BY:
	PREPARED BI:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of potassium chlor	ide in N,N-dimethylformamide at 25 [°] C
0.05 g/100 g	solvent
AUXILIARY	INFORMATION
METHOD / AP PARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS;
Saturated solutions of potassium chloride were prepared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamide contained in Pyrex test tubes, until an appreciable amount remained undissolved. The tubes were then sealed and fixed to a shaft immersed and arranged to rotate in a thermostatted water-bath at 25 °C. The temperature was maintained using a toluene regulator. After 24-30 hours of thorough mixing the seal was broken in a dry box and the slurry rapidly filtered under dry conditions. From a known quantity of the	Purity of potassium chloride was not stated. N,N-dimethylformamide (J.T.Baker analysed) was stored over A.R. anhydrous sodium carbonate for 48 hours with occasional shaking. The solvent was then decanted and distilled through a short column. The middle fraction distilling at 149-151 °C/ 745 mm was collected and redistilled under a reduced pressure of 35 mm in an atmosphere of dry nitrogen. The specific conductance of this N,N-dimethylformamide measured 3 x 10 ⁻⁵ S m ⁻¹ at 25 °C.
solution the metal was estimated and the ^{Sol} ubility of the salt determined. No	ESTIMATED ERROR: Solubility [±] 10%
details of the analytical methods used were Siven.	Temperature $\stackrel{+}{=}$ 0.1 °C (author)
	REFERENCES:
L	1

COMPONENTS :	EVALUATOR:
 (1) Potassium bromide; KBr; [7758-02-3] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Three investigations of the solubility of KBr in N,N-dimethylformamide (DMF) have been reported (1-3). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (4) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments but concluded that some water always remained in the solvent.

None of the groups studying the solubility of KBr report analysing the solid in equilibrium with the solution in order to exclude the possible complication of solvate formation. The value of the molar solubility determined by Alexander et al. (2) cannot be directly compared with the molal solubilities of the other two groups. However since the solubility of this salt is rather low and the density of the solvent is close to unity the discrepancy between molal and molar values is unlikely to be large. This suggests that the solubility reported by Pistoia et al. (1) is a reasonable estimate and that the very low value of Paul et al. (3) should be rejected.

Recommended value at 298 K

8.20 [±] 0.50 g/kg of solvent

- 1. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.
- Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u>, 89, 3703-3712.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 4. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

N, N-Dimethylformamide 195		
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Potassium bromide; KBr; [7758-02-3] (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u> , 11, 1024-1026.	
VARIABLES:	PREPARED BY:	
Temperature	J.S. McKechnie	
EXPERIMENTAL VALUES:	L	
t/ [°] C	g/100 g solvent	
25	0.12	
35	0.10	
45	0.09	
	· · · · · · · · · · · · · · · · · · ·	
AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Saturated solutions of potassium bromide were Prepared by adding excess of the powdered	rerystallised and dried under vacuum at	
salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to	110-125 ^o C. N,N-dimethylformamide (Riedel Pure) was	
10 °C above the required temperature of the	dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced	
bath prior to final thermostatting. The attainment of the equilibrium was checked	pressure. The fraction boiling at 73 °C/	
by intermittent analyses. The solutions were then filtered under nitrogen and analysed	57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ;	
for halide. No details of analytical ^{methods} were given. Transference of	specific conductance 2.5 x 10^{-5} S m ⁻¹ at 25 °C. The solvent was freshly distilled for	
material, as far as possible, was carried	each experiment.	
Out in a nitrogen filled dry box. Measurements were made in triplicate.	ESTIMATED ERROR:	
	Solubility = 1% Temperature = 0.05 °C (author)	
	REFERENCES :	
L		

196 <i>N, N</i> -Dimethylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Potassium bromide; KBr;[7758-02-3] (2) N,N-dimethylformamide; C₃H₇NO;[68-12-2] 	Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1167-1172.
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie
EXPERIMENTAL VALUES:	L
Solubility of potassium bromide in N,N-dimethylformamide at 25 ^O C 0.82 g/100 g solvent	
AUXILIARY INFORMATION	
METHOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain water free solutions and all the solutions were prepared in the dry box. Saturated potassium bromide solutions were prepared by dissolving excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was collected and weighed. This solution was then diluted with water (1:10) and the amount of dissolved halide determined volumetrically by titration with standard silver nitrate using potassium chromate as indicator. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution made at progressive periods of time, gave the same value for dissolved halide.	Potassium bromide, reagent grade, was dried under vacuum before use. Reagent grade N,N-dimethylformamide (C. Erba RP) was purified by fractional distillation under reduced pressure (2 mm Hg). The final product had a specific conductance of 3 x 10 ⁻⁵ S m ⁻¹ .
	ESTIMATED ERROR: Solubility <u>+</u> 1% Temperature <u>+</u> 0.05 °C (author) REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Potassium bromide; KBr; [7758-02-3] (2) N,N-dimethylformamide; C₃H₇NO;[68-12-2]</pre>	Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u> , 89, 3703-3712.
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie.

EXPERIMENTAL VALUES:

Solubility of potassium bromide in N,N-dimethylformamide at 25 $^{\rm o}{\rm C}$

7.51 g/1000 cm^3 of saturated solution

Value calculated from solubility product data given as:

 $\log(K_{s}o/mol dm^{-3}) = -2.4$

AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Saturated solutions of potassium bromide were	A.R. grade potassium bromide was used	
prepared by shaking the solid with solvent in	without further purification.	
a stoppered flask at 35 °C for 24 hours. The	DMF was dried with Type 4A molecular	
flask was then shaken for a further 24 hours	sieves and fractionated twice under a	
at 25 ^o C. The liquid phase was analysed for halide potentiometrically using silver concen-	reduced pressure of dry nitrogen. It was stored over molecular sieves in dark bottles	
tration cells with a tetraethylammonium	and was used within 7 days of purification.	
Picrate salt bridge. The cells were wrapped	The stock solution of 0.01 mol dm ⁻³ AgNO ₂	
in aluminium foil and immersed in a 25 °C	for the potentiometric titrations was also ³	
thermostat and stirred magnetically, as	stored in a light proof container.	
standard 0.01 mol dm ⁻³ silver nitrate was		
added. The emf was measured on a Radiometer		
pH meter (type PHM 22r). It was established	ESTIMATED ERROR:	
early in the work that effectively the same results were obtained no matter whether meas-		
urements were made in the dark on freshly	Solubility + 2%	
Prepared solutions, on solutions exposed to	Temperature $\stackrel{+}{\sim}$ 0.1 °C (compiler).	
the atmosphere for 30 minutes, or on solutions		
kept under dry nitrogen during preparation and		
titration.		
The usual procedure was to titrate 0.01 mol		
dm ⁻³ AgNO ₃ solution into a half-cell contain-		
ing the silver wire immersed in 20 cm^3 of 0.01 mol dm ⁻³ potassium bromide in DMF.		
The reference half-cell was a silver wire in		
0.01 mol dm ⁻³ AgNO ₂ in the same solvent.		

COMPONENTS:	EVALUATOR:
 (1) Potassium iodide; KI; [7681-11-0] (2) N,N-dimethylformamide; C₃H₇NO;[68-12-2] 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Four investigations of the solubility of KI in N,N-dimethylformamide (DMF) have been reported (1-4). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (5). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (6) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

In the case of KI, the possible complication of solvate formation was excluded by Paul and Sreenathan (1) who analysed the solid phase in equilibrium with the solution. However the results of these solubility studies show little agreement. Alexander et al. (3) found that some oxidation of the iodide ion always occurred and this gave the solution a yellow tinge. Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by the latter authors with the molal solubilities of the other groups. However the value of Alexander et al. (295 g/dm³ of saturated solution) is likely to fall between the central values of Paul et al. (4) (296 g/kg of solvent and of the duPont study (5) (250 g/kg of solvent). If the high value of Pistoia et al. (2) and the low value of Paul and Sreenathan (1) are excluded, the mean of the remaining values appears to be a reasonavle estimate of the solubility.

Tentative value at 298 K

273 ⁺ 23 g/kg of solvent

- 1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.
- 2. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.
- Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u>, 89, 3703-3712.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 5. "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.) Wilmington, Delaware, U.S.A.
- 6. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

COMPONENTIC	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0]	Properties and uses of DIMETHYLFORMAMIDE
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	(DMF), Du Pont Information Booklet, 1976.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
	9
Solubility of potassium iodide in N,N-	dimethylformamide at 25 °C
25 g/100 g solvent	
Data taken from table of semi-quantita	tive solubilities of increanic
materials in DMF at 25 °C.	cive solubilities of inorganic
,	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Net stated	Net stated
Not stated	Not stated
· ·	
	ESTIMATED ERROR:
	REFERENCES :
	REFERENCES :
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COMPONENTS: (1) Potassium iodide; KI; [7681-11-0] (2) N,N-dimethylformamide; C ₃ H ₃ N0;[66-12-2] (3) N,N-dimethylformamide; C ₃ H ₃ N0;[66-12-2] VARIABLES: Temperature EXPERIMENTAL VALUES: C/ ⁰ C C C C C C C C C C C C C C	00 N, N-Dimethylformamide	
(2) N,N-dimethylformamide; C ₃ H ₂ NO;[68-12-2] Gill, D.S.; Narula, S.P. Indum J. Chem., 1973, 12, 1024-1026. VARIABLES: Temperature J.S. McKechnie EXPERIMENTAL VALUES: PREPARED BY: J.S. McKechnie ±/°C g/100 g solvent 25 29.6 35 31.9 45 35.4 EHOD/APPARATUS/FROCEDURE: Source AND PURITY OF MATERIALS: A.R. grade potassium iodide work sprare by adding excess of the powdered prit to N, Primetrylformamide (12-20 g) in sprare tubes (15 x 2.5 cm) and proheeting to hat prior to final thermostating. The statiament of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analytical methods were given. Transformero of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate. Source AND PURITY OF MATERIALS: A.R. grade potassium iodide (Bill) was for 24 hours before distilling under reduced pressure. The fraction boiling at 73 °C/ Source and experiment ESTIMATED ERROR: Solubility ² 1% Temperature [±] 0.05 °C (author)	COMPONENTS:	ORIGINAL MEASUREMENTS:
J.S. McKechnie EXPERIMENTAL VALUES: t/°C g/100 g solvent 25 29.6 35 31.9 45 35.4 AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium iodide were prepared by adding excess of the powdered salt to N. M-dimethylformamide (15-20 g) in Pyrex tubes (15 × 2.5 cm) and preheating to 10 °C dover the required temperature of the solutions of the equilibrium was checked by intermittent analysees. The solutions was collected and had the following at 73 °C/ 57 mm was collected and had the following physical constants: density 0.94/2 g/cm ² ; messurements were made in triplicate.		Gill, D.S.; Narula, S.P. Indian J. Chem.,
AUXILIARY INFORMATION AUXILIARY INFORMATION AUXILIARY INFORMATION AUXILIARY INFORMATION AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium iodide were prepared by adding excess of the powdered salt to N. N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the salting to the fatainment of the equilibrium was checked by intermittent analysee. The solutions were then filtered under mitrogen and analysed for halide. No identials of analytical methods were given. Transference of material, as far as possible, was carried analytical methods were given. Transference of material, as far as possible, was carried of conductance 2.5 x 10 ⁻⁵ S may as collected and had the following physical constants: density 0.94/2 g/cm ² ; memperature [±] 0.05 °C (author)	VARIABLES:	PREPARED BY:
t/°C g/100 g solvent 25 29.6 35 31.9 45 35.4 AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassiun iodide were prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostating. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analytical methods were given. Transference of material, as far as possible, was carried analytical methods were given. Transference of material, as far as possible, was carried to the intermet material day box. Source and presument Messurements were made in triplicate. Source and cheven in trip	Temperature	J.S. McKechnie
25 29.6 35 31.9 45 35.4 45 35.4 MUNILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium iodide were grepared by adding excess of the powdered salt to N.N-dimethylformamide (15-20 g) in Fyrex tubes (15 × 2.5 cm) and preheating to 10 °C above the required temperature of the attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analytical methods were given. Transforence of material, as far as possible, was carried to the subtime time filted dry box. Solubility ± 1% Kensurements were made in triplicate. FSTIMATED ERROR: Solubility ± 1%	EXPERIMENTAL VALUES:	
35 31.9 45 35.4 AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium iodide were prepared by adding excess of the powdered salt to N.N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Solubility ± 1% Temperature ± 0.05 °C (author)	t/°C	g/100 g solvent
45 35.4 AS 35.4 MUXILIARY INFORMATION INFORMATION METHOD/APPARATUS/PROCEDURE: Suturated solutions of potassium iodide were status of the powdered salt to N,M-dimethylformamide (15-20 g) in Pyres tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried analytical methods were given. Transference of material, as far as possible, was carried over A.R. anhytorous solium carbonate for each experiment. Source AND PURITY OF MATERIALS: Material, as far as possible, was carried and prove the regulation of material, as far as possible, was carried over A.R. anhytorous solium carbonate for each experiment. Source AND PURITY OF MATERIALS: Material, as far as possible, was carried and the following at 73 °C/ 57 mm was collected and had the following at 73 °C/ 57 mm was collected and had the following at 73 °C/ 57 mm was collected and had the following at 73 °C/ 50 °C. The solvent was freshly distilled for each experiment. Source AND PURITY ERROR: Solubility ± 17 Temperature ± 0.005 °C (author).	25	29.6
$eq:spectral_$		
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium iodide were prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate. SOURCE AND PURITY OF MATERIALS: A.R. grade potassium iodide (BDH) was recrystallised and dried under vacuum at 110-125 °C. N,N-dimethylformamide (Riedel Pure) was dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced pressure. The fraction boiling at 73 °C/ 57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ; specific conductance 2.5 x 10 ⁻⁵ S m ⁻¹ at 25 °C. The solvent was freshly distilled for each experiment ESTIMATED ERROR: Solubility $\frac{+}{2}$ 1% Temperature $\frac{+}{2}$ 0.05 °C (author)		
METHOD/APPARATUS/PROCEDURE:Source and prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.Source AND PURITY OF MATERIALS: A.R. grade potassium iodide (BDH) was recrystallised and dried under vacuum at 110-125 °C. N,N-dimethylformamide (Riedel Pure) was dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced pressure. The fraction boiling at 73 °C/ 57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ; specific conductance 2.5 x 10 ⁻⁵ S m ⁻¹ at 25 °C. The solvent was freshly distilled for each experimentESTIMATED ERROR: Solubility $\frac{1}{2}$ 1% Temperature $\frac{1}{2}$ 0.05 °C (author)		
Saturated solutions of potassium iodide were prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No 'details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate. A.R. grade potassium iodide (BDH) was recrystallised and dried under vacuum at 110-125 °C. N,N-dimethylformamide (Riedel Pure) was dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced pressure. The fraction boiling at 73 °C/ 57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ; specific conductance 2.5 x 10 ⁻⁵ S m ⁻¹ at 25 °C. The solvent was freshly distilled for each experiment ESTIMATED ERROR: Solubility $\stackrel{+}{-17}$ Temperature $\stackrel{+}{-0.05}$ °C (author)	AUXILIARY	INFORMATION
	METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium iodide were prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No 'details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box.	SOURCE AND PURITY OF MATERIALS: A.R. grade potassium iodide (BDH) was recrystallised and dried under vacuum at 110-125 °C. N,N-dimethylformamide (Riedel Pure) was dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced pressure. The fraction boiling at 73 °C/ 57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ; specific conductance 2.5 x 10 ⁻⁵ S m ⁻¹ at 25 °C. The solvent was freshly distilled for each experiment ESTIMATED ERROR: Solubility ⁺ 1% Temperature ⁺ 0.05 °C (author)

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COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI;[7681-11-0]	Pistoia, G.; Pecci, G.; Scrosati, B.
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	Ric. Sci., <u>1967</u> , 37, 1167-1172
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES: Solubility of potassium iodide in N,N-	-dimethylformamide at 25 °C
41.6 g/100 g solvent	

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Considerable effort was made to obtain water free solutions and all the solutions were prepared in the dry box. Saturated potassium iodide solutions were prepared by dissolving excess of the halide in about 20-25 cm of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was collected and weighed. This solution was then diluted with water (1:10) and the amount of dissolved halide determined volumetrically by adding an excess of standard silver nitrate and back titrating against standard ammonium thiocyanate using ferric alum as indicator. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive Periods of time, gave the same value for dissolved halide.	(C. Erba RP) was purified by fractional distillation under reduced pressure (2mm Hg). The final product had a specific conductance of 3 x 10^{-5} S m ⁻¹ .

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COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0]	Alexander, R.; Ko, E.C.F.; Mac, Y.C.;
(2) N,N-dimethylformamide; $C_{3}H_{7}NO$; [68-12-2]	Parker, A.J. J. Am. Chem. Soc., <u>1967</u> , 89,
	3703-3712.
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	······································
Solubility of potassium iodide in	N,N-dimethylformamide at 25 [°] C
295.21 g/1000 cm ³ of sat	
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Value calculated from solubility p	foduct data given as:
$\log (\kappa_{\rm s} o/mo1 \rm{dm}^{-3}) = 0.5$	
	i
	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium iodide were	SOURCE AND PURITY OF MATERIALS: A.R. grade potassium iodide was used
prepared by shaking the solid with solvent in	
a stoppered flask at 35 °C for 24 hours. The flask was then shaken for a further 24 hours	sieves and fractionated twice under a reduced
at 25 °C. The liquid phase was analysed for halide potentionmetrically using silver con-	pressure of dry nitrogen. It was stored over molecular sieves in dark bottles and was used
centration cells with a tetraethylammonium	within 7 days of purification.
picrate salt bridge. The cells were wrapped in aluminium foil and immersed in a 25 °C	The stock solution of 0.01 mol dm AgNO for the potentiometric titrations was also ³
thermostat and stirred magnetically, as	stored in a light proof container.
standard 0.01 mol dm silver nitrate was added. The emf was measured on a Radiometer	
pH meter (type PHM 22r). It was established	ESTIMATED ERROR:
early in the work that effectively the same results were obtained no matter whether	Solubility [±] 2%
measurements were made in the dark on freshly	-
prepared solutions, on solutions exposed to the atmosphere for 30 minutes, or on solution	
kept under dry nitrogen during preparation	REFERENCES :
and titration. The usual procedure was to titrate 0.01 mol	
dm ⁻³ AgNO ₃ solution into a half-cell contain- ing the silver wire immersed in 20 cm ³ of	
0.01 mol dm potassium iodide in DMF.	
The reference half-cell was a silver wire in 0.01 mol dm ⁻³ AgNO ₃ in the same solvent.	

N, N-Dimethylformamide 203	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-82-5]	Paul, R.C.; Sreenathan, B.R.
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of potassium iodide in N,N	-dimethylformamide at 25 ^O C
13.7 g/100 g solvent	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of potassium iodide were prepared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamide contained in Pyrex test tubes, until an	Purity of potassium iodide was not stated. N,N-dimethylformamide (J.T.Baker analysed) was stored over A.R. anhydrous sodium carbonate for 48 hours with occasional
appreciable amount remained undissolved. The tubes were then sealed and fixed to a shaft immersed and arranged to rotate in a thermostatted water-bath at 25 °C. The	shaking. The solvent was then decanted and distilled through a short column. The middle fraction distilling at 149-151 °C/ 745 mm was collected and redistilled under a
temperature was maintained using a toluene regulator. After 24-30 hours of thorough mixing the seal was broken in a dry box and the slurry rapidly filtered under dry	reduced pressure of 35 mm in an atmosphere of dry nitrogen. The specific conductance of this N,N-dimethylformamide measured 3×10^{-5} S m ⁻¹ at 25 °C.
conditions. From a known quantity of the solution the metal was estimated and the	ESTIMATED ERROR:
solubility of the salt determined. No details of the analytical methods used were	Solubility $\frac{1}{2}$
given.	Temperature ⁺ 0.1 ^o C (author)
	REFERENCES :
L	

COMPONENTS :	EVALUATOR:
 (1) Potassium cyanide; KCN; [151-50-8] (2) N,N-dimethylformamide; C₃H₇NO;[68-12-2] 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

The only investigation of the solubility of KCN in N,N-dimethylformamide (DMF) so far reported has been a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) (1). No details are given of either the analytical methods used nor of the purity of the solvent. At best the duPont value can be recommended as a tentative value. Note should be taken, however of the very large discrepancy between the duPont value for the solubility of NaCN in this solvent and that reported by Paul and Sreenathan (2).

Tentative value at 298 K

 $2.2 \stackrel{+}{=} 0.2 \text{ g/kg of solvent}$

- 1. "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.
- 2. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966,4, 382-386.

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium cyanide; KCN; [151-50-8]	Properties and uses of DIMETHYLFORMAMIDE
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of potassium cyanide in N,N-	dimethylformamide at 25 °C
0.22 g/100 g solvent	
Data taken from table of semi-quantitat	ive solubilities of inorganic
materials in DMF at 25 °C.	
AUXILIARY	INFORMATION
METHOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Not stated	Not stated
	ESTIMATED ERROR:
	REFERENCES:
h	

COMPONENTS :	EVALUATOR:
<pre>(1) Potassium cyanate; KCNO; (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2</pre>	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978

CRITICAL EVALUATION:

The only investigation of the solubility of KCNO in N,N-dimethylformamide (DMF) so far reported has been a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) (1). No details are given of either the analytical methods used nor of the purity of the solvent. At best the duPont value can be recommended as a tentative value. Note should be taken, however, of the very large discrepancy between the duPont value for the solubility of NaCN in this solvent and that reported by Paul and Sreenathan (2).

Tentative value at 298 K

 $1.2 \stackrel{+}{=} 0.2 \text{ g/kg of solvent}$

- 1. "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.
- 2. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium cyanate; KCNO;	Properties and uses of DIMETHYLFORMAMIDE
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	(DMF), Du Pont Information Booklet, 1976.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
	<u>^</u>
Solubility of potassium cyanate :	in N,N-dimethylformamide at 25 ^o C
0.12 g/100 g so	lvent
Data taken from table of semi-qua	antitative solubilities of inorganic
materials in DMF at 25 °C.	
· · · · · · · · · · · · · · · · · · ·	
	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Not stated	Not stated.
	ESTIMATED ERROR:
	REFERENCES :

COMPONENTS :	EVALUATOR:
 (1) Potassium thiocyanate; KCNS; [333-20-2] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	

CRITICAL EVALUATION:

Two investigations of the solubility of KCNS in N,N-dimethylformamide (DMF) have been reported (1-2). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (3). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (4) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

A major problem in the determination of the solubility of KCNS in this solvent is that the equilibrium state of the solid phase is almost certainly a solvate. Paul and Sreenathan (1) isolated a colourless solid of composition KCNS.DMF (K: found 23.42%, required 22.94%; S 19.31%, required 18.83%). However it is not clear from their results whether the solution was in equilibrium with this solvate, or, as seems more likely, more than one solid phase was present and no equilibrium was established. None of the other authors considered the possibility of solvate formation. The wide range of solubilities reported for KCNS (159,7 - 400.7 g/kg of solvent) suggests that a normal solubility equilibrium cannot be measured and no solubility value is therefore recommended.

- 1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.) Wilmington, Delaware, U.S.A.
- 4. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

COMPONENTS :	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium thiocyanate; KCNS; [333-20-0]	Properties and uses of DIMETHYLFORMAMIDE
	(DMF), Du Pont Information Booklet, 1976.
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	(DMF), Du Font Information Dooklet, 1970.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of potassium thiocyanate in N	,N-dimethylformamide at 25 °C
18.2 g/100 g solvent	
Data taken from table of semi-quantitati	ve solubilities of inorganic
materials in DMF at 25 °C.	
	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS;
Not stated	Not stated
	ESTIMATED ERROR:
	REFERENCES :
L	

210 N,	N-Dimethylformamide
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium thiocyanate; KCNS;[333 (2) N,N-dimethylformamide; C ₃ H ₇ NO;[6	
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	40.1
35	29.8
45	27.9
Al	UXILIARY INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of potassium thic were prepared by adding excess of the powdered salt to N,N-dimethylformamic (15-20 g) in Pyrex tubes (15 x 2.5 cr and preheating to 10 °C above the rec temperature of the bath prior to find thermostatting. The attainment of the equilibrium was checked by intermitte analyses. The solutions were then funder nitrogen and analysed for halid details of analytical methods were gis Transference of material, as far as p was carried out in a nitrogen filled Measurements were made in triplicated	SOURCE AND PURITY OF MATERIALS: A.R. grade potassium thiocyanate (BDH) was recrystallised and dried under vacuum at 110-125 °C. N,N-dimethylformamide (Riedel Pure) was dried over A.R. anhydrous sodium carbonate al for 24 hours before distilling under reduced pressure. The fraction boiling at 73 °C/ 57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ; specific conductance 2.5 x 10 ⁻⁵ S m ⁻¹ at 25 °C. The solvent was freshly distilled possible, for each experiment. dry box.

COMPONENTS:	ORIGINAL MEASUREMENTS:
 Potassium thiocyanate; KCNS; [333-20-0] N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Paul, R.C.; Sreenathan, B.R. Indian J. Chem., <u>1966</u> , 4, 382-386.
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of potassium thiocyanate in 16.0 g/100 g solve	-
	THEODIA
METHOD/APPARATUS/PROCEDURE:	INFORMATION
Saturated solutions of potassium thiocyanate were prepared by adding the salt in small quantities to 10 cm^3 of N,N-dimethylformamide contained in Pyrex test tubes, until an appreciable amount remained undissolved. The tubes were then sealed and fixed to a shaft immersed and arranged to rotate in a thermostatted water-bath at 25 °C. The temperature was maintained using a toluene regulator. After 24-30 hours of thorough mixing the seal was broken in a dry box and the slurry rapidly filtered under dry	ace for 40 hours with occasional shaking, the
^{Conditions.} From a known quantity of the ^{Solution} the metal was estimated and the	ESTIMATED ERROR:
solubility of the salt determined. No details of the analytical methods used were given.	Solubility ⁺ 1% Temperature ⁺ 0.1 ^O C (author)
"Sie given.	REFERENCES :

	EVALUATOR:	
 (1) Caesium chloride; CsCl; [7647-17-8] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] (3) C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland. December 1978. 	lformamide; C _{3H7} NO;[68-12-2] University of St. Andrews, St. Andrews, Fife, Scotland.	

CRITICAL EVALUATION:

Two investigations of the solubility of CsCl in N,N-dimethylformamide (DMF) have been reported (1-2). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (3) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. The most anhydrous solvent used in these studies of CsCl solubility is likely to have been that of Criss and Luksha (2) who found a water content of $\leq 0.002\%$ by Karl Fischer titration. The other major difficulty which may arise in solubility measurements in DMF, namely solvate formation, was shown to be absent in this particular system.

Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by Alexander et al. (1) with the molal solubility of Criss and Luksha (2). However the two results would seem to be consistent. The recommended value is taken to be that of the latter authors who are known to have used particularly pure solvent.

Recommended value at 298 K

0.52 ⁺ 0.01 g/kg of solvent.

- Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., <u>1967</u>, 89, 3703-3712.
- 2. Criss, C.M.; Luksha, E. J. Phys. Chem., 1968, 72, 2966-2970.
- 3. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Caesium chloride; CsCl; [7647-17-8]	Criss, C.M.; Luksha, E.
 (2) N,N-dimethylformamide; C₃H₇NO;[68-12-2] 	J. Phys. Chem., <u>1968</u> , 72, 2966-2970.
3, 1	
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
one temperature: 25 C	J.J. MCRECHITE
EXPERIMENTAL VALUES:	
Solubility of caesium chloride in N,N-di	methylformamide at 25 °C
0.052 g/100 g solvent	
Value calculated from solubility date gi	ven as:
$3.06 \div 0.02 \times 10^{-3} \text{ mol/kg}$	3 N,N-dimethylformamide
, ·	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of caesium chloride were Prepared in special Pyrex glass cells, 18 cm	pure) was used without further purification,
long and 4.5 cm in diameter. The cells were drawn out at the top in order that they could	except for the removal of water by drying in
be fitted with rubber serum caps. They were	
supported in a water bath by a rocker which oscillated 200 times/minute through an arc	The salt was stored at 400 °C until
of about 30°. Solubilities were obtained by approaching	ready for use. (continued)
equilibrium from both under- and supersatur- ation. In both approaches solutions were	(continued)
analysed daily until no further concentration	
changes could be detected. The solutions were analysed either by a flame photometer	ESTIMATED ERROR:
or by carefully evaporating to dryness and weighing the residue. In cases where both	Solubility + 1%
techniques were employed, the agreement was within 3%.	Temperature ⁺ 0.05 ^o C (author)
	REFERENCES :
	(1) Prue, J.E.; Sherrington, P.J.
	Trans. Faraday Soc., <u>1961</u> , 57, 1795-1808.
	(continued)

Caesium chloride; CsC1; [7647-17-8]
 N,N-dimethylformamide; C₃H₇NO; [68-12-2]

ORIGINAL MEASUREMENTS: Criss, C.M.; Luksha, E. *J. Phys. Chem.*, <u>1968</u>, 72, 2966-2970.

SOURCE AND PURITY OF MATERIALS: (continuation)

N,N-dimethylformamide was purified by treatment with calcium hydride accompanied by vigorous stirring and then distilling under reduced pressure through a 35 cm Vigreux column. The boiling point was 68 °C at 53 mm pressure. The reported value is 79 °C at 61-62 mm (1). The specific conductance was in the range $0.32 - 1.5 \times 10^{-5} \text{ S m}^{-1}$, which is in good agreement with the lowest values reported in the literature $(0.6 - 18.3 \times 10^{-5} \text{ S m}^{-1} (1), (2))$. There was no noticeable increase in conductivity with time.

Karl Fischer titrations indicated the water content to be less than 0.002%.

REFERENCES: (continuation)

(2) Ames, D.P.; Sears, P.G. J. Phys. Chem., 1955, 59, 16-19.

N, N-Dimethylformamide		21
COMPONENTS:	ORIGINAL MEASUREMENTS:	
 (1) Caesium chloride; CsC1; [7647-17-8] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. <i>J. Am. Chem. Soc.</i> , <u>1967</u> , 89 3703-3712.	2
VARIABLES:	PREPARED BY:	
One temperature: 25 ^O C	J.S. McKechnie	
EXPERIMENTAL VALUES:		
0.60 g/1000 cm ³ of satur Value calculated from solubility pr log(K _s o/mol dm ⁻³) = -4.9	oduct data given as:	
AUXILIARY	INFORMATION	

kept under dry nitrogen during preparation and titration. The usual procedure was to titrate 0.01 mol dm AgNO₃ solution into a half-cell containing the silver wire immersed in 20 cm of 0.01 mol dm⁻³ caesium chloride in DMF. The reference half-cell was a silver wire in 0.01 mol dm⁻³ AgNO₃ in the same solvent.

REFERENCES:

COMPONENTS:		EVALUATOR:
(1) Caesium bromide; CsBr; (2) N,N-dimethylformamide;	C_H_NO;[68-12-2]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1979

CRITICAL EVALUATION:

Two investigations of the solubility of CsBr in N,N-dimethylformamide (DMF) have been reported (1-2). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (3) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. The most anhydrous solvent used in these studies of CsBr solubility is likely to have been that of Criss and Luksha (2) who found a water content of $\leq 0.002\%$ by Karl Fischer titration. The other major difficulty which may arise in solubility measurements in DMF, namely solvate formation, was shown to be absent in this particular system.

Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by Alexander et al. (1) with the molal solubility of Criss and Luksha (2). However the two results would seem to be consistent. The recommended value is taken to be that of the latter authors who are known to have used particularly pure solvent.

Recommended value at 298 K

5.58 [±] 0.20 g/kg of solvent.

- Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., 1967, 89, 3703-3712.
- 2. Criss, C.M.; Luksha, E. J. Phys. Chem., 1968, 72, 2966-2970.
- 3. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

/v,/v-Dimetri	yiformamide 217
COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Caesium bromide; CsBr; [7787-69-1] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Criss, C.M.; Luksha, E. J. Phys. Chem., <u>1968</u> , 72, 2966-2970.
	<u></u>
VARIABLES: One temperature: 25 ^o C	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of caesium bromide in N,N-di	methylformamide at 25 ^O C
0.56 g/100 g solvent	
Value calculated from solubility data given as:	
$2.62 \pm 0.01 \times 10^{-2} \text{ mol/kg}$	N,N-dimethylformamide
	·····
	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of caesium bromide were	SOURCE AND PURITY OF MATERIALS:
prepared in special Pyrex glass cells, 18 cm long and 4.5 cm in diameter. The cells were drawn out at the top in order that they could be fitted with rubber serum caps. They were supported in a water bath by a rocker	Caesium bromide (Henley and Co., 99.9% pure) was used without further purification, except that it was dried by heating at 400 °C.
which oscillated 200 times/minute through an arc of about 30° Solubilities were obtained by approaching equilibrium from both under- and supersatur- ation. In both approaches solutions were	(continued)
analysed daily until no further concentration changes could be detected. The solutions	
were analysed either by a flame photometer or by carefully evaporating to dryness and	ESTIMATED ERROR: Solubility [±] 1%
Weighing the residue. In cases where both techniques were employed, the agreement was	Temperature ⁺ 0.05 ^o C
Within 3%.	REFERENCES:
	(1) Prue, J.E.; Sherrington, P.J.
	Trans. Faraday Soc., <u>1961</u> , 57, 1795-1808
	(continued)

COMPONENTS:

(1) Caesium bromide; CsBr; [7787-69-1]
 (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Criss, C.M.; Luksha, E. J. Phys. Chem., 1968, 72, 2966-2970.

SOURCE AND PURITY OF MATERIALS: (continuation)

N,N-dimethylformamide was purified by treatment with calcium hydride accompanied by vigorous stirring and then distilling under reduced pressure through a 35 cm Vigreux column. The boiling point was 68 °C at 53 mm pressure. The reported value is $79 \stackrel{0}{}^{\circ}C$ at 61-62 mm (1). The specific conductance was in the range $0.32 - 1.5 \times 10^{-5} \text{ sm}^{-1}$, which is in good agreement with the lowest values reported in the literature $(0.6 - 18.3 \times 10^{-5} \text{ sm}^{-1} (1), (2))$. There was no noticeable increase in conductivity with time.

Karl Fischer titrations indicated the water content to be less than 0.002%.

REFERENCES: (continuation)

(2) Ames, D.P.; Sears, P.G. J. Phys. Chem., <u>1955</u>,59, 16-19.

N, N-Dimethylformamide		219
COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Caesium bromide; CsBr; [7787-69-1] (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. <i>J. Am. Chem. Soc.</i> , <u>1967</u> , 89, 3703-3712.	
VARIABLES:	PREPARED BY:	
One temperature: 25 ^O C	J.S. McKechnie	
Value calculated from solubil: log(K _s o/mol dm ⁻³) =		

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of caesium bromide were prepared by shaking the solid with solvent in a stoppered flask at 35 °C for 24 hours. The flask was then shaken for a further 24 hours at 25 °C. The liquid phase was analysed for halide potentiometrically using silver con- centration cells with a tetraethylammonium picrate salt bridge. The cells were wrapped in aluminium foil and immersed in a 25 °C thermostat and stirred magnetically, as standard 0.01 mol dm ⁻³ silver nitrate was added. The emf was measured on a Radiometer PH meter (type PHM 22r). It was established early in the work that effectively the same results were obtained no matter whether meas- urements were made in the dark on freshly prepared solutions, on solutions exposed to the atmosphere for 30 minutes, or on solutions kept under dry nitrogen during preparation and titration. The usual procedure was to titrate 0.01 mol dm AgNO ₃ solution into a half-cell gontain- ing the sflyer wire immersed in 20 cm of 0.01 mol dm caesium bromide in DMF. The reference half-cell was a silver wire in 0.01 mol dm AgNO ₃ in the same solvent.	SOURCE AND PURITY OF MATERIALS: A.R. grade caesium bromide was used without further purification. DMF was dried with Type 4A molecular sieves and fractionated twice under a reduced pressure of dry nitrogen. It was stored over molecular sieves in dark bottles and was used within 7 days of purification. The stock solution of 0.01 mol dm ⁻³ AgNO for the potentiometric titrations was also ³ stored in a light proof container. ESTIMATED ERROR: Solubility [±] 2% Temperature [±] 0.1 ^o C (compiler) REFERENCES:

COMPONENTS :	EVALUATOR:
 (1) Caesium iodide; CsI; [7789-17-5] (2) N, N-dimethylformamide; C₃H₇NO; [68-12-2 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of CsI in N,N-dimethylformamide (DMF) has been reported (1). This solvent is readily prepared free from ionic impurities by vacuum distillation. However as Thomas and Rochow (2) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. In this study no analysis of the solid phase in contact with the solution is reported, so it is not possible to exclude the possibility of solvate formation.

Tentative value at 298 K

 $36.7 \stackrel{+}{-} 4.0 \text{ g/dm}^3$ of saturated solution.

- Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. J. Am. Chem. Soc., 1967, 89, 3703-3712.
- 2. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Caesium iodide; CsI; [7789-17-5]	Alexander, R.; Ko, E.C.F.; Mac, Y.C.;	
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]		
	3703-3712.	
VARIABLES:	PREPARED BY:	
One temperature: 25 °C	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of caesium iodide in N,N-dimethylformamide at 25 $^{\circ}$ C 36.70 g/1000 cm ³ of saturated solution		
Value calculated from solubility	product data given as:	
$\log(\kappa_o/mol \ dm^{-3}) = -1.$		
AUXILIARY	INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Saturated solutions of caesium iodide were	A.R. grade caesium iodide was used without	
prepared by shaking the solid with solvent in a stoppered flask at 35 °C for 24 hours. The	DMF was dried with Type 4A molecular	
flask was then shaken for a further 24 hours at 25 °C. The liquid phase was analysed for	sieves and fractionated twice under a reduced	
halide potentiometrically using silver con-	pressure of dry nitrogen. It was stored over molecular sieves in dark bottles and was used	
centration cells with a tetraethylammonium	within 7 days of purification.	
Picrate salt bridge. The cells were wrapped in aluminium foil and immersed in a 25 °C	The stock solution of 0.01 mol dm AgNO for the potentiometric titrations was also ³	
thermostat and stirred magnetically, as standard 0.01 mol dm ⁻³ silver nitrate was	stored in a light proof container.	
added. The emf was measured on a Radiometer		
PH meter (type PHM 22r). It was estabilshed early in the work that effectively the same	ESTIMATED ERROR:	
results were obtained no matter whether	Solubility ± 2%	
measurements were made in the dark on freshly prepared solutions, on solutions exposed to	Temperature ⁺ 0.1 ^o C (compiler)	
the atmosphere for 30 minutes, or on	DEFEDENCES.	
solutions kept under dry nitrogen during Preparation and titration.	REFERENCES :	
The usual procedure was to titrate 0.01 mol		
dm ⁻³ AgNO ₃ solution into a half-cell contain- ing the silver wire immersed in 20 cm ³ of		
0.01 mol dm ⁻³ caesium iodide in DNF.		
The reference half-cell was a silver wire in 0.01 mol dm ⁻³ AgNO ₃ in the same solvent.		
3		

COMPONENTS:	EVALUATOR:
 (1) Ammonium chloride; NH₄C1; [12125-02-9] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland.
	December 1978.

CRITICAL EVALUATION:

Two investigations of the solubility of NH_4C1 in N,N-dimethylformamide (DMF) have been reported (1-2). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (3). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (4) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

In the case of NH_4Cl , the possible complication of solvate formation was excluded by Paul and Sreenathan (1) who analysed the solid phase in equilibrium with the solution. However the results of these solubility studies show little agreement, falling within a wide range of values (0.5 - 3.7 g/kg of solvent). No explanation of such inconsistent results can be offered and no recommended value is given.

- 1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- 3. "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.
- 4. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

	,
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ammonium chloride; NH ₄ C1;[12125-02-9]	Properties and uses of DIMETHYLFORMAMIDE
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	(DMF), Du Pont Information Booklet, 1976.
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	
one temperature: 25 C	J.S. McKechnie
EXPERIMENTAL VALUES:	I,,
Solubility of ammonium chloride in N,N	-dimethylformamide at 25 °C
	dimethyllolmamide at 25 0
0.1 g/100 g solvent	
Data taken from table of semi-quantit	ative solubilities of inorganic
materials in DMF at 25 ^O C	
AIIXILIARY	INFORMATION
METHOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
	SOUND AND FORTH OF PATERIALS;
Not stated	Not stated
	ESTIMATED ERROR:
	REFERENCES:
	1

4 <i>N,N</i> -Dimethylformamide	
COMPONENTS :	ORIGINAL MEASUREMENTS:
<pre>(1) Ammonium chloride; NH₄C1; [12125-02-9] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2]</pre>	
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	0.37
35	0.30
45	0.28
AUXILIA	RY INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium chloride we prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of th bath prior to final thermostatting. The attainment of the equilibrium was checked b intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	SOURCE AND PURITY CF MATERIALS: re A.R. grade ammonium chloride (BDH) was recrystallised and dried under vacuum at 110-125 °C. N,N-dimethylformamide (Riedel Pure) was e dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced y pressure. The fraction boiling at 73 °C/

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ammonium chloride; NH ₄ C1; [12125-02-9]	Paul, R.C.: Sreenathan, B.R.
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	<i>inatan 0</i> , <i>chem</i> , <u>1700</u> , 1 , 562-566.
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
one temperature. 25 C	J.J. Ackechnie
EXPERIMENTAL VALUES:	
Solubility of ammonium chloride in	N,N-dimethylformamide at 25 °C
0.05 g/100 g so	lvent
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of ammonium chloride were	Purity of ammonium chloride was not stated.
Prepared by adding the salt in small	N,N-dimethylformamide (J.T. Baker analysed) was stored over A.R. anhydrous sodium carbon-
Quantities to 10 cm^3 of N,N-dimethylformamide Contained in Pyrex test tubes, until an	ate for 48 hours with occasional shaking.
appreciable amount remained undissolved.	The solvent was then decanted and distilled through a short column. The middle fraction
The tubes were then sealed and fixed to a shaft immersed and arranged to rotate in a	distilling at 149-151 °C/745 mm was collected
thermostatted water-bath at 25 °C. The	and redistilled under a reduced pressure of 35 mm in an atmosphere of dry nitrogen. The
temperature was maintained using a toluene regulator. After 24-30 hours of thorough	specific conductance of this N,N-dimethyl-
mixing the seal was broken in a dry box and	formamide measured 3 x 10^{-5} S m ⁻¹ at 25 °C.
the slurry rapidly filtered under dry Conditions. From a known quantity of the	ESTIMATED EDDOR.
Solution the metal was estimated and the	ESTIMATED ERROR:
^{solubility} of the salt determined. No	Solubility $\frac{1}{2}$ 10%
details of the analytical methods used were given.	Temperature ⁺ 0.1 [°] C (author)
	REFERENCES :
	4

COMPONENTS:	EVALUATOR:
(1) Ammonium bromide; NH ₄ Br; [12124-97-9] (2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland.
	December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of NH_4Br in N,N-dimethylformamide (DMF) has been reported (1). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (2). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (3) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂0. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

Agreement between the two reported solubilities is rather poor. In neither case are details given of the analytical methods used. While it might be argued that more weight should be given to the results of Paul et al., it should be noted that solubilities in DMF determined by this group are almost always higher than those found by other workers. In the case of NH₄Br it has therefore been decided to recommend as a tentative value the mean between the two reported solubilities.

Tentative value at 298 K

147 $\frac{+}{-}$ 20 g/kg of solvent.

- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Naruła, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.
- "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co. (Inc.), Wilmington, Delaware, U.S.A.
- 3. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

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COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Ammonium bromide; NH ₄ Br; [12124-97-9]	Properties and uses of DIMETHYLFORMAMIDE	
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	(DMF), Du Pont Information Booklet, 1976.	
VARIABLES:	PREPARED BY:	
One temperature: 25 °C	J.S. McKechnie	
<u></u>	L	
EXPERIMENTAL VALUES:		
Solubility of ammonium bromide in N,N	-dimethylformamide at 25 °C	
12.7 g/100 g solvent (1)		
Data taken from table of semi-quantit	ative solubilities of inorganic	
materials in DMF at 25 °C.		
materials in DMF at 25 C.		
AUXILIARY	INFORMATION	
ME THOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:	
Not stated	Not stated	
	ESTIMATED ERROR:	
	REFERENCES:	
	(1) Jones, G.D. British Patent 654,855,	
	July 4, 1951.	

228 <i>N, N</i> -Dimeth	
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Ammonium bromide; NH ₄ Br; [12124-97-9]	Paul, R.C.; Singla, J.P.; Lamba, M.S.;
(2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	Gill, D.S.; Narula, S.P. Indian J. Chem.,
	1973, 11, 1024-1026.
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
	<u> </u>
EXPERIMENTAL VALUES:	
t/ [°] C	
	g/100 g solvent
25	16.6
35	15.7 13.8
45	T3.0
AUXILIARY	INFORMATION
ME THOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of ammonium bromide were	A.R. grade ammonium bromide (BDH) was
prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in	recrystallised and dried under vacuum at 110-125 °C.
Pyrex tubes (15 x 2.5 cm) and preheating to	N,N-dimethylformamide (Riedel Pure) was
10 °C above the required temperature of the bath prior to final thermostatting. The	dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduced
attainment of the equilibrium was checked	pressure. The fraction boiling at 73 °C/
by intermittent analyses. The solutions were then filtered under nitrogen and	57 mm was collected and had the following physical constants: density 0,9442 g/cm ³ ;
analysed for halide. No details of	specific conductance 2.5 x 10^{-5} S m ⁻¹ at
analytical methods were given. Transference of material, as far as possible, was carried	25 ^o C. The solvent was freshly distilled for each experiment.
out in a nitrogen filled dry box.	ESTIMATED ERROR:
Measurements were made in triplicate.	Solubility [±] 1%
	Temperature $\stackrel{+}{=} 0.05 ^{\circ}C$ (author)
	Temperature - 0.05 C (author)
	REFERENCES :

COMPONENTS:	EVALUATOR:
<pre>(1) Ammonium iodide; NH₄I; [12027-06-4] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2]</pre>	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of $\mathrm{NH}_4\mathrm{I}$ in N,N-dimethylformamide (DMF) has been reported (1). This solvent is readily prepared free from ionic impurities by vacuum distillation and the conductance of the solvent was below 3 x 10⁻⁵ S m⁻¹ in this study. However as Thomas and Rochow (2) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. In this study no analysis of the solid phase in contact with the solution is reported, so it is not possible to exclude the possibility of solvate formation.

Tentative values

T/K Solubility (g/kg of solvent) 298 459 + 5 308 617 + 6 318 820 + 8

References

 Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., <u>1973</u>, 11, 1024-1026.

2. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

30 N, N-Dimeth	iyitormamide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Ammonium iodide; NH ₄ I; [12027-06-4] (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	I
t/ [°] C	g/100 g solvent
25	45.9
35	61.7
45	82.0
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Saturated solutions of ammonium iodide were prepared by adding excess of the powdered salt to N,N-dimethylformamide (15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 °C above the required temperature of the bath prior to final thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No details of analytical methods were given. Transference of material, as far as possible, was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	

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COMPONENTS :	EVALUATOR:
 (1) Ammonium thiocyanate; NH₄CNS; [1762-95-4] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife. Scotland. December 1978.

CRITICAL EVALUATION:

Two investigations of the solubility of NH_4CNS in N,N-dimethylformamide (DMF) have been reported (1-2). In addition results from a 'semiquantitative' study by the Industrial Chemicals Department of E.I. duPont de Nemours and Co. (Inc.) have been published (3). This solvent is readily prepared free from ionic impurities by vacuum distillation, and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in all the investigations. However, as Thomas and Rochow (4) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂0. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

A major problem in the determination of the solubility of NH₄CNS in this solvent is that the true equilibrium state of the solid phase is almost certainly a solvate. Paul and Sreenathan (1) isolated a colourless solvate of composition NH₄CNS.DMF (S: found 21.04%, required 21.44%). However it is not clear from their results whether the solution was in equilibrium with the solvate or whether both solvate and salt solid phases were present. None of the other authors considered the possibility of solvate formation. However the solubilities reported by two of the groups show good consistency and the mean of these two results is recommended as a tentative value.

Tentative value at 298 K

 $154 \stackrel{+}{-} 2 \text{ g/kg of solvent.}$

- 1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.
- Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. Indian J. Chem., 1973, 11, 1024-1026.
- 3. "Properties and Uses of Dimethylformamide", E.I. duPont de Nemours and Co., (Inc.), Wilmington, Delaware, U.S.A.
- 4. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

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COMPONENTS :	ORIGINAL MEASUREMENTS:
 (1) Ammonium thiocyanate; NH₄CNS; [1762-95-4] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of ammonium thiocyanate in	N,N-dimethylformamide at 25 [°] C
15.2 g/100 g solvent	
Data taken from table of semi-quantita	tive solubilities of inorganic
materials in DMF at 25 ^o C	
1	
	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Not stated	Not stated
	ESTIMATED ERROR:
	REFERENCES :
	1

N, N-Dimethy	/lformamide 2
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Ammonium thiocyanate; NH₄CNS; [1762-95-4] N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.C.; Narula, S.P. Indian J. Chem.,
	<u>1973, 11, 1024-1026.</u>
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie
EXPERIMENTAL VALUES:	
t/ ^o C	g/100 g solvent
25	
35	17.0
45	18.6
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of ammonium thiocyanate were prepared by adding excess of the powdered salt to N,N-dimethylformamide	A.R. grade ammonium thiocyanate (BDH) wa recrystallised and dried under vacuum at 110-125 °C.
(15-20 g) in Pyrex tubes (15 x 2.5 cm) and preheating to 10 $^{\circ}$ C above the required temperature of the bath prior to final	N,N-dimethylformamide (Riedel Pure) was dried over A.R. anhydrous sodium carbonate for 24 hours before distilling under reduce
thermostatting. The attainment of the equilibrium was checked by intermittent analyses. The solutions were then filtered under nitrogen and analysed for halide. No	pressure. The fraction boiling at 73 °C/ 57 mm was collected and had the following physical constants: density 0.9442 g/cm ³ ; specific conductance 2.5 x 10^{-5} S m ⁻¹ at
details of analytical methods were given. Iransference of material, as far as possible,	25 °C. The solvent was freshly distilled
was carried out in a nitrogen filled dry box. Measurements were made in triplicate.	ESTIMATED ERROR: Solubility + 1%
	Temperature $\stackrel{+}{=} 0.05 ^{\circ}C$ (author)
	REFERENCES:

COMPONENTS: (1) Magnesium chloride; MgC1 ₂ ; [7786-30-3] (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	EVALUATOR: C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland.
	December 1978.

Two investigations of the solubility of $MgCl_2$ in N,N-dimethylformamide (DMF) have been reported (1-2). This solvent is readily prepared free from ionic impurities by vacuum distillation and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in both investigations. However, as Thomas and Rochow (3) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF. $2H_2O$. These last authors examined a number of chemical treatments, but concluded that some water always remained in the solvent.

A major problem in the determination of the solubility of MgCl₂ in this solvent is that the equilibrium state of the solid phase is almost certainly a solvate. Paul and Sreenathan (1) isolated solids of composition MgCl₂. 2 DMF and MgCl₂. DMF. However it is not clear from their results whether or not the solution was in equilibrium with these solvates. Such an uncertainty makes it impossible to recommend a value for the solubility of this salt.

References

1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.

- 2. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37 1167-1172.
- 3. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

6 N, N-Dimethylformamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Magnesium chloride; MgCl ₂ ; [7786-30-3] Pistoia. G.; Pecci, G.; Scrosati, B.
(2) N,N-dimethylformamide; $C_{3}H_{7}N0$; [68-12-	
VARIABLES:	PREPARED BY:
One temperature: 25 ^O C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of magnesium chloride i	n N,N-dimethylformamide at 25 ^O C
12.5 g/100 g solve	
AUXILIA	ARY INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Considerable effort was made to obtain wat	
free solutions and all the solutions were prepared in the dry box. Saturated	under vacuum before use. Reagent grade N,N~dimethylformamide
magnesium chloride solutions were prepared dissolving excess of the halide in about	by (C. Erba RP) was purified by fractional distillation under reduced pressure (2 mm Hg).
$20-25 \text{ cm}^3$ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint.	The final product had a specific conductance of 3×10^{-5} S m ⁻¹ .
The solution was then stirred for three or	
more days in a thermostatted bath. With the aid of a syphon provided with a termin	
G-3 gooch, an aliquot of the saturated solution was collected and weighed. This	
solution was then diluted with water (1:10) ESTIMATED ERROR:
and the amount of dissolved halide determi volumetrically by Fajan's titration using	50100111Cy = 1%
dichlorofluorescein as indicator. Equilibrium between solid and solution	Temperature ⁺ 0.05 ^o C (author)
phases was considered to be attained when analysis of the solution, made at progress	ive REFERENCES:
periods of time, gave the same value for	
dissolved halide.	

N, N-Dimethylformamide

COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Magnesium chloride; MgCl₂[7786-30-3] (2) N,N-dimethylformamide: C₃H₇NO;[68-12-2] 	Paul, R.C.; Sreenathan, B.R. Indian J. Chem., <u>1966</u> , 4, 382-386.
VARIABLES:	PREPARED BY:
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of magnesium chloride in N 8.04 g/100 g solven	
AUXILIARY	INFORMATION
METHOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of magnesium chloride were prepared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamide contained in Pyrex test tubes, until an appreciable amount remained undissolved. The tubes were then sealed and fixed to a shaft immersed and arranged to rotate in a thermostatted water-bath at 25 °C. The temperature was maintained using a toluene regulator. After 24-30 hours of thorough mixing the seal was broken in a dry box and the slurry rapidly filtered under dry conditions. From a known quantity of the	ate for 48 hours with occasional shaking. The solvent was then decanted and distilled through a short column. The middle fraction distilling at 149-151 °C/745 mm was collected and redistilled under a reduced pressure of 35 mm in an atmosphere of dry nitrogen. The specific conductance of this N,N-dimethy1- formamide measured 3 x 10 ⁻⁵ S m ⁻¹ at 25 °C.
solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.	ESTIMATED ERROR: Solubility ⁺ 1% Temperature ⁺ 0.1 ^o C (author) REFERENCES:

N, N-Dimethylformamide

COMPONENTS :	EVALUATOR:
(1) Magnesium iodide; MgI ₂ ; [10377-58-9] (2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife. Scotland. December 1978.

CRITICAL EVALUATION:

One investigation of the solubility of MgI₂ in N,N-dimethylformamide (DMF) has been reported (1). This solvent is readily prepared free from ionic impurities by vacuum distillation and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in this study. However as Thomas and Rochow (2) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. In this study no analysis of the solid phase in contact with the solution is reported, so it is not possible to exclude the possibility of solvate formation.

Tentative value at 298 K

1190 [±] 20 g/kg of solvent.

References

- 1. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.
- 2. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

N,N-Dimeth	vitormamide 23s
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Magnesium iodide; MgI ₂ ;[10377-58-9] (2) N,N-dimethylformamide; C ₃ H ₇ NO;[68-12-2]	Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1167-1172
VARIABLES:	
One temperature: 25 °C	PREPARED BY: J.S. McKechnie
EXPERIMENTAL VALUES:	
Solubility of magnesium iodide in N,N-	dimethylformamide at 25 ^O C
119 g/100 g solvent	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Considerable effort was made to obtain water free solutions and all the solutions were prepared in the dry box. Saturated magnesium iodide solutions were prepared by dissolving excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was collected and weighed. This solution was then diluted with water (1:10) and the amount of dissolved halide determined volumetrically by adding an excess of standard silver nitrate and back titrating against standard ammonium thiocyanate using ferric alum as indicator. Equilibrium between solid and solution phases was considered to be attained when analysis of the solution, made at progressive periods of time, gave the same value for dissolved halide.	ESTIMATED ERROR: Solubility ⁺ 1% Temperature ⁺ 0.05 [°] C (author) REFERENCES:

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COMPONENTS:	EVALUATOR:
<pre>(1) Calcium chloride; CaCl₂; [10043-52-4] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2]</pre>	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1978.

Two investigations of the solubility of $CaCl_2$ in N,N-dimethylformamide (DMF) have been reported (1-2). This solvent is readily prepared free from ionic impurities by vacuum distillation and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in both investigations. However, as Thomas and Rochow have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF. $2H_2O$. These last authors examined a number of chemical treatments but concluded that some water always remained in the solvent.

A major problem in the determination of the solubility of CaCl₂ in this solvent is that the equilibrium state of the solid phase is almost certainly a solvate. Paul and Sreenathan (1) isolated a solid of composition CaCl₂. 2 DMF. However it is not clear from their results whether or not the solution was in equilibrium with this solvate. Under these circumstances it is not possible to recommend a value for the solubility of this salt.

References

1. Paul, R.C.; Sreenathan, B.R. Indian J. Chem., 1966, 4, 382-386.

- 2. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.
- 3. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

COMPONENTS:	ORIGINAL MEASUREMENTS:
 Calcium chloride; CaCl₂; [10043-52-4] N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1167-1172
VARIABLES:	PREPARED BY:
One temperature: 25 ⁰ C	J.S. McKechnie
EXPERIMENTAL VALUES:	·J
Solubility of calcium chlor	ride in N,N-dimethylformamide at 25 ^O C

6.01 g/100 g solvent

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Considerable effort was made to obtain water free solutions and all the solutions were Prepared in the dry box. Saturated calcium chloride solutions were prepared by dissolv- ing excess of the halide in about 20-25 cm ³ of solvent contained in a 50 cm ³ Pyrex flask With a standard taper joint. The solution Was then stirred for three or more days in a thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch, an aliquot of the saturated solution was collected and weighed. This solution was then diluted with water (1:10) and the amount of dissolved halide determined volumetrically by Fajan's titration using dichlorofluore- scein as indicator. Equilibrium between solid and solution	SOURCE AND PURITY OF MATERIALS: Calcium .chloride, reagent grade, was dried under vacuum before use. Reagent grade N,N-dimethylformamide (C. Erba RP) was purified by fractional distillation under reduced pressure (2 mm Hg). The final product had a specific conductance of 3×10^{-5} S m ⁻¹ . ESTIMATED ERROR: Solubility $\frac{+}{-}$ 1% Temperature $\frac{+}{-}$ 0.05 °C (author)
phases was considered to be attained when analysis of the solution, made at progressive Periods of time, gave the same value for dissolved halide.	REFERENCES :

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Calcium chloride; CaCl ₂ ; [10043-52-4]	Paul, R.C.; Sreenathan, B.R.,
(2) N,N-dimethylformamide; C ₃ H ₇ NO; [68-12-2]	Indian J. Chem., <u>1966</u> , 4, 382–386
VARIABLES:	PREPARED BY:
0	
One temperature: 25 ^o C	J.S. McKechnie
EXPERIMENTAL VALUES:	
	0
Solubility of calcium chloride in N	
1.98 g/100 g solvent	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions of calcium chloride were	Purity of calcium chloride was not stated.
prepared by adding the salt in small quantities to 10 cm ³ of N,N-dimethylformamide	N,N-dimethylformamide (J.T.Baker analysed) was stored over A.R. anhydrous sodium carbon-
contained in Pyrex test tubes, until an	ate for 48 hours with occasional shaking.
appreciable amount remained undissolved. The tubes were then sealed and fixed to a	The solvent was then decanted and distilled through a short column. The middle fraction
shaft immersed and arranged to rotate in a	distilling at 149-151 °C/745 mm was collected
thermostatted water-bath at 25 °C. The temperature was maintained using a toluene	and redistilled under a reduced pressure of 35 mm in an atmosphere of dry nitrogen. The
regulator. After 24-30 hours of thorough	specific conductance of this N,N-dimethyl-
mixing the seal was broken in a dry box and the slurry rapidly filtered under dry	formamide measured 3 x 10^{-5} S m ⁻¹ at 25 °C.
conditions. From a known quantity of the	ESTIMATED ERROR:
solution the metal was estimated and the solubility of the salt determined. No	Solubility ⁺ 1%
details of the analytical methods used were	Temperature $+ 0.1 °C$ (author)
given.	
	REFERENCES :

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COMPONENTS:	EVALUATOR:
<pre>(1) Calcium Bromide; CaBr₂; [7789-41-5] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2]</pre>	St. Andrews, Fife, Scotland.
	December 1978.

One investigation of the solubility of CaBr_2 in N,N-dimethylformamide (DMF) has been reported (1). This solvent is readily prepared free from ionic impurities by vacuum distillation and the conductance of the solvent was below 3×10^{-5} S m⁻¹ in this study. However as Thomas and Rochow (2) have pointed out, distillation is an ineffective method for removing water from DMF, mainly because of the existence of aquo-complexes such as DMF.2H₂O. The latter authors examined a number of chemical treatments, but concluded that some water always remained in the solvent. In this study no analysis of the solid phase in contact with the solution is reported, so it is not possible to exclude the possibility of solvate formation.

Tentative value at 298 K

 $192 \stackrel{+}{=} 2 \text{ g/kg of solvent.}$

References

1. Pistoia, G.; Pecci, G.; Scrosati, B. Ric. Sci., 1967, 37, 1167-1172.

2. Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc., 1957, 79, 1843-1848.

COMPONENTS:	ORIGINAL MEASUREMENTS:	
 (1) Calcium bromide; CaBr₂; [7789-41-5] (2) N,N-dimethylformamide; C₃H₇NO; [68-12-2] 	Pistoia, G.; Pecci, G.; Scrosati, B. <i>Ric. Sci</i> ., <u>1967</u> , <i>37</i> , 1167-1172	
VARIABLES:	PREPARED BY:	
One temperature: 25 [°] C	J.S. McKechnie	
EXPERIMENTAL VALUES:		
Solubility of calcium bromide in N,N-dimethylformamide at 25 $^{\circ}$ C		
19.2 g/100 g solvent		
AUXILIARY	INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
Considerable effort was made to obtain water	Calcium bromide, reagent grade, was dried under vacuum before use.	
free solutions and all the solutions were prepared in the dry box. Saturated calcium	Reagent grade N.N-dimethylformamide	
bromide solutions were prepared by dissolving excess of the halide in about 20-25 cm ³ of	distillation under reduced pressure (2 mm Hg).	
solvent contained in a 50 cm ³ Pyrex flask with a standard taper joint. The solution	The final product had a specific conductance of $3 \times 10^{-5} \text{ S m}^{-1}$.	
was then stirred for three or more days in a		
thermostatted bath. With the aid of a syphon provided with a terminal G-3 gooch,		
an aliquot of the saturated solution was collected and weighed. This solution was		
then diluted with water (1:10) and the amount of dissolved halide determined volumetrically	ESTIMATED ERROR:	
by Fajan's titration using tetrabromofluore-	Solubility ⁺ 1%	
scein as indicator. Equilibrium between solid and solution	Temperature ⁺ 0.05 ^o C (author)	
phases was considered to be attained when analysis of the solution, made at progressive	REFERENCES :	
periods of time, gave the same value for dissolved halide.		

COMPONENTS:	EVALUATOR:	
(1) Sodium iodide; NaI; 7681-82-5 (2) Acetamide; C ₂ H ₅ NO; 60-35-5	Bruno Scrosati Istituto di Chimica Fisica, University of Rome, Italy.	
	March 1979	

The sodium iodide - acetamide system has been investigated by Sarkisov et al (1) and by Menshutkin (2).Sarkisov et.al.(1) have reported an analytical study of the sodium iodide - potassium iodide - acetamide system, in the course of which the concentrations of saturated NaI solutions in acetamide were determined gravimetrically at three temperatures with the following results

T/K	g/kg of solution
343	340
353	359
363	372

It has not been possible for the evaluator to obtain a copy of the original paper of Menshutkin (2). However, his data have been listed in a reference book translated by Stephen and Stephen (3). Since the original reference is not available, the original results of Menshutkin are not evaluated nor compiled and here are reported (for sake of comparison) only the values at 343, 353 and 363 K as obtained from Stephen and Stephen's book. They are

т/к	g/kg of solvent
343	348
353	359
363	372

The agreement with the data of Sarkisov et al (1) is quite good. Nevertheless, the procedure used by Sarkisov et.al.to determine solubility does not appear very accurate and no information is available for that adopted by Menshutkin. Therefore no values of solubility of sodium iodide in acetamide are recommended.

References

- 1. Sarkisov, A.G.; Melamud, S.F.; Sakharova, N.F. Khimiya, 1969, 3 11.
- 2. Menshutkin, B.N. Izv. SPb, Politekh, Inst, 1908, 9, 200.
- 3. Stephen H.; Stephen T. Solubility of Inorganic and Organic Compounds, 1963, Pergammon Press, Oxford, Vol.1

Acetamice			
COMPONENTS:	ORIGINAL MEASUREMENTS:		
(1) Sodium iodide; NaI; 7681-82-5	Sarkisov, A.G; Melamud, S.F.;		
(2) Acetamide; C ₂ H ₅ NO; 60-35-5	Sakharova, N.F. Khimiya, 1969, 3-11		
2 5			
VARIABLES:	PREPARED BY:		
Temperatures	Bruno Scrosati		
EXPERIMENTAL VALUES:			
Solubility of sodium oidide at thre	e temperatures		
	•		
t/°C	g/100 g of solution		
70	34.0		
80	35.9		
90	37.2		
	INFORMATION		
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:		
Saturated solutions were prepared by	Anhydrous and chemically pure potassium		
dissolving the salt in acetamide in a vessel kept in a thermostatted oil bath. Samples	iodide was used. Acetamide was synthesised from acetic anhydride dissolved in ether and		
of the solution were withdrawn with a pipet	saturated with ammonia. Upon evaporation of		
and transferred to a crucible. After evaporation of the solvent the residue was	the solvent, white crystals of acetamide precipitated. The product was then		
weighed and the concentration determined.	recrystallised twice from benzene. Acetamide		
	so purified had a melting point of 82 ^o C, a value slightly higher than that reported		
	by Wallace (1)		
	ESTIMATED ERROR:		
	Solubility [±] 5% (compiler)		
	Comprises)		
]			
	REFERENCES:		
	1. Wallace, R.A. Inorg. Chem. <u>1972</u> ,		
	11(2), 414-415		
,			

Acetamide

246

COMPONENTS:	EVALUATOR:
(1) Potassium chloride, KC1, 7447-40-7 (2) Acetamide, C ₂ H ₅ NO, 60-35-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy
	March 1979

Very few data on the solubility of halides in acetamide are available in the literature. In particular, the potassium chloride-acetamide system has been examined only by Wallace (1).

The results appear very reliable since this author has carefully purified the salt (by crystallization and fusion) and the solvent (by crystallization) and controlled the temperature.

Nevertheless, because of the lack of data from other sources which may serve as terms of comparison and evaluation, the results of Wallace (1) may only be reported as tentative values of the solubility.

Tentative values of solubility of KCl as function of temperature

T/K	g/kg of solvent
353	24.5
359	25.2
376	25.8
388	26.2
393	26.8
403	27.4

References

1. Wallace, R.A. Inorg. Chem., 1972, 11(2), 414-415.

248 Acetamide			
COMPONENTS: ORIGINAL MEASUREMENTS:			
(1) Potassium chloride; KCl; 7447-40-7 (2) Acetamide; C ₂ H ₅ NO; 60-35-5	Wallace, R.A. Inorg. Chem., 1972, 11(2), 414-415.		
VARIABLES:	PREPARED BY:		
Temperature	Bruno Scrosati		
EXPERIMENTAL VALUES:	L		
Solubility of potassium chloride at	various temperatures		
t/ ^o C g/	100g of solvent		
80	2.45		
86	2.52		
103	2.58		
115	2.62		
120	2.68		
130	2.74		
AUXILIARY	INFORMATION		
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS;		
Purified acetamide containing an excess of potassium chloride was placed in a 500 cm ³ pyrex tube fitted with a glass stopper, a thermometer and a glass stirrer. The tube was heated to the desired temperature in a silicone oil bath and allowed to equili- brate with vigorous stirring. Aliquot portions of the supernatant liquid were drawn off with a pipet at the same temperat- ure and placed in a 250 cm ³ flask. The solution was then weighed, dissolved in water, and titrated using the Volhard	Analytical grade potassium chloride was recrystallized from distilled water and fused. Analytical grade acetamide was further purified by recrystallization in pure benzene. The solvent so purified had a melting point of 80.0-80.5°C and a mean specific conductance of 8.5x10 ⁻⁴ Sm ⁻¹ at 94°C.		
method.	ESTIMATED ERROR: Temperature ⁺ 0.1 ^o C (author) Solubility ⁺ 1% (compiler)		
	REFERENCES :		

COMPONENTS :		EVALUATOR:	
	(1) Potassium bromide; KBr; 7558-02-3 (2) Acetamide; C ₂ H ₅ NO; 60-35-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy	
		March 1979	

Very few data on the solubility of halides in acetamide are available in the literature. In particular, the potassium bromide-acetamide system has been examined only by Wallace (1).

The results appear very reliable since the author has carefully purified the salt (by crystallization and fusion) and the solvent (by crystallization) and controlled the temperature.

Nevertheless because of the lack of data from other sources which may serve as terms of comparison and evaluation, the results of Wallace (1) may only be reported as tentative values of the solubility.

Tentative values of solubility of KBr as function of temperature.

T/K	g/kg of solvent
354	103
363	105
373	108
383	110
393	112
403	113

References

1. Wallace, R.A., Inorg. Chem., 1972, 11(2), 414-415.

50 Acetamide		
COMPONENTS: ORIGINAL MEASUREMENTS:		
(1) Potassium bromide; KBr; 7558-02-3 (2) Acetamide; C ₂ H ₅ NO; 60-35-5	Wallace, R.A. Inorg. Chem., <u>1972</u> , 11(2), 414-415.	
VARIABLES:	PREPARED BY:	
Temperature	Bruno Scrosati	
-		
EXPERIMENTAL VALUES:		
Solubility of potassium bromi	de at various temperatures	
t/ ^o C	g/100 g of solvent	
81	10.3	
90	10.5	
100	10.8	
110	11.0	
120	11.2	
130	11.3	
equation log c (g/100g) = 9.40 x	$10^{-4}t + 0.937$	
-	10 ⁻⁴ t + 0.937	
log c (g/100g) = 9.40 x	10 ⁻⁴ t + 0.937 INFORMATION	
log c (g/100g) = 9.40 x AUXILIARY	INFORMATION	
log c (g/100g) = 9.40 x AUXILIARY METHOD/APPARATUS/PROCEDURE: Purified acetamide containing an excess of potassium bromide was placed in a 500 cm pyrex tube fitted with a glass stopper, a		
log c (g/100g) = 9.40 x AUXILIARY METHOD/APPARATUS/PROCEDURE: Purified acetamide containing an excess of potassium bromide was placed in a 500 cm	INFORMATION SOURCE AND PURITY OF MATERIALS: Analytical grade potassium bromide was recrystallized from distilled water and	
log c (g/100g) = 9.40 x AUXILIARY METHOD/APPARATUS/PROCEDURE: Purified acetamide containing an excess of potassium bromide was placed in a 500 cm pyrex tube fitted with a glass stopper, a thermometer and a glass stirrer. The tube was heated to the desired temperature in a silicone oil bath and allowed to equilibrate with vigorous stirring. Aliquot portions of the supernatant liquid were drawn off with a pipet at the same temperature and placed in a 250 cm flask. The solution was then weighed, dissolved in water, and	INFORMATION SOURCE AND PURITY OF MATERIALS: Analytical grade potassium bromide was recrystallized from distilled water and fused. Analytical grade acetamide was further purified by recrystallization in pure benzene. The solvent so purified had a melting point of 80.0-80.5°C and a mean specific conductance of 8.5x10 'Sm ⁻¹ at 94°C. ESTIMATED ERROR:	
Log c (g/100g) = 9.40 x AUXILIARY METHOD/APPARATUS/PROCEDURE: Purified acetamide containing an excess of potassium bromide was placed in a 500 cm pyrex tube fitted with a glass stopper, a thermometer and a glass stirrer. The tube was heated to the desired temperature in a silicone oil bath and allowed to equilibrate with vigorous stirring. Aliquot portions of the supernatant liquid were drawn off with a pipet at the same temperature and placed in a 250 cm flask. The solution was then weighed, dissolved in water, and	INFORMATION SOURCE AND PURITY OF MATERIALS: Analytical grade potassium bromide was recrystallized from distilled water and fused. Analytical grade acetamide was further purified by recrystallization in pure benzene. The solvent so purified had a melting point of 80.0-80.5°C and a mean specific conductance of 8.5x10 °Sm at 94°C. ESTIMATED ERROR: Temperature $\stackrel{+}{=}$ 0.1°C (author)	
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Log c (g/100g) = 9.40 x AUXILIARY METHOD/APPARATUS/PROCEDURE: Purified acetamide containing an excess of potassium bromide was placed in a 500 cm pyrex tube fitted with a glass stopper, a thermometer and a glass stirrer. The tube was heated to the desired temperature in a silicone oil bath and allowed to equilibrate with vigorous stirring. Aliquot portions of the supernatant liquid were drawn off with a pipet at the same temperature and placed in a 250 cm flask. The solution was then weighed, dissolved in water, and	INFORMATION SOURCE AND PURITY OF MATERIALS: Analytical grade potassium bromide was recrystallized from distilled water and fused. Analytical grade acetamide was further purified by recrystallization in pure benzene. The solvent so purified had a melting point of 80.0-80.5°C and a mean specific conductance of 8.5x10 ⁻⁴ Sm ⁻¹ at 94°C. ESTIMATED ERROR: Temperature [±] 0.1°C (author) Solubility [±] 1% (compiler)	
log c (g/100g) = 9.40 x AUXILIARY METHOD/APPARATUS/PROCEDURE: Purified acetamide containing an excess of potassium bromide was placed in a 500 cm pyrex tube fitted with a glass stopper, a thermometer and a glass stirrer. The tube was heated to the desired temperature in a silicone oil bath and allowed to equilibrate with vigorous stirring. Aliquot portions of the supernatant liquid were drawn off with a pipet at the same temperature and placed in a 250 cm flask. The solution was then weighed, dissolved in water, and	INFORMATION SOURCE AND PURITY OF MATERIALS: Analytical grade potassium bromide was recrystallized from distilled water and fused. Analytical grade acetamide was further purified by recrystallization in pure benzene. The solvent so purified had a melting point of 80.0-80.5°C and a mean specific conductance of 8.5x10 ⁻⁴ Sm ⁻¹ at 94°C. ESTIMATED ERROR: Temperature [±] 0.1°C (author) Solubility [±] 1% (compiler)	

COMPONENTS:	EVALUATOR:
(1) Potassium iodide; KI; 7681-11-0 (2) Acetamide; C ₂ H ₅ NO; 60-35-5	Bruno Scrosati Istituto di Chimica Fisica University of Rome, Italy

March, 1979

CRITICAL EVALUATION:

The potassium iodide-acetamide system has been investigated by Wallace (1), by Sarkisov et al. (2) and by Menshutkin (3).

Wallace has determined volumetrically the solubility at five temperatures i.e. 353, 358, 370, 391 and 403 K and the results seem reliable since the author has carefully purified the salt (by crystallization and fusion) and the solvent (by crystallization) and controlled the temperature of operation.

Sarkisov et al (2) have reported an analytical study of the sodium iodide-potassium iodide-acetamide system, in the course of which the concentrations of saturated NaI solutions in acetamide were determined gravimetrically at 343, 353 and 363 K.

The procedure for determining the solubility, however, does not appear very accurate and the data may be affected by an error which is estimated by the evaluator to be around 5%.

Considering this error, the values reported by Sarkisov et al (2) are in fair agreement with those obtained by Wallace (1).

It has not been possible for the evaluator to obtain a copy of the original paper by Menshutkin (3). However, his data have been listed in a reference book translated by Stephen and Stephen (4). Since the original reference is not available, the original results of Menshutkin (3) are not evaluated nor compiled and here are reported only the values at a few temperatures (as obtained from Stephen and Stephen's book) to compare them with those obtained by the other authors.

Wallace (1)		Sarkisov et al (2)		Menshutkin (4)	
T/K	g/kg of solvent	T/K	g/kg of solution	T/K	g/kg of solvent
		343	287	343	287
353	323	353	290		
358	325			358	291
363	327 (calc)	363	292		
373	329 (calc)			373	294

252 Ace	etamide
COMPONENTS:	EVALUATOR:
(1) Potassium iodide; KI; 7681-11-0 (2) Acetamide; C ₂ H ₅ NO; 60-35-5	Bruno Scrosati Istituto di Chimica Fisica University of Rome, Italy
	March 1979

CRITICAL EVALUATION: (Continuation):

The agreement between the data of Sarkisov et.al. (2) and those of Menshutkin (4) is quite good. However, since the procedure used by Sarkisov et.al.to determine solubility is not considered very accurate and no information is available for that adopted by Menshutkin (3), the results of Wallace (4) are here preferred to give a tentative indication of the solubility.

Tentative values of Solubility of KI as function of temperature

T/K	g/kg of solvent
353	323
358	325
370	328
391	332
403	336

References

- 1. Wallace, R.A. Inorg. Chem., 1972, 11(2), 414-415.
- 2. Sarkishov, A.G.; Melamed, S.Z.; Sakharova, N.F., Khimiya, 1969, 3-11.
- 3. Menshutkin, B.N., Izv. SPb. Politekh, Inst. 1908, 9, 200.
- 4. Stephen H.; Stephen T., Solubility of Inorganic and Organic Compounds, 1963, Pergammon Press, Oxford, Vol.1.

Ace	amide 2
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Potassium iodide; KI: 7681-11-0 Acetamide; C₂H₅NO; 60-35-5 	Wallace, R.A. Inorg.Chem., <u>1972</u> , 11(2), 414-415.
VARIABLES:	PREPARED BY:
Temperature	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of potassium iodide at va	rious temperatures
t/ ^o C	g/100 g of solvent
80	32.3
85	32.5
97	32.8
118	33.2
130	33.6
the following equation	
log c (g/100g) = 3.33	$\times 10^{-4} t + 1.484$
log c (g/100g) = 3.33	x 10 ⁻⁴ t + 1.484 INFORMATION SOURCE AND PURITY OF MATERIALS: Analytical grade potassium iodide was recrystallized from distilled water and

254 Aceta	amide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; 7681-11-0 (2) Acetamide; C ₂ H ₅ NO; 60-35-5	Sarkisov, A.G.; Melamud, S.F.; Sakharova, N.K. <i>Khimiya</i> , <u>1969</u> , 3-11.
VARIABLES:	PREPARED BY:
Temperature	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of potassium	iodide at three temperatures
t/°C	g/100 g of solution
70	28.7
80	29.0
90	29.2
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Saturated solutions were prepared by dissolving the salt in acetamide in a vessel kept in a thermostatted oil bath. Samples of the solution were withdrawn with a pipet and transferred in a crucible. After evaporation of the solvent the resid- ual was weighed and the concentration determined.	Anhydrous and chemically pure potassium iodide was used. Acetamide was synthesised from acetic anhydride dissolved in ether and saturated with ammonia. Upon solvent evaporation, white bright crystals of aceta- mide precipitated. The product was then recrystallized twice from benzene. Acetamide so purified had a melting point of 82°C, a value slightly higher than that reported by Wallace (1)
	ESTIMATED ERROR:
	Solubility <u>+</u> 5% (compiler)
	REFERENCES: 1. Wallace, R.A. Inorg.Chem., <u>1972</u> , 11(2), 414-415.

COMPONENTS: (1) Lithium chloride; LiC1; 7447-41-8 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	EVALUATOR: Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy.
	March 1979.

As far as the evaluator knows the lithium chloride-N-methylacetamide system has been investigated only by Dawson, Berger, Vaughn and Eckstrom (1).

These authors allowed long equilibration times to ensure saturation and carefully controlled the conditions of operation. The concentration of LiCl was then calculated from conductivity measurements as 17.51 g dm^{-3} of saturated solution at 313 K.

Even if the conductometric technique might be rigorously applied only to dilute solutions, this value appears acceptable as reliable indication of the solubility of LiC1 in N-methylacetamide.

Tentative value of solubility of LiCl at 313 K

17.51 g dm⁻³ of saturated solution

Since the density of the saturated solution is not known, it is not possible to convert the data to molality units.

References

 Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium chloride; LiC1; 7447-41-8 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283
VARIABLES:	PREPARED BY:
One temperature: 40°C	Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of lithium chloride at 40°C.

0.413 mol dm⁻³

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined by preparing solutions containing 25 cm of solvent and excess of solute in glass stoppered flasks. These were placed in an oven at 40°C for one week and shaken frequently during this time. The flasks were then transferred to a thermostatted bath and held there for an additional week with frequent shaking to ensure saturation. To determine the concentration of the solute, a weighted amount of the solution was withdrawn and transferred to a tared conductance cell containing a known amount of solvent. The cell was weighed again and placed in a thermostatted bath where it was held for 12-24 hours with frequent shaking.

The conductance of this solution was then determined by standard procedure using a 1000 Hz impedance bridge (1).

The concentration of the solution was calculated from conductivity measurements using the following equation

$$c = \frac{1000 L}{a - b\sqrt{c}}$$
 (continued)

SOURCE AND PURITY OF MATERIALS: Reagent grade lithium chloride was dried in a vacuum desicator over anhydrous magnesium perchlorate and used without further treatment. N-methylacetamide was prepared by reacting monomethylamine with glacial acetic acid and subsequent heating to distil off water. The product was purified by fractional distillation followed by five or more fractional freezing cycles, using a procedure described in detail by Berger and Dawson(3).

(Continued

ESTIMATED ERROR:

Solubility [±] 0.05% (compiler)

REFERENCES:

- Dawson, L.R.; Sears, P.G.; Graves, R.H. J. Am. Chem. Soc., <u>1955</u>, 77, 1986-1989
- Vaughn, J.W. Dissertation Thesis, Univ. Kentucky, <u>1959</u>, 31.
- Berger, C.; Dawson, R.L.; Anal Chem., <u>1952</u>, 24, 994-996.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium chloride; LiCl; 7447-41-8 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. <i>J. Phys. Chem.</i> , <u>1963</u> , 67, 281-283

METHOD/APPARATUS/PROCEDURE: (Continuation)

where <u>c</u> is the concentration of the solution, L the experimentally measured specific conductance and <u>a</u> and <u>b</u> the intercept and the slope, respectively, of a Kohlraushch plot, determined in the same laboratory, for solutions at various LiCl concentration (2).

SOURCE AND PURITY OF MATERIALS: (Continuation)

The specific conductance of N-methylacetamide so purified ranged from 0.6×10^{-5} S m⁻¹ to 2×10^{-5} S m⁻¹ at 40° C.

COMPONENTS :	EVALUATOR:
(1) Sodium chloride; NaCl; 7647-14-5 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979.

CRITICAL EVALUATION: Two investigations of the solubility of NaCl in N-methylacetamide have been reported (1-2).

Dawson et.al.(1) allowed long equilibration times to ensure saturation and carefully

controlled the conditions of operation. The concentration of NaCl was then calculated from Kohlrausch plots of solutions of the same salt.

Even if the conductometric technique might be rigorously applied only to dilute solutions, the value obtained by Dawson et.al. (1) appears as a valuable indication of the solubility of NaCl in N-methylacetamide.

Chandra and Gopal (2) have determined the solubility of NaCl in N-methylacetamide at five temperatures, i.e. 305 K, 308 K, 313 K, 318 K and 323 K, respectively. These authors have used precautions both in assuring high purity of the salt (by repeated crystallizations) and in reducing contamination of the solvent and the solutions by moisture (by performing all the manipulations in a dry environment). The solubility of NaCl was obtained gravimetrically by determining the anion content in the saturated solutions.

The method and the procedure seem to assure good reliability to the data of Chandra and Gopal (2), as also remarked by Lemire and Sears (3) in a recent review on the general properties of N-methylacetamide as solvent.

Finally, it has to be pointed out that Bonner, Jordon and Bunzl (4), in a paper dealing with the freezing point of electrolyte solutions in N-methylacetamide, briefly mentioned that alkali halides (and therefore possibly also sodium chloride, even if not specifically reported) are virtually insoluble in this solvent, without, however, giving experimental evidences to support this statement.

This observation largely contrasts with the experimental results of Dawson et.al. (1) and of Chandra and Gopan (2) who have both found consistent solubility values of NaCl in particular and of alkali halides in general, in N-methylacetamide. However, as remarked by Gopal and Bhatnagar (5), electrolytes are generally extremely slow to dissolve in N-methylacetamide. Therefore, too short equilibration times may lead to underestimation of the solubility, as probably was the case of Bonner et.al. (4).

Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by Dawson et.al. (1) with the molal solubility of Chandra and Gopal (2). However, considering that the time allowed by the latter authors for the equilibration of the solutions, i.e. 5 or 6 hours, may be too short to ensure complete saturation, the value of Dawson et.al. (1), obtained after an equilibration time exceeding two weeks, is preferable as a tentative indication of the solubility.

(Continued)

COMPONENTS: EVALUATOR: (1) Sodium chloride; NaCl; 7647-14-5 Bruno Scrosati, Istituto di Chinica Fisica, University of Rome, Italy. March 1979. CRITICAL EVALUATION: (Continuation) Tentative value of solubility of NaCl at 313 K 19.81 g dm ⁻³ of saturated solution Solubility of NaCl as a function at temperature For temperatures different than 313 K, only the data of Chandra and Gopal (2) are available, and these are $\frac{T/K}{305}$ 20.54 308 20.75 313 21.09 318 21.44 323 21.62 References 1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353. 3. Lemire, R.J.; Sears, P.G. Fortsoh, Chem. Forsoh., <u>1978</u> , 74, 71. 4. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., <u>1964</u> , 68, 2450-2454. 5. Gopal, R.; Bhatnagar, O.N. J. Phys. Chem., <u>1965</u> , 69, 2382-2385		
Tentative value of solubility of NaCl at 313 K 19.81 g dm ⁻³ of saturated solution Solubility of NaCl as a function at temperature For temperatures different than 313 K, only the data of Chandra and Gopal (2) are available, and these are T/K g/kg of solvent 305 20.54 308 20.75 313 21.09 318 21.44 323 21.82 References 1 Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. Phys. Chem., <u>1963</u> , 67, 281-283 2 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353. 3. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Forsch., <u>1978</u> , 74, 71. 4. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., <u>1964</u> , 68, 2450-2454.	(1) Sodium chloride; NaC1; 7647-14-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy.
T/K g/kg of solvent 305 20.54 308 20.75 313 21.09 318 21.44 323 21.82 References 1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. <i>J. Phys. Chem.</i> , <u>1963</u> , 67, 281-283 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353. 3. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Forsch., <u>1978</u> , 74, 71. 4. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., <u>1964</u> , 68, 2450-2454.	<u>Tentative value of solubility of NaCl at 313</u> 19.81 g dm <u>Solubility of NaCl as a function at temperatu</u> For temperatures different than 313 K, only t	⁻³ of saturated solution
305 20.54 308 20.75 313 21.09 318 21.44 323 21.82 References 1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353. 3. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Forsch., 1978, 74, 71. 4. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., 1964, 68, 2450-2454.	available, and these are	
308 20.75 313 21.09 318 21.44 323 21.82 References 1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353. 3. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Forsch., 1978, 74, 71. 4. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., 1964, 68, 2450-2454.	<u>T/K</u>	g/kg of_solvent_
 313 21.09 318 21.44 323 21.82 References Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. <i>J. Phys. Chem.</i>, <u>1963</u>, 67, 281-283 Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u>, 45(4), 351-353. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Forsch., <u>1978</u>, 74, 71. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., <u>1964</u>, 68, 2450-2454. 	305	20.54
 318 21.44 323 21.82 References 1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353. 3. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Forsch., 1978,74, 71. 4. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., 1964, 68, 2450-2454. 	308	20.75
 <u>References</u> 1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u>, 45(4), 351-353. 3. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Forsch., <u>1978</u>, 74, 71. 4. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., <u>1964</u>, 68, 2450-2454. 	313	21.09
 <u>References</u> 1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u>, 45(4), 351-353. 3. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Forsch., <u>1978</u>, 74, 71. 4. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., <u>1964</u>, 68, 2450-2454. 	318	21.44
 Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283 Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u>, 45(4), 351-353. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Forsch., <u>1978</u>, 74, 71. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. J. Phys. Chem., <u>1964</u>, 68, 2450-2454. 	323	21.82
	 Dawson, R.L.; Berger, J.E.; Vaughn, J.W. J. Phys. Chem., <u>1963</u>, 67, 281-283 Chandra, D.; Gopal, R. J. Indian Chem. S. Lemire, R.J.; Sears, P.G. Fortsch. Chem. Bonner, O.D.; Jordan, C.F.; Bunzl, K.W. 	Soc., <u>1968</u> , 45(4), 351–353. . Forsch., <u>1978</u> ,74, 71. . J. Phys. Chem., <u>1964</u> , 68, 2450–2454.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; 7647-14-5	Chandra, D.; Gopal, R.
(2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353
5,	
VARIABLES:	PREPARED BY:
Т	
	Bruno Scrosati
EXPERIMENTAL VALUES:	
Temperature	PREPARED BY: Bruno Scrosati

Solubility of sodium chloride at five temperatures

<u>t/°C</u>	g/100 g of solvent
32	2.05
35	2.08
40	2.11
45	2.14
50	2.18

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The solubility was determined gravimetrically. The procedure was described in detail in a previous paper from the same laboratory (1). Finely divided solute was added to a suit- able quantity of N-methylacetamide contained in a flask. The flask was shaken contin- uously and then placed in a thermostat where the temperature was controlled within $\stackrel{+}{-} 0.05$ C in the lower range (35-40°C) and within $\stackrel{+}{-} 0.1$ C in the upper range (45-50°C). After stirring the solvent and the solute in the flask for 5 or 6 hours, the solute was allowed to settle. Three samples of the clear saturated solution were withdrawn at successive intervals with a pipette and weighted. The samples were then diluted with water and the anion content in the	crystallizations. N-methylacetamide was dried over freshly ignited quicklime and then distilled under reduced pressure. The middle fraction was collected, fractionally crystallized twice and then distilled again under reduced pressure. The specific conductance of N-methylacetamide so purified was about 10 ⁻⁴ S.m-1
	ESTIMATED ERROR: Solubility [±] 0.5% Temperature [±] (0.05-0.1) [°] C (authors)
solution determined gravimetrically.	<pre>REFERENCES: 1. Gopal, R.; Hussain, M.M. J. Indian Chem. Soc., 1963, 40(4), 272-274.</pre>

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; 7647-14-5 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283.
VARIABLES:	PREPARED BY:
One temperature: 40 [°] C	Bruno Scrosati
EXPERIMENTAL VALUES:	

Solubility of sodium chloride at 40°C

0.339 mol dm⁻³

NFORMATION
OURCE AND PURITY OF MATERIALS:
Reagent grade NaCl was dried in a vacuum esiccator over anhydrous magnesium perch- lorate and used without further treatment. The preparation and the purification of N-methylacetamide have been described in the compilation of the lithium chloride-N- methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for letails. Solubility ⁺ 0.5% (compiler). REFERENCES:
Res Lo Theome Lo Theome Lo Theome Lo Theome Lo So

COMPONENTS :	EVALUATOR:
(1) Sodium bromide; NaBr; 7647-15-6 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979.

Two investigations of the solubility of NaBr in N-methylacetamide have been reported (1-2).

Dawson et.al. (1) have determined an approximate value of solubility at 313 K by incremental addition of small amounts of NaBr to a known volume of N-methylacetamide until saturation was visually observed. The solution was then heated to 333 K and it was assumed to have reached saturation if solute precipitated upon coolikg to 313 K. This procedure is certainly open to some criticism and indeed the authors themselves attribute an error of $\frac{+}{5}$ % to their 'approximate' data (1).

Chandra and Gopal (2) have determined gravimetrically the solubility of NaBr in N-methylacetamide at five temperatures, i.e. 305 K, 308 K, 313 K, 318 K and 323 K, respectively. These authors have used precautions both in assuring high purity of the salt (by repeated crystallizations) and in reducing contamination of the solvent and the solutions by moisture (by performing all the manipulations in a dry environment).

Even if the result of Chandra and Gopal (2) may be affected by a too short equilibration time, it still appears more reliable than the approximate value of Dawson et.al. (1) for a tentative indication of the solubility.

Tentative value of solubility of NaBr at 313 K

192.31 g/kg of solvent

Solubility of NaBr as a function of temperature

For temperatures other than 313 K, only the data of Chandra and Gopal (2) are available and these are

<u>T/K</u>	g/kg of Solvent
305	190.8
308	191.6
313	192.3
318	193.1
323	194.2

References

- Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283.
- 2. Chandra, D.; Gopal, R. J. Indian. Chem. Soc., 1968, 45(4), 351-353.

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N-Methylacetamide 26	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; 7647-15-6	Chandra, D.; Gopal, R.
(2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353.
<u></u>	
VARIABLES:	PREPARED BY:
Temperature	Bruno Scrosati
EXPERIMENTAL VALUES:	J
Solubility of sodium bromide at five temperat	tures.
t/°C g/100) g of solvent
32	19.08
35	19.16
40	19.23
45	19.31
50	19.42
AUXILIARY	INFORMATIÔN
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The solubility was determined gravimetrically	Sodium bromide was purified by repeated crystallizations.
The method and the procedure have been described in the course of the compilation of the sodium chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.	The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.
	ESTIMATED ERROR:
	Solubility [±] 0.5%
	Temperature $\stackrel{+}{=}$ (0.05-0.1) ^o C (authors)
	REFERENCES :

264 /V-N	Aethylacetamide
COMPONENTS: (1) Sodium bromide; NaBr; 7647-15-6 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-	ORIGINAL MEASUREMENTS: Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283
VARIABLES:	PREPARED BY:
One temperature: 40°C	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of sodium bromide at 40 ⁰ C	
1.85	mol dm ⁻³
-	LIARY INFORMATION
METHOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
An 'approximate' (authors) solubility value determined by the use of a large test tu fitted with a stopper and covered with aluminium foil, into which was placed 10 of solvent. The solute was added in sma increments with constant shaking. When the solution appeared to be saturat	perchlorate and used without further treat- ment. As described in another paper from the same laboratory (1), N-methylacetamide was prepared by a reaction of monomethylami with glacial acetic acid and subsequent
the test tube was heated to 60° C in a was bath with frequent shaking. It was assu	iter heating to crack out and distil off water.
that the solution had indeed reached	distillation followed by five or more

The product was purified by fractional distillation followed by five or more (Continued)

ESTIMATED ERROR:

Solubility [±] 5% (authors)

REFERENCES:

saturation if solute precipitated upon cooling to 40°C.

added.

The approximate solubility value, which the

authors consider to have a precision of 5%, was determined on the basis of the salt

Dawson, L.R.; Sears, P.G.; Groves, R.H. J. Am. Chem. Soc., <u>1955</u>, 77, 1986-1989

2. Berger, C.; Dawson, L.R. Anal. Chem., 1952, 24, 994-996.

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; 7647-15-6 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283

SOURCE AND PURITY OF MATERIALS: (Continuation)

fractional freezing cycles, using a procedure described in detail by Berger and Dawson (2).

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The specific conductance of N-methylacetamide so purified ranged from 0.6 \times 10^{-5} S m<sup>-1</sup> to 2 \times 10^{-5} S m<sup>-1</sup>.
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EVALUATOR: Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy.
March 1979

The system sodium iodide-N-methylacetamide has been investigated by Dawson, Berger, Vaughn and Eckstrom (1) and by Chandra and Gopal (2).

Dawson et.al. (1) have reported an approximate value of solubility at 313 K determined by addition of small increments of NaI to a known volume of N-methylacetamide until saturation was visually observed. The solution was then heated to 333 K and it was assumed to have reached saturation if solute precipitated upon cooling to 313 K. This procedure is certainly open to some criticism and indeed the authors themselves attribute an error of $\frac{+}{-}$ 5% to their 'approximate' data (1).

Chandra and Gopal (2) have determined gravimetrically the solubility of NaI in N-methylacetamide at five temperatures, i.e. 305 K, 308 K, 313 K, 318 K and 323 K, respectively. These authors have used precautions both in assuring high purity of the salt (by repeated crystallizations) and in reducing contamination of the solvent and the solutions by moisture (by performing all the manipulations in a dry environment).

Even if the result of Chandra and Gopal (2) may be affected by a too short equilibration time, it still appears more reliable than the approximate value of Dawson et.al. (1) for a tentative indication of the solubility.

Tentative value of solubility of NaI at 313 K

562.7 g/kg of solvent.

Solubility of NaI as a function of temperature

For temperatures different than 313 K, only the data of Chandra and Gopal (2) are available, and these are

<u>T/K</u>	g/kg of solvent
305	514.8
308	533.0
313	562.7
318	592.3
323	623 · 2

References

 Dawson, R.L.; Berger, J.E.: Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283.

2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353.

<i>N</i> -Methylacetamide	
OMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium Iodide; NaI; 7681-82-5	Chandra, D; Gopal, R.
(2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353
37	
VARIABLES :	PREPARED BY:
Temperature	Bruno Scrosati
XPERIMENTAL VALUES:	
Solubility of sodium iodide at five temperat	ures
0	0 g of solvent
<u>1/ c</u> <u>g/10</u> 32	51.48
32	53.30
40	56.27
40	59.23*
50	62.32
* In the original paper this value is r	
number <u>2</u> was a printing mistake.	
number <u>2</u> was a printing mistake.	
	INFORMATION
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically	SOURCE AND PURITY OF MATERIALS:
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically The method and the procedure have been described in the course of the compilation of the sodijm chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for	SOURCE AND PURITY OF MATERIALS: y. Sodium iodide was purified by repeated crystallizations. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically The method and the procedure have been described in the course of the compilation of the sodijm chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for	SOURCE AND PURITY OF MATERIALS: y. Sodium iodide was purified by repeated crystallizations. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N-
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically The method and the procedure have been described in the course of the compilation of the sodijm chloride-N-methylacetamide system, originally measured by Chandra and	SOURCE AND PURITY OF MATERIALS: y Sodium iodide was purified by repeated crystallizations. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically The method and the procedure have been described in the course of the compilation of the sodijm chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for	SOURCE AND PURITY OF MATERIALS: y. Sodium iodide was purified by repeated crystallizations. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details. ESTIMATED ERROR:
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically The method and the procedure have been described in the course of the compilation of the sodijm chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for	SOURCE AND PURITY OF MATERIALS: y. Sodium iodide was purified by repeated crystallizations. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically The method and the procedure have been described in the course of the compilation of the sodijm chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for	SOURCE AND PURITY OF MATERIALS: y Sodium iodide was purified by repeated crystallizations. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details. ESTIMATED ERROR: Solubility [±] 0.5% Temperature [±] (0.05-0.1) ^o C (authors).
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically The method and the procedure have been described in the course of the compilation of the sodijm chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for	SOURCE AND PURITY OF MATERIALS: Sodium iodide was purified by repeated crystallizations. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details. ESTIMATED ERROR: Solubility ± 0.5%
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically The method and the procedure have been described in the course of the compilation of the sodijm chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for	SOURCE AND PURITY OF MATERIALS: y Sodium iodide was purified by repeated crystallizations. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details. ESTIMATED ERROR: Solubility [±] 0.5% Temperature [±] (0.05-0.1) ^o C (authors).
AUXILIARY ÆTHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetrically The method and the procedure have been described in the course of the compilation of the sodijm chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for	SOURCE AND PURITY OF MATERIALS: y Sodium iodide was purified by repeated crystallizations. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details. ESTIMATED ERROR: Solubility [±] 0.5% Temperature [±] (0.05-0.1) ^o C (authors).

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium iodide; NaI; 7681-82-5	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.;
(2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Eckstrom, H.C.
37	J. Phys. Chem., <u>1963</u> , 67, 281–283.
VARIABLES:	PREPARED BY:
One temperature: 40°C	Bruno Scrosati
one temperature. 40 C	
EXPERIMENTAL VALUES:	
Solubility of sodium iodide at 40 ⁰ C	
2.86 mo	$1 dm^{-3}$
2.00 m	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
An 'approximate' (authors) value of	Reagent grade NaI was dried in a vacuum
solubility was determined by progressive additions in small increments of solute to	desiccator over anhydrous magnesium perchlorate and used without further
the solvent until saturation was visually	treatment. The preparation and purifica-
observed.	tion of N-methylacetamide have been described in the compilation of the lithium
Details on the procedure have been described in the compilation of the sodium	chloride-N-methylacetamide system,
bromide-N-methylacetamide system,	originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the
originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer	reader for details.
the reader.	
	ESTIMATED ERROR:
	Solubility [±] 5% (authors)
	DEPENDING .
	REFERENCES :

1	COMPONENTS :	EVALUATOR:
	(1) Sodium thiocyanate; NaSCN; 540-72-7 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

Very few solubility data of pseudohalides in amides are available from the literature. In particular, so far as the evaluator knows, the system sodium thiocyanate-N-methylacetamide has been investigated only by Dawson, Berger, Vaughn and Eckstrom (1).

These authors have reported an approximate value of the solubility (295.09 g dm⁻³ of saturated solution at 313 K), determined by addition of small increments of NaSCN to a known volume of N-methylacetamide until saturation was visually observed. The solution was then heated to 333 K and it was assumed to have reached saturation if solute precipitated upon cooling to 313 K. This procedure is open to some criticism and indeed the authors themselves attribute an error of $\frac{+}{2}$ 5% to their datum.

Therefore only an approximate value of the solubility of sodium thiocyanate in N - methylacetamide may be reported.

Approximate value of solubility of NaSCN at 313 K 295.1 g dm⁻³ of saturated solution

Since the density of the saturated solution is not known it is not possible to convert this datum into molal units.

References

 Dawson, R.L.: Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium thiocyanate; NaSCN; 540-72- (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Fekstrom H.C.
VARIABLES: One temperature: 40 ⁰ C	PREPARED BY: Bruno Scrosati
EXPERIMENTAL VALUES:	

Solubility of sodium thiocyanate at 40° C

 $3.64 \text{ mol } \text{dm}^{-3}$

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: An 'approximate' (authors) value of solubil- ity was determined by progressive additions in small increments of solute to the solvent until saturation was visually observed. Details on the procedure have been described in the compilation of the sodium bromide-N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader.	desiccator over anhydrous magnesium perchlorate and used without further treatment. The preparation and the purification of N-methylacetamide have been described in the compilation of the lithium chloride-N-

COMPONENTS:	EVALUATOR:
(1) Potassium chloride; KC1; 7447-40-7 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

The system potassium chloride-N-methylacetamide has been investigated by Dawson, Berger, Vaughn and Eckstrom (1) and by Chandra and Gopal (2).

Dawson et.al. (1) have determined the solubility of KC1 in N-methylacetamide at 313 K by measuring the conductivity of the saturated solution. The concentration of the latter was obtained from Kohlrausch plots of solutions of the same salt. It has to be remarked that these authors allowed long equilibration times (over two weeks) to ensure saturation, and carefully controlled the conditions of operation. Chandra and Gopal (2) have determined gravimetrically the solubility of KC1 in N-methylacetamide at five temperatures, i.e., 305 K, 308 K, 313 K, 318 K and 323 K. These authors have used precautions both in assuring high purity of the salt (by repeated crystallizations) and in reducing contamination of the solvent and the solutions by moisture (by performing all the manipulations in a dry environment).

Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by Dawson et.al. (1) with the molal solubility of Chandra and Gopal (2). However, considering that the time allowed by the latter authors for the equilibration of the solutions, i.e. 5 or 6 hours, may result too short to ensure complete saturation, the value of Dawson et.al. (1), obtained after equilibration times exceeding two weeks, becomes preferable as a tentative indication of the solubility.

Tentative value of solubility of KCl at 313 K

8.95 g dm⁻³ of saturated solution.

Solubility of KCl as a function of temperature

For temperatures different than 313 K, only the data of Chandra and Gopal (2) are available and these are

<u>T/K</u>	g/kg of solvent
305	8.65
308	8.99
313	9.58
318	10.07
323	10.71

References

- Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283.
- 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353.

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KCl; 7447-40-7	Chandra, D.; Gopal, R.
(2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353
VARIABLES: Temperature	PREPARED BY: Bruno Scrosati
EXPERIMENTAL VALUES:	

Solubility of potassium chloride at five temperatures.

<u>t/°C</u>	g/100 g of solvent
32	0.865
35	0.899
40	0.958
45	1.007
50	1.071

AUXILIARY INFORMATION

SOURCE AND PURITY OF MATERIALS: Potassium chloride was purified by repeated crystallizations.		
The purification of N-methylacetamide has been described in the course of the compil- ation of the sodium chloride-N-methylaceta- mide system, originally measured by Chandra and Gopal, to which we refer the reader for details.		
ESTIMATED ERROR:		
Solubility ± 0.5% (authors)		
Temperature $\stackrel{+}{=}$ (0.05-0.1) ^o C (authors).		
REFERENCES :		

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KC1; 7447-40-7 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283.
VARIABLES: One temperature: 40 ⁰ C	PREPARED BY: Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of potassium chloride at 40° C

 $0.120 \text{ mol } dm^{-3}$

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: The solubility was determined conducti- metrically. The method and the procedure for preparing the saturated solutions and determining their concentration are the same as described in the course of the compilation of the lithium chloride-N-methylacetamide system, originally measured by Dawson, Berger Vaughn and Eckstrom, to which we refer the reader for details.	SOURCE AND PURITY OF MATERIALS: Reagent grade KCl was dried in a vacuum desiccator over anhydrous magnesium perchlorate and used without further treatment. The preparation and the purification of N-methylacetamide have been described in the compilation of the lithium chloride-N- methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details. ESTIMATED ERROR: Solubility [±] 0.5% (compiler) REFERENCES:

COMPONENTS:		EVALUATOR:
 Potassium bromide; N-methylacetamide; 	KBr; 7558-02-3 C ₃ H ₇ NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

The system potassium bromide-N-methylacetamide has been investigated by Dawson, Berger, Vaughn and Eckstrom (1) and by Chandra and Gopal (2).

Dawson et.al. (1) have determined the solubility of KBr in N-methylacetamide by measuring the conductivity of the saturated solution. The concentration of the latter was obtained from Kohlrausch plots of solutions of the same salt. It has to be remarked that these authors allowed long equilibration times (over two weeks) to ensure saturation and carefully controlled the conditions of operation.

Chandra and Gopal (2) have determined gravimetrically the solubility of KBr in N-methylacetamide at five temperatures, i.e. 305 K, 308 K, 313 K, 318 K and 323 K. These authors have used precautions both in assuring high purity of the salt (by repeated crystallizations) and in reducing contamination of the solvent and the solutions by moisture (by performing all the manipulations in a dry environment). However, the time allowed for equilibration of the solutions, i.e. 5 or 6 hours, may be in certain cases too short to ensure complete saturation. Indeed, as remarked by the same Gopal in a paper with Bhatnagar (3), electrolytes are generally extremely slow to dissolve in N-methylacetamide.

Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by Dawson et.al. (1) with the molal solubility of Chandra and Gopal (2).

The possibility that the data of Chandra and Gopal (2) may be affected by a certain degree of error due to the short equilibration time has to be considered and the data of Dawson et.al. (1), obtained after equilibration times exceeding two weeks, became preferable in comparison for a tentative indication of the solubility of KBr in N-methyl-acetamide at 313 K.

Tentative value of solubility of KBr at 313 K

51.06 g dm⁻³ of saturated solution.

Solubility	of	KBr	as	function	of	temperatures

For temperatures different than 313 K, only the data of Chandra and Gopal (2) are available and these are

<u>т/к</u>	g/kg of solvent
305	47.73
308	48.62
313	50.03
318	51.42
323	52.78

(Continued)

COMPONENTS:	EVALUATOR:
(1) Potassium bromide; KBr; 7558-02-3 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION: (Continuation)

References

- Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283
- 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u>, 45(4), 351-353.
- 3. Gopal, R.; Bhatnagar, O.N. J. Phys. Chem., 1965 69, 2382-2385.

COMPONENTS:		ORIGINAL MEASUREMENTS:
 Potassium bromide; N-methylacetamide; 	-	Chandra, D.; Gopal, R J. Indian Chem. Soc., <u>1968</u> ,45(4) , 351-353
VARIABLES: Temperature		PREPARED BY: Bruno Scrosati
EXPERIMENTAL VALUES: Solubility of potassium bromide at five temperatures		
	t/ ^o C g/10	00 g of solvent

<u>t/C</u>	g/100 g of solve
32	4.773
35	4.862
40	5.003
45	5.142
50	5.278

AUXILIARY INFORMATION		
METHOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:	
The solubility was determined gravimetri- cally.	Potassium bromide was purified by repeated crystallizations.	
The method and the procedure have been described in the course of the compilation of the sodium chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.	The purification of N-methylacetamide has been described in the course of the compil- ation of the sodium chloride-N-methylacet- amide system, originally measured by Chandra and Gopal, to which we refer the reader for details.	
	ESTIMATED ERROR:	
	Solubility ⁺ 0.5% (authors)	
	Temperature $\frac{+}{2}$ (0.05-0.1) ^o C (authors)	
	REFERENCES:	

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium bromide; KBr; 7558-02-3 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283
VARIABLES: One temperature: 40 ⁰ C	PREPARED BY: Bruno Scrosati
EXPERIMENTAL VALUES: Solubility of potassium bromide at 40 ⁰ C	I

 $0.429 \text{ mol } \text{dm}^{-3}$

AUXILIARY	INFORMATION		
METHOD/APPARATUS/PROCEDURE: The solubility was determined conductometric-			
The solubility was determined conductometric- ally. The method and the procedure for preparing the saturated solutions and determining their concentration are the same described in the course of the compilation of the lithium chloride-N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.	desiccator over anhydrous magnesium perchlorate and used without further treatment.		
	The preparation and the purification of N-methylacetamide have been described in the compilation of the lithium chloride-N- methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details		
	ESTIMATED ERROR: Solubility ⁺ 0.5% (compiler)		
	REFERENCES :		

COMPONENTS:	EVALUATOR:
 (1) Potassium iodide; KI; 7681-11-0 (2) N-methylacetamide; C₃H₇NO; 79-16-3 	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

The system potassium iodide-N-methylacetamide has been investigated by Dawson, Berger, Vaughn and Eckstrom (1) and by Chandra and Gopal (2).

Dawson et.al.(1) have determined the solubility of KI in N-methylacetamide at 313 K by measuring the conductivity of the saturated solution. The concentration of the latter was obtained from Kohlransch plots of solutions of the same salt. It has to be remarked that these authors allowed long equilibration times (over two weeks) to ensure saturation and carefully controlled the conditions of operation.

Chandra and Gopal (2) have determined gravimetrically the solubility of KI in N-methylacetamide at five temperatures, i.e., 305 K, 308 K, 313 K, 318 K and 323 K. These authors have used precautions both in assuring high purity of the salt (by repeated crystallizations) and in reducing contamination of the solvent and the solutions by moisture (by performing all the manipulations in a dry environment).

However, the time allowed for equilibration of the solutions, i.e. 5 or 6 hours, may be in certain cases too short to ensure complete saturation. Indeed, as remarked by the same Gopal in a paper with Bhatnagar (3), electrolytes are generally extremely slow to dissolve in N-methylacetamide.

Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by Dawson et.al.(1) with the molal solubility of Chandra and Gopal (2).

The possibility that the data of Chandra and Gopal (2) may be affected by a certain degree of error due to the short equilibration time has to be considered and the data of Dawson et.al. (1), obtained after equilibration times exceeding two weeks, become preferable in comparison, for a tentative indication of the solubility of KI in N-methylacetamide at 313 K.

Tentative value of solubility of KI at 313 K

225.1 g dm⁻³ of saturated solution.

Solubility of KI as a function of temperature

For temperatures different than 313 K, only the data of Chandra and Gopal (2) are available and these are

<u>t/k</u>	g/kg of solvent	
305	304.3	
308	306.8	
313	316.6	
318	316.9	
323	322.8	

(Continued)

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COMPONENTS:	EVALUATOR:
(1) Potassium iodide; KI; 7681-11-0 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION: (Continuation)

References

- Dawson, R.L.; Berger, J.E.; Vaughn, J.W.: Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283.
- 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353.
- 3. Gopal, R.; Bhatnagar, O.N. J. Phys. Chem., 1965, 69, 2382-2385.

280 N-Methyl	lacetamide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; 7681-11-0	Chandra, D.; Gopal, R.
(2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	J. Indian Chem. Soc., 1968, 45(4), 351-353
VARIABLES:	PREPARED BY:
Temperature	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of KI at five temperatures	
$\frac{t/^{\circ}C}{g/1}$	00 g of solvent
32	30.43
35	30.68
40	31.16
45	31.69
50	32.28
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The solubility was determined gravimetric- ally.	Potassium iodide was purified by repeated crystallizations.
The method and the procedure have been described in the course of the compilation of the sodium chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.	The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.
	ESTIMATED ERROR:
	Solubility ⁺ 0.5% (authors)
	Temperature $\stackrel{+}{=}$ (0.05-0.1) ^o C (authors)
	REFERENCES :

N-Methylacetamide 28	
COMPONENTS: (1) Potassium iodide; KI; 7681-11-0 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	ORIGINAL MEASUREMENTS: Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283.
VARIABLES: One temperature: 40 [°] C	PREPARED BY: Bruno Scrosati
EXPERIMENTAL VALUES: Solubility of potassium iodide at 40 ⁰ C	
METHOD/APPARATUS/PROCEDURE: The solubility was determined conductometri- cally. The method and the procedure for preparing the saturated solutions and determining their concentration are the same described in the course of the compilation of the lithium chloride-N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.	INFORMATION SOURCE AND PURITY OF MATERIALS: Reagent grade KI was dried in a vacuum desiccator over anhydrous magnesium perchlorate and used without further treatment. The preparation and the purification of N-methylacetamide have been described in the compilation of the lithium chloride-N- methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details. ESTIMATED ERROR: Solubility + 0.5% (compiler)
	REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Potassium thiocyanate; KSCN; 333-20-0 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

Very few solubility data of pseudohalides in amides are available from the literature. In particular, the potassium thiocyanate-N-methylacetamide system has been investigated only by Dawson, Berger, Vaughn and Eckstrom (1).

These authors reported a solubility value at 313 K of 210.88 g dm⁻³ of saturated solution. The solubility was determined by measuring the conductivity of the saturated solution. The concentration of the latter was obtained from Kohlrausch plots of solutions of the same salt. It has to be remarked that precautions were taken to ensure saturation and to achieve precision in the measurements.

Nevertheless, because of the lack of data from other sources which may serve as terms of comparison and evaluation, the result of Dawson et.al. is reported as tentative value of the solubility of potassium thiocyanate in N-methylacetamide.

Tentative value of solubility of KSCN at 313 K

210.88 g dm⁻³ of saturated solution

Since the density of the saturated solution is not known, it is not possible to convert this datum into molal units.

References

 Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283.

COMPONENTS: (1) Potassium thiocyanate; KSCN; 333-20-0 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	ORIGINAL MEASUREMENTS: Dawson, E.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283.	
VARIABLES: One temperature: 40 [°] C	PREPARED BY: Bruno Scrosati	
EXPERIMENTAL VALUES: Solubility of potassium thiocyanate at 40 [°] C		
2.17 mol dm^{-3}		

AUXILIARY INFORMATION		
METHOD/APPARATUS/PROCEDURE: The solubility was determined conductometr- ically. The method and the procedure for preparing the saturated solutions and determining their concentration are the same as described in the course of the compilation of the lithium chloride-N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.	SOURCE AND PURITY OF MATERIALS: Reagent grade KSCN was dried in a vacuum desiccator over anhydrous magnesium perchlorate and used without further treatment. The preparation and purification of N-methylacetamide have been described in the compilation of the lithium chloride- N-methylacetamide system originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details. ESTIMATED ERROR: Solubility ⁺ 0.5% (compiler) REFERENCES:	

COMPONENTS:			EVALUATOR:
 (1) Ammonium chloride; 12125-02-9 (2) N-methylacetamide; 	4	79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

The system ammonium chloride-N-methylacetamide has been investigated by Dawson, Berger, Vaughn and Eckstrom (1) and by Chandra and Gopal (2).

Dawson et.al. (1) have determined the solubility of NH_4C1 in N-methylacetamide at 313 K by measuring the conductivity of the saturated solution. The concentration of the latter was obtained from Kohlrausch plots of solutions of the same salt.

It has to be remarked that these authors allowed long equilibration times (over two weeks) to ensure saturation and carefully controlled the conditions of operation.

Chandra and Gopal (2) have determined gravimetrically the solubility of NH₄Cl in N-methylacetamide at five temperatures, i.e., 305 K, 308 K, 313 K, 318 K and 323 K. These authors have used precautions both in assuring high purity of the salt (by repeated crystallizations) and in reducing contamination of the solvent and the solutions by moisture (by performing all the manipulations in a dry environment).

However, the time allowed for equilibration of the solutions, i.e., 5 or 6 hours, may be in certain cases too short to ensure complete saturation. Indeed, as remarked by the same Gopal in a paper with Bhatnagar (3), electrolytes are generally extremely slow to dissolve in N-methylacetamide.

Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by Dawson et.al.(1) with the molal solubility of Chandra and Gopal (2).

The possibility that the data of Chandra and Gopal (2) may be affected by a certain degree of error due to the short equilibration time has to be considered and the data of Dawson et.al. (1), obtained after equilibration times exceeding two weeks, become preferable in comparison for a tentative indication of the solubility of NH₄Cl in N-methylacetamide of 313 K.

Tentative value of solubility of NH_4C1 at 313 K 47.93 g dm⁻³ of saturated solution

Solubility of NH, Cl as a function of temperature

For temperatures other than 313 K, only the data of Chandra and Gopal (2) are available and these are

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(Continued)

COMPONENTS: (1) Ammonium chloride; 12125-02-9 (2) N-methylacetamide;	NH ₄ C1; C ₃ H ₇ NO; 79-16-3	EVALUATOR: Bruno Scrosati. Istituto di Chimica Fisica, University of Rome, Italy. March 1979
CRITICAL EVALUATION:	(continuation) <u>T/K</u> g/kg 305 308 313 318 323	g of solvent 48.83 49.67 50.93 52.21 53.53

References

- Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283.
- 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353.
- 3. Gopal, R.; Bhatnagar, O.N. J. Phys. Chem., 1965, 69, 2382-2385.

ORIGINAL MEASUREMENTS:
Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353.
PREPARED BY:
Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of NH_4C1 at five temperatures

<u>t/°c</u>	g/100 g of solvent
32	4.883
35	4.967
40	5.093
45	5.221
50	5.353

AUXILIAR	(INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The solubility was determined gravi- metrically.	Ammonium chloride was purified by repeated crystallizations.
The method and the procedure have been described in the course of the compilation of the sodium chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.	The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.
	ESTIMATED ERROR:
	Solubility \pm 0.5% (authors)
	Temperature $\stackrel{+}{=}$ (0.05-0.1) ^o C (authors.
	REFERENCES :

COMPONENTS :	ORIGINAL MEASUREMENTS:
<pre>(1) Ammonium chloride; NH₄Cl; 12125-02-9 (2) N-methylacetamide; C₃H₇NO; 79-16-3</pre>	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283.
VARIABLES:	PREPARED BY:
One temperature: 40 [°] C	Bruno Scrosati
EXPERIMENTAL VALUES:	

Solubility of ammonium chloride at 40⁰C

0.896 mol dm⁻³

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The solubility was determined conducto- metrically. The method and the procedure for preparing	Reagent grade NH ₂ Cl was dried in a vacuum desiccator over anhydrous magnesium perchlorate and used without further treatment.
the saturated solutions and determining their concentration are the same described in the course of the compilation of the lithium chloride-N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.	The preparation and the purification of N-methylacetamide have been described in the compilation of the lithium chloride-N- methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.
	ESTIMATED ERROR: Solubility ⁺ 0.5% (compiler)
	REFERENCES:

COMPONENTS :	EVALUATOR:
 (1) Ammonium bromide; NH₄Br; 12124-97-9 (2) N-methylacetamide; C₃H₇NO; 79-16-3 	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

The ammonium bromide-N-methylacetamide system has been investigated by Dawson, Berger, Vaughn and Eckstrom (1) and by Chandra and Gopal (2).

Dawson et.al. (1) have determined the solubility of NH_4Br in N-methylacetamide at 313 K by measuring the conductivity of the saturated solution. The concentration of the latter was obtained from Kohlrausch plots of solutions of the same salt. It has to be remarked that these authors allowed long equilibration times (over two weeks) to ensure saturation and carefully controlled the conditions of operation.

Chandra and Gopal (2) have determined the solubility of NH₄Br in N-methylacetamide gravimetrically at five temperatures, i.e., 305 K, 308 K, 313 K, 318 K and 323 K. These authors have used precautions both in assuring high purity of the salt (by repeated crystallizations) and in reducing contamination of the solvent and the solutions by moisture (by performing all the manipulations in a dry environment.

However, the time allowed for equilibration of the solutions, i.e., 5 or 6 hours, may be in certain cases too short to ensure complete saturation. Indeed, as remarked by the same Gopal in a paper with Bhatnagar (3), electrolytes are generally extremely slow to dissolve in N-methylacetamide.

Since the density of the saturated solution is not known, it is not possible to make a direct comparison of the molar solubility given by Dawson et.al. (1) with the molal solubility of Chandra and Gopal (2).

The possibility that the data of Chandra and Gopal (2) may be affected by a certain degree of error due to the short equilibration time has to be considered and the data of Dawson et.al. (1), obtained after equilibration times exceeding two weeks, become preferable in comparison for a tentative indication of the solubility of NH_4Br in N-methylacetamide at 313 K.

Tentative value of solubility of NH₄Br at 313 K 170.92 g dm⁻³ of saturated solution

Solubility of NH, Br as a function of temperature

For temperatures different than 313 K, only the data of Chandra and Gopal (2) are available and these are

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COMPONENTS: (1) Ammonium bromide; NH ₄ Br; 12124-97-9 (2) N-methylacetamide; C ₃ H ₇ NO;	79-16-3	EVALUATOR: Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979
CRITICAL EVALUATION: (Continua	ation)	
	T/K g/kg	; of solvent
	305	218.6
	308	219.6
	313	221.1
	318	222.6
	323	224.3
1		

References

 Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u>, 67, 281-283.

2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353.

3. Gopal, R.; Bhatnagar, O.N. J. Phys. Chem., 1965, 69, 2382-2385.

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90 <i>N</i> -Methylacetamide	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ammonium bromide; NH ₄ Br;	Chandra, D.; Gopal, R.
12124-97-9	J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353
(2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	
VARIABLES:	PREPARED BY:
Temperature	Bruno Scrosati
EXPERIMENTAL VALUES:	l
Solubility of NH ₄ Br at five temperatures	
· · · · · · · · · · · · · · · · · · ·	
	0 g of solvent
32	21.86
35	21.96
40	22.11
45	22.26
50	22.43
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The solubility was determined gravi-	Ammonium bromide was purified by repeated
metrically.	crystallizations.
The method and the procedure have been described in the course of the compilation	The purification of N-methylacetamide has been described in the course of the
of the sodium chloride-N-methylacetamide	compilation of the sodium chloride-N-
system, originally measured by Chandra and	methylacetamide system, originally measured by Chandra and Gopal, to which we refer the
Gopal, to which we refer the reader for details.	reader for details.
	ESTIMATED ERROR:
	Solubility + 0.5% (authors)
	Temperature ⁺ (0.05-0.1) ^o C (authors)
	REFERENCES :
	ALL DEMOLD.
}	

COMPONENTS :	ORIGINAL MEASUREMENTS:
<pre>(1) Ammonium bromide; NH₄Br;</pre>	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.;
12124-97-9	Eckstrom, H.C.
(2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	J. Phys. Chem., <u>1963</u> , 67, 281-283
VARIABLES:	PREPARED BY:
One temperature: 40 [°] C	Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of ammonium bromide at 40° C

 $1.745 \text{ mol } dm^{-3}$

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The solubility was determined conducto- metrically. The method and the procedure for preparing the saturated solutions and determining their concentration are the same described in the course of the compilation of the lithium chloride-N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.	Reagent grade NH,Br was dried in a vacuum desiccator over anhydrous magnesium perchlorate and used without further treatment.
	Treatment. The preparation and the purification of N-methylacetamide have been described in the compilation of the lithium chloride- N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.
	ESTIMATED ERROR:
	Solubility ⁺ 0.5% (compiler)
	REFERENCES:

COMPONENTS:	EVALUATOR:
<pre>(1) Ammonium iodide; NH₄I; 12027-06-4 (2) N-methylacetamide; C₃H₇NO; 79-16-3</pre>	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

Very few solubility data for halides in amides are available in the literature. In particular, the potassium iodide-N-methylacetamide system has been investigated only by Dawson, Berger, Vaughn and Eckstrom (1).

These authors reported a solubility value at 313 K of 358.00 g dm⁻³ of saturated solution. The solubility was determined by measuring the conductivity of the saturated solution. The concentration of the latter was obtained from Kohlrausch plots of solutions of the same salt. It has to be remarked that precautions were taken to ensure saturation and to achieve precision in the measurements.

Nevertheless, because of the lack of data from other sources which may serve as terms of comparison and evaluation, the result of Dawson et.al. is reported as a tentative value of the solubility .

Tentative value of solubility of NH4I at 313 K

358.00 g dm⁻³ of saturated solution.

Since the density of the saturated solution is not known, it is not possible to convert this datum into molal units.

References

 Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283.

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PONENTS:	ORIGINAL MEASUREMENTS:	
<pre>(1) Ammonium iodide; NH₄I;</pre>	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.	
12027-06-4	Eckstrom, H.C.	
(2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	J. Phys. Chem., <u>1963</u> , 67, 281-283	
VARIABLES:	PREPARED BY:	
One temperature: 40 ⁰ C	Bruno Scrosati	
EXPERIMENTAL VALUES:		

Solubility of potassium thiocyanate at 40° C

2.47 mol dm⁻³

AUXILIARY INFORMATION			
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:		
The solubility was determined conducto- metrically. The method and the procedure for preparing the saturated solutions and determining their concentration are the same described in the course of the compilation of the lithium chloride-N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.	Reagent grade NH ₄ I was dried in a vacuum desiccator over anhydrous magnesium perchlorate and used without further treatment.		
	The preparation and purification of N-methylacetamide have been described in the compilation of the lithium chloride- N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.		
	ESTIMATED ERROR: Solubility ⁺ 0.5% (compiler).		
	REFERENCES :		

EVALUATOR:		
Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy.		
March 1979		
amides are available from the literature. thylacetamide system has been investigated (1).		
These authors reported a solubility value at 313K of 352.13 g dm ⁻³ of saturated solution The solubility was determined by measuring the conductivity of the saturated solution. The concentration of the latter was obtained from Kohlrausch plots of solutions of the same salt. It has to be remarked that precautions were taken to ensure saturation and to achieve precision in the measurements.		
Nevertheless, because of the lack of data from other sources which may serve as terms of comparison and evaluation, the result of Dawson et.al.is reported as a tentative value of the solubility of ammonium thiocyanate in N-methylacetamide.		
Tentative value of solubility of NH ₄ SCN at 313K. 352.1 g dm ⁻³ of saturated solution		
Since the density of the saturated solution is not known, it is not possible to convert this datum into molal units.		
Eckstrom, H.C. <i>J. Phys. Chem.</i> , <u>1963</u> ,67,		

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N-Methylacetamide	
COMPONENTS: (1) Ammonium thiocyanate; NH ₄ SCN; 1762-95-4 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	ORIGINAL MEASUREMENTS: Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283
VARIABLES:	PREPARED BY:
One temperature: 40°C	Bruno Scrosati
EXPERIMENTAL VALUES:	
4.626 mol dm ⁻³	

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:	
The solubility was determined conductometric- ally. The method and the procedure for preparing	desiccator over anhydrous magnesium perchlorate and used without further treatment. The preparation and purification of N- methylacetamide have been described in	
the saturated solutions and determining their concentration are the same described in the course of the compilation of the lithium chloride-N-methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.		
	ESTIMATED ERROR:	
	Solubility ⁺ 0.5% (compiler)	
	REFERENCES :	

COMPONENTS:

(1) Calcium chloride; CaCl₂; 10043-52-4
 (2) N-methylacetamide; C₂H₇NO; 79-16-3

EVALUATOR:

Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy.

March 1979

CRITICAL EVALUATION:

So far as the evaluator knows, the calcium chloride-N-methylacetamide system has been investigated only by Dawson, Berger, Vaughn and Eckstrom (1).

These authors have reported and approximate value of the solubility (29.97 g dm⁻³ of saturated solution at 313K), determined by addition of small increments of CaCl₂ to a known valume of N-methylacetamide until saturation was visually observed. The solution was then heated to 333K and it was assumed to have reached saturation if solute precipitated upon cooling to 313K. This procedure is open to some criticism and indeed the authors themselves attribute an error of $\frac{+}{2}$ 5% to their datum.

Therefore only an approximate value of the solubility of calcium chloride in Nmethylacetamide may be reported.

Approximate value of solubility of CaCl, at 313K

29.97 g dm $^{-3}$ of saturated solution

Since the density of the saturated solution is not known it is not possible to convert this datum into molal units.

References

 Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283.

COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Calcium chloride; CaCl ₂ ; 10043-52-4 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283	
VARIABLES: One temperature: 40 ⁰ C	PREPARED BY: Bruno Scrosati	
EXPERIMENTAL VALUES:	-1	
Solubility of calcium chloride at 40 [°] C 0.27 mol dm ⁻³		

INFORMATION
SOURCE AND PURITY OF MATERIALS:
Reagent and grade CaC1 ₂ was dried in a vacuum desiccator over anhydrous magnesium
The preparation and the purification of N- methylacetamide have been described in the compilation of the lithium chloride-N- methylacetamide system originally described by Dawson, Berger, Vaughn and Eckstrom to which we refer the reader for details.
ESTIMATED ERROR:
Solubility [±] 5% (authors)
REFERENCES :

COMPONENTS:

Calcium bromide; CaBr₂; 7789-41-5
 N-methylacetamide; C₂H₂NO; 79-16-3

EVALUATOR:

Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy.

March 1979

CRITICAL EVALUATION:

So far as the evaluator knows, the calcium bromide-N-methylacetamide system has been investigated only by Dawson, Berger, Vaughn and Eckstrom (1).

These authors have reported an approximate value of the solubility (79.96 g dm⁻³ of saturated solution at 313K), determined by addition of small incremenets of CaBr₂ to a known volume of N-methylacetamide until saturation was visually observed. The solution was then heated to 333 K and it was assumed to have reached saturation if solute precipitated upon cooling to 313K. This procedure is open to some criticism and indeed the authors themselves attribute an error of \pm 5% to their datum.

Therefore only an approximate value of the solubility of calcium bromide in N-methylacetamide may reported.

Approximate value of solubility of CaBr₂ at 313K

79.96 g dm⁻³ of saturated solution.

Since the density of the saturated solution is not known it is not possible to convert this datum into molal units.

References

 Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., 1963, 67, 281-283.

<i>N</i> -Methyl	acetamide 25
COMPONENTS: (1) Calcium bromide; CaBr ₂ ; 7789-41-5 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	ORIGINAL MEASUREMENTS: Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem., <u>1963</u> , 67, 281-283
VARIABLES: One temperature: 40 [°] C	PREPARED BY: Bruno Scrosati
EXPERIMENTAL VALUES:	L
Solubility of calcium bromide at 40 ⁰ C 0.40 mol dm ⁻³	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
An 'approximate' (authors) value of solubil- ity was determined by progressive additions in small increments of solute to the solvent until saturation was visually observed.	Reagent grade CaBr ₂ was dried in a vacuum desiccator over anyhdrous magnesium perchlorate and used without further treat- ment.
Details on the procedure have been described in the compilation of the sodium bromide-N- methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader.	The preparation and the purification of N- methylacetamide have been described in the compilation of the lithium chloride-N- methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.
	ESTIMATED ERROR:
	Solubility ± 5% (authors)

REFERENCES:

COMPONENTS:		EVALUATOR:
 (1) Strontium chloride; SrCl₂; (2) N-methylacetamide; C₃H₇NO; 	10476-85-4 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy.

March 1979

CRITICAL EVALUATION:

So far as the evaluator knows, the strontium chloride-N-methylacetamide system has been investigated only by Chandra and Gopal(1).

These authors have determined gravimetrically the solubility of strontium chloride at five temperatures using precautions both in assuring high purity of the salt (by repeated crystallizations and heating to a constant weigh) and in reducing contamination of the solvent and the solutions by moisture (by performing all the manipulations in a dry environment).

However, the time allowed for equilibration of the solutions, i.e. 5 or 6 hours, may be in certain cases too short to ensure complete saturation. Indeed, as remarked by the same Gopal in a paper with Bhatnagar (2), electrolytes are generally extremely slow to dissolve in N-methylacetamide. Therefore, the possibility that the data of Chandra and Gopal (1) may be affected by a certain error has to be considered.

Solubility of SrCl, as a function of temperature

T/K	g/kg of solvent
305	161.3
308	166.3
313	174.7
318	182.2
323	193.4

References

- 1. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353.
- 2. Gopal, R.; Bhatnagar, O.N. J. Phys. Chem., 1965,69, 2382-2385.

N-Methyl	acetamide
COMPONENTS :	ORIGINAL MEASUREMENTS:
 (1) Strontium chloride; SrCl₂; 10476-85-4 (2) N-methylacetamide; C₃H₇NO; 79-16-3 	Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353
VARIABLES:	PREPARED BY:
Temperature	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of strontium chloride at f	ive temperatures
t/ ^o C	g/100 g of solvent
32	16.13
35	16.63
40	17.47
45	18.22
50	19.34
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The solubility was determined gravimetric- ally. The method and the procedure have been described in the course of the compilation of the sodium chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.	Strontium chloride has been purified by repeated crystallizations. The salt was then heated to a constant weight to make it anhydrous. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader.
	ESTIMATED ERROR: Solubility [±] 0.5% (authors) Temperature [±] (0.05-0.1) ^o C (authors)
	REFERENCES:

COMPONENTS: (1) Strontium bromide; SrBr₂; 10476-81-0 (2) N-methylacetamide; C₃H₇NO; 79-16-3 EVALUATOR:

Bruno Scrosati Istituto di Chimica Fisica, University of Rome, Italy.

March 1979

CRITICAL EVALUATION:

The system strontium bromide-N-methylacetamide has been investigated by Dawson, Berger, Vaughn and Eckstrom (1) and by Chandra and Gopal (2).

Dawson et al (1) have reported an approximate value of solubility at 313K determined by addition of small increments of SrBr_2 to a known volume of N-methylacetamide until saturation was visually observed. The solution was then heated to 333K and it was assumed to have reached saturation if solute precipitated upon cooling to 313K. This procedure is certainly open to some criticism and indeed the authors themselves attribute an error of $\frac{+}{5}$ % to their approximate data.

Chandra and Gopal (2) have determined gravimetrically the solubility of SrBr₂ in Nmethylacetamide at five temperatures, i.e. 305K, 308K, 313K, 318K and 323K. These authors have used precautions both in assuring high purity and dryness of the salt (by repeated crystallizations and heating to a constant weigh) and in reducing contamination by moisture of the solvent and the solutions (by performing all the manipulations in a dry environment).

Even if the result of Chandra and Gopal (2) may be affected by a too short equilibration time, it still appears more reliable than the approximate value of Dawson et al (1) for a tentative indication of the solubility.

Tentative value of solubility of SrBr, at 313K

430.0 g/kg of solvent.

Solubility of SrBr, as a function of temperature.

For temperatures different than 313K, only the data of Chandra and Gopal (2) are available and these are

т/к	g/kg of solvent
305	415.0
308	421.2
313	430.4
318	439.5
323	449.0

References.

- Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. J. Phys. Chem. <u>1963</u>, 67, 281-283.
- 2. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968,45(4), 351-353.

N-Methylacetamide 3	
COMPONENTS :	ORIGINAL MEASUREMENTS:
 (1) Strontium bromide; SrBr₂; 10476-81-0 (2) N-methylacetamide; C₃H₇NO; 79-16-3 	Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353
VARIABLES:	PREPARED BY:
	Bruno Scrosati
Temperature	
EXPERIMENTAL VALUES:	
Solubility of strontium bromide at five tempe	eratures
t/ ^o C	g/100 g of solvent
32	41.50
35	42.12
40	43.04
45	43.95
50	44.90
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: The solubility was determined gravimetric- ally. The method and the procedure have been described in the course of the compilation	SOURCE AND PURITY OF MATERIALS: Strontium bromide was purified by repeated crystallizations. The salt was then heated to a constant weight to make it anhydrous. The purification of N-methylacetamide has
of the sodium chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.	been described in the course of the compilation of the sodium chloride-N- methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader for details.
	ESTIMATED ERROR:
	Solubility + 0.5% (authors)
	Temperature $\stackrel{+}{=}$ (0.05-0.1) ^o C (authors)
	REFERENCES:

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N-Methylacetamide

COMPONENTS:	OPICINAL MEASUDEMENTS .
 (1) Strontium bromide; SrBr₂; 10476-81-0 (2) N-methylacetamide; C₃H₇NO; 79-16-3 	ORIGINAL MEASUREMENTS: Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. <i>J.Phys.Chem.</i> , <u>1963</u> ,67, 281-283
VARIABLES:	PREPARED BY:
One temperature: 40 [°] C	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of strontium bromide at 40 ⁰ C 1.64 mol dm ⁻³	
1.64 mol dm	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
An 'approximate' (authors) value of solubility was determined by progressive additions in small increments of solute to the solvent until saturation was visually observed.	Reagent grade SrBr, was dried in a vacuum desiccator over anhydrous magnesium perchlorate and used without further treatment.
Details on the procedure have been described in the compilation of the sodium bromide-N- methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader.	The preparation and the purification of N- methylacetamide have been described in the compilation of the lithium chloride-N- methylacetamide system, originally measured by Dawson, Berger, Vaughn and Eckstrom, to which we refer the reader for details.
	ESTIMATED ERROR:
	Solubility ± 5% (authors)
	REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Barium chloride; BaC1 ₂ ; 10361-37-2 (2) N-methylacetamide; C ₃ H ₇ NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy.

March 1979.

CRITICAL EVALUATION:

So far as the evaluator knows the barium chloride-N-methylacetamide system has been investigated only by Chandra and Gopal (1).

These authors have determined gravimetrically the solubility of barium chloride at five temperatures using precautions both in assuring high purity and dryness of the salt (by repeated crystallizations and heating to a constant weight) and in reducing contamination by moisture of the solvent and the solutions (by performing all the manipulations in a dry environment).

However, the time allowed for equilibration of the solutions, i.e. 5 or 6 hours, may be in certain cases too short to ensure complete saturation. Indeed, as remarked by the same Gopal in a paper with Bhatnagar (2), electrolytes are generally extremely slow to dissolve in N-methylacetamide. Therefore, the possibility that the data of Chandra and Gopal (1) may be affected by a certain error has to be considered.

Solubility of BaCl, as a function of temperature

T/K	g/kg of solvent
305	124.7
308	127.3
313	132.3
318	137.3
323	141.7

References.

- 1. Chandra, D.; Gopal, R. J. Indian Chem. Soc. 1968, 45(4), 351-353.
- 2. Gopal, R.: Bhatnagar, O.N. J. Phys. Chem., 1965, 69, 2382-2385

	ylacetamide
COMPONENTS:	ORIGINAL MEASUREMENTS:
 Barium chloride; BaCl₂; 10361-37-2 N-methylacetamide; C₃H₇NO; 79-16-3 	Chandra, D.; Gopal, R. J.Indian Chem. Soc., <u>1968</u> , 45(4), 351-353.
VARIABLES:	PREPARED BY:
Temperature	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of barium chloride at five tempe	ratures
t/ ^o C	g/100 g of solvent
32	12.47
35	12.73
40	13.23
45	13.73
50	14.17
AUXILIAF	RY INFORMATION
METHOD / APPARATUS / PROCEDURE :	
	SOURCE AND PURITY OF MATERIALS:
The solubility was determined gravimetrical The method and the procedure have been des- cribed in the course of the compilation of the sodium chloride-N-methlyacetamide system originally measured by Chandra and Gopal, to which we refer the reader for details.	 ly. Barium chloride was purified by repeated crystallizations. The salt was then heater to a constant weight to make it anhydrous. m, The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N-methylacetamide system, originally measured by Chandra and Gopal, to which we refer the reader. ESTIMATED ERROR:
The solubility was determined gravimetrical The method and the procedure have been des- cribed in the course of the compilation of the sodium chloride-N-methlyacetamide system originally measured by Chandra and Gopal, to	 ly. Barium chloride was purified by repeated crystallizations. The salt was then heate to a constant weight to make it anhydrous. m, The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N-methylacetamide system, originally measured by Chandrand Gopal, to which we refer the reader.
The solubility was determined gravimetrical The method and the procedure have been des- cribed in the course of the compilation of the sodium chloride-N-methlyacetamide system originally measured by Chandra and Gopal, to	 Barium chloride was purified by repeated crystallizations. The salt was then heater to a constant weight to make it anhydrous. The purification of N-methylacetamide has been described in the course of the compilation of the sodium chloride-N-methylacetamide system, originally measured by Chandr and Gopal, to which we refer the reader. ESTIMATED ERROR: Solubility [±] 0.5% (authors)

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COMPONENTS:	EVALUATOR:
(1) Barium bromide; BaBr ₂ 10553-31-8 (2) N-methylacetamide; C _{3^H7} NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy.
	March 1979

CRITICAL EVALUATION:

So far as the evaluator knows, the barium bromide-N-methylacetamide system has been investigated only by Chandra and Gopal (1).

These authors have determined gravimetrically the solubility of barium bromide at five temperatures using precautions both in assuring high purity and dryness of the salt (by repeated crystallizations and heating to a constant weight) and in reducing contamination by moisture of the solvent and the solutions (by performing all the manipulations in a dry environment).

However, the time allowed for equilibration of the solutions, i.e. 5 or 6 hours, may be in certain cases too short to ensure complete saturation. Indeed, as remarked by the same Gopal in a paper with Bhatnagar (2), electrolytes are generally extremely slow to dissolve in N-methylacetamide. Therefore, the possibility that the data of Chandra and Gopal (1) may be affected by a certain error has to be considered.

Solubility of BaBr, as a function of temperature

T/K	g/kg of solvent
305	385.0
308	388.1
313	394.3
318	400.6
323	406.7

References.

- 1. Chandra, D.; Gopal, R. J. Indian Chem. Soc., 1968, 45(4), 351-353.
- 2. Gopal, R.; Bhatnagar, O.N. J. Phys. Chem., 1965, 69, 2382-2385.

acetamide
ORIGINAL MEASUREMENTS:
Chandra, D.; Gopal, R. J. Indian Chem. Soc., <u>1968</u> , 45(4), 351-353.
PREPARED BY:
Bruno Scrosati
ures
g/100 g of solvent
38.50
38.81
39.43
40.06
40.67
INFORMATION
SOURCE AND PURITY OF MATERIALS:
Barium bromide was purified by repeated crystallizations. The salt was then heated to a constant weight to make it anhydrous. The purification of N-methylacetamide has been described in the course of the compil- ation of the sodium chloride-N-methylace- tamide system, originally measured by Chandra and Gopal, to which we refer the reader for details. ESTIMATED ERROR: Solubility [±] 0.5% (authors) Temperature [±] (0.05-0.1) ^o C (authors) REFERENCES:

COMPONENTS:	EVALUATOR:
<pre>(1) Lithium chloride; LiCl; 7447-41-8 (2) N, N-dimethylacetamide; C₄H₉NO; 127-19-5</pre>	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

The lithium chloride-N, N-dimethylacetamide system has been examined by Pistoia and Scrosati (1), by Lesteva and Chernaya (2) and by Federov et. al. (3).

The result of the latter authors (2.00 mol dm^{-3} at 295-298 K) is rejected since no purification of the solute and the solvent was described and no control of the temperature was attempted.

The solubility determined by Pistoia and Scrosati (1) at 298 K (86.0 g/kg of solvent) appears to be a more accurate value since these authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

Lesteva and Chernaya (3) have examined the ternary lithium chloride-dimethylacetamidewater system and found that several compounds are present in equilibrium with the liquid phase. Four compounds of lithium chloride with dimethylacetamide (DMA) were detected in the system, with the following compositions, 4LiCl.DMA, 1.5LiCl.DMA, LiCl.DMA and LiCl.2DMA, respectively.

When describing their solubility studies, Lesteva and Chernaya (3) neither reported the source and the purity of the materials nor the purification of the solvent. However, the procedure to obtain the solubility values appears accurate both in controlling the temperature of operation and in determining the LiCl concentration in the saturated solutions.

The authors (3) have reported values of solubility of LiCl in N, N-dimethylacetamide at three temperatures, i.e. 293K, 313K and 333K respectively, with the results reported in the following table.

<u>T/K</u>	mo1/kg
293	2.03
313	2.52
333	2.92

The solubility data may be represented as function of temperature by the equation (evaluator)

 $\log c (mo1/kg) = -386.522/(T/K) + 1.629$

The calculated value at 298 K, i.e. 91.1 g/kg of solvent, is in fair agreement with that reported by Pistoia and Scrosati (1). As recommended value is then taken the average between the datum of Pistoia and Scrosati (1) and that of Lesteva and Chernaya (3).

Recommended value of solubility of LiCl at 298 K.

88-3 g/kg of solvent

(Continued)

AEM VOL 11-L*

COMPONENTS :		EVALUATOR:
 Lithium chloride: LiCl; N, N-dimethylacetamide; 127-19-5 	7447-41-8 C ₄ H ₉ NO;	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

CRITICAL EVALUATION: (continuation)

Tentative values of solubility of LiCl as function of temperature.

For temperatures different than 298 K, only the data of Lesteva and Chernaya (3) are available. They are

<u>T/K</u>	g/kg of solvent
293	86.05
313	126.8
333	123.8

References

1. Pistoia, G.; Scrosati, B. Ric. Sci., 1967, 37, 1173-1177.

2. Lesteva, T. M.; Chernaya, V. I. Russ. J. Inorg. Chem., 1978, 23(9), 1416-1417.

3. Federov, A. A.; Sokolov, L. B.; Savinov, V. M.; Shilionskaya, M. A.;

Filatova, N. I. Zh. Prikl. Khim., <u>1971</u>, 44(10), 2361-2362.

N, N-Dimeth	nylacetamide 3
COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Lithium chloride; LiCl; 7447-41-8 (2) N, N-dimethylacetamide; C₄H₉NO; 127-19-5 	Lesteva, T.M.: Chernaya, V.I. Russ.J.Inorg.Chem., <u>1978</u> , 23(9), 1416-1417 Translated from Z.Neorg.Khim., <u>1978</u> , 23, 2556-2558.
VARIABLES:	PREPARED BY:
Temperature	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of LiCl at three temperatures.	
<u>t/^oC</u> <u>mol/</u> 20.0 40.0	kg of solvent 2.03 2.52
60.0	2.92
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: The solubility value was determined by mixing the halide with the solvent in a vessel maintained at constant temperature. The solution was stirred until equilibrium was established in the system. The amount of time allowed for reaching this condition is not reported by the authors.	SOURCE AND PURITY OF MATERIALS: Not reported.
Specimen of the liquid phase were analysed for lithium chloride argentometrically.	
The composition of the solid phase was determined from the Schreinemakers rays (1).	
	ESTIMATED ERROR: Solubility ⁺ 0.2-0.5%
	Solubility - $0.2-0.5\%$ Temperature $\frac{1}{2}$ 0.05 K (authors)
	REFERENCES :
	(1) Schreinemakers, F.A.H., J. Phys. Chem. <u>1906</u> , 55, 73-75

COMPONENTS :	ORIGINAL MEASUREMENTS:
 (1) Lithium chloride; LiCl; 7447-41-8 (2) N, N-dimethylacetamide; C₄H₉NO;	Pistoia, G.; Scrosati, B.
127-19-5	<i>Ric.Sci.</i> , <u>1967</u> , 37, 1173-1177
VARIABLES:	PREPARED BY:
One temperature: 25 [°] C	Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of lithium chloride at 25°C

8.60 g/100g of solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: The method and the apparatus for the solubility measurements were those described in a previous paper from the same laboratory (1). The solubility value was determined by dissolving the halide in about $20-25 \text{ cm}^3$ of solvent contained in a 50 cm² pyrex flask with a standard taper joint. The addition of the salt was continued until apparent saturation was reached. The solution was then stirred at room temperature for several hours and finally left for three or more days in a stirred thermostatic bath. An aliquot of the saturated solution was then collected with the aid of a siphon provided with a terminal G-3 gooch. This solution was diluted with water in the 1/10 ratio and the amount of the dissolved salt was determined by volumetric Mohr titration. The reliability of the method was checked by dissolving known amounts of the salt and then titrating the resulting solution with the procedure above described. 0.1400 g of LiCl were added and 0.1395g found, thus showing an accuracy of 0.4% (authors).

SOURCE AND PURITY OF MATERIALS: Lithium chloride, reagant grade commercial product, was dried under vacuum. Reagent grade dimethylacetamide was purified by fractional distillation under reduced pressure (2mm Hg). The final product had a specific conduct-ance of 2.10^{-5} S m⁻¹, in good agreement and Sears (2). i.e. (0.8-2), 10 S m⁻¹. and Sears (2), i.e. (0.8-2), 10 To avoid contamination with the atmosphere the solution was prepared in a dry-box. ESTIMATED ERROR: Solubility [±] 0.4% Temperature ⁺ 0,05°C (authors) REFERENCES: (1) Pistoia, G.; Pecci, G.; Scrosati, B, Ric.Sci., 1967, 37, 1167-1172

(2) Lester, G.; Gover, T.; Sears, P. J. Phys. Chem., <u>1956</u>, 60 1076-1079.

COMPONENTS :	EVALUATOR:
 (1) Lithium bromide; LiBr; 7550-35-8 (2) N, N-dimethylacetamide; C₄H₉NO; 127-19-5 	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

The system lithium bromide-N, N-dimethylacetamide has been examined by Pistoia and Scrosati (1) and by Federov et al. (2).

The result of the latter authors $(2.60 \text{ mol dm}^{-3})$ is rejected since no purification of the solute and the solvent was described and the temperature (reported as 295-298 K) was not controlled.

The solubility determined by Pistoia and Scrosati(1) at 298 K (262 g/kg of solvent) appears to be more accurate since these authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

Nevertheless, because of the lack of reliable data from other sources which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value.

Tentative value of solubility of LiBr at 298 K

262 g/kg of solvent

References

1. Pistoia, G.; Scrosati, B.; Ric. Sci., 1967, 37, 1173-1177.

 Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Zh.Prikl.Khim.* 1971, 44(10), 2361-2362.

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Lithium bromide; LiBr; 7550-35-8	Pistoia G.; Scrosati, B.
(2) N, N-dimethylacetamide; C ₄ H _o NO;	Ric.Sci., <u>1967</u> , 37 1173-1177.
127-19-5	
VARIABLES:	PREPARED BY:
One temperature: 25°C	Bruno Scrosati
•	
EXPERIMENTAL VALUES:	
Solubility of lithium bromide at 25°C	
26.2 g/100	g of solvent
、 	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The method and the apparatus for obtaining	Lithium bromide, reagent grade commercial
the saturated solution and determining the	product, was dried under vacuum.
solubility was described in the course of the compilation of the lithium chloride-N, N-	N,N-dimethylacetamide was purilied by
dimethylacetamide system, originally	fractional distillation under reduced pressure (2mm Hg).
measured by Pistoia and Scrosati, to which we refer the reader for details.	
	ESTIMATED ERROR: Solubility ⁺ 0.4%
	Temperature $\stackrel{+}{=}$ 0.05 °C (authors).
	Tembergrane 0.02 0 (anthors).
	REFERENCES :

 (1) Lithium iodide; LiI; 10377-51-2 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5 Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. 	EVALUATOR:
May 1979	acetamide; C ₄ H ₉ NO; Istituto di Chimica Fisica, University of Rome, Italy.

Very few solubility data of halides in acetamides are available from the literature. In particular, the system lithium iodide-N,N-dimethylacetamide has been examined only by Fedorov et.al. (1), who reported a solubility value of 0.95 mol/liter at 295-298 K

In other systems, where more reliable data from other sources are available (see for instance the lithium chloride-N,N-dimethylacetamide one), the results of Fedorov et.al.(1) are rejected since these are affected by lack of information on the purification of the solute and the solvent and by no control of the temperature.

However, in cases such as the present one, where the data of Fedorov et.al. (1) are the only ones available, the results of these authors have been compiled and reported as very approximate values of solubility.

Approximate value of solubility of LiI at 298 K 43 g dm⁻³ of saturated solution.

Since the density of the saturated solution is not known it is not possible to convert this datum into molal units.

References

 Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. Zh. Prikl. Khim., <u>1971</u>, 44 (10), 2361-2362

COMPONENTS :	ORIGINAL MEASUREMENTS:
<pre>(1) Lithium iodide; LiI; 10377-51-2 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5</pre>	Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M Shilionskaya, M.A.; Filatova, N.I. Z.Prikl.Khim., 1971, 44(10), 2361-2362.
VARIABLES: One temperature: 22-25 [°] C	PREPARED BY: Bruno Scrosati
EXPERIMENTAL VALUES:	

Solubility of lithium iodide at 22-25°C.

0.95 mol dm⁻³

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: The saturated solution was prepared at 'ambient' temperature (22-25°C) by adding excess of solute to freshly distilled solvent. The solution was kept in a desiccator for three days. The amount of dissolved salt in the solution was then evaluated by titrating the iodide ions with standard mercuric solution.	SOURCE AND PURITY OF MATERIALS: Lithium iodide was dried under vacuum at 120°C for 6 hours. No details of the purification of N,N-dimethylacetamide are reported.
	ESTIMATED ERROR:
	Solubility [±] 10% (compiler)
	REFERENCES:

COMPONENTS:	EVALUATOR:
 (1) Sodium chloride; NaCl; 7647-14-5 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5 	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

The sodium chloride-N,N-dimethylacetamide system has been investigated by Pistoia and Scrosati (1) and by Fedorov et.al. (2).

The latter authors stated that sodium chloride is 'not soluble' in N,N-dimethylacetamide. This statement is however rejected since no purification of the solvent was described and the temperature not controlled in the investigation of Fedorov et.al. (2).

The solubility determined by Pistoia and Scrosati (1) at 298 K (0.2 g/kg of solvent) appears to be more accurate since these authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

Nevertheless, because of the lack of reliable data from other sources which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value of the solubility of sodium chloride in N,N-dimethylacetamide at 298 K.

Tentative value of the solubility of NaCl at 298 K 0.2 g/kg of solvent.

References

1. Pistoia, G.; Scrosati, B.; Ric.Sci., 1967, 37, 1173-1177.

 Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Zh.Prikl Khim.*, <u>1971</u>, 44(10), 2361-2362.

318 N, N-Dimeth	nylacetamide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; 7647-14-5 (2) N,N-dimethylacetamide; C ₄ H ₉ NO; 127-19-5	Pistoia, G.; Scrosati, B. <i>Ric.Sci.</i> , <u>1967</u> , <i>3</i> 7, 1173-1177.
VARIABLES:	PREPARED BY:
One temperature: 25 [°] C	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of sodium chloride at 25 ⁰ C	
0.02 g/100 g	of solvent
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The method and the apparatus for obtaining the saturated solution and determining	Sodium chloride, reagent grade commercial product, was dried under vacuum.
the solubility was described in the course of the compilation of the lithium chloride- N,N-dimethylacetamide system, originally measured by Pistoia and Scrosati, to which we refer the reader for details.	N,N-dimethylacetamide was purified by fractional distillation under reduced pressure (2 mm Hg).
	ESTIMATED ERROR:
	Solubility $\stackrel{+}{=}$ 0.4%
	Temperature ⁺ 0.05 [°] C (authors)
	REFERENCES :

COMPONENTS:	EVALUATOR:
<pre>(1) Sodium bromide; NaBr; 7647-15-6 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5</pre>	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

Very few solubility data of halides in acetamides are available from the literature. In particular the system sodium bromide-N,N-dimethylacetamide has been investigated only by Pistoia and Scrosati (1) who have reported a solubility value at 298 K of 65.1 g/kg of solvent.

The result appears reliable since the authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

However, because of the lack of data from other sources, which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value of the solubility of sodium bromide in N, N-dimethylacetamide at 298 K.

Tentative value of solubility of NaBr at 298 K 65.1 g/kg of solvent.

References

1. Pistoia, G.; Scrosati, B., Ric. Sci., 1967, 37, 1173-1177.

COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; 7647-15-6	Pistoia, G.; Scrosati, B.,
(2) N,N-dimethylacetamide; C ₄ H ₉ NO;	<i>Ric.Sci.</i> , <u>1967</u> , <i>37</i> , 1173–1177
127-19-5	
VARIABLES:	PREPARED BY:
One temperature: 25 ⁰ C	Bruno Scrosati
·	
EXPERIMENTAL VALUES:	
Solubility of sodium bromide at 25 ⁰ C.	
Solubility of source browide at 25 c.	
6.51 g/100 g	; of solvent.
0.51 g/ 100 g	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The method and the procedure for obtaining	Sodium bromide, reagent grade commercial
the saturated solution and determining the solubility was described in the course of the	product, was dried under vacuum.
compilation of the lithium chloride-N,	N,N-dimethylacetamide was purified by fractional distillation under reduced
N-dimetylacetamide system, originally measured by Pistoia and Scrosati, to which	pressure (2 mmHg).
we refer the reader for details.	
	ESTIMATED ERROR:
	Solubility + 0.4%
	Temperature $\stackrel{+}{=} 0.05^{\circ}$ C (authors)
	REFERENCES:

COMPONENTS: (1) Sodium iodide; NaI; 7681-82-5 (2) N,N-dimethylacetamide; C ₄ H ₉ NO; 127-19-5	EVALUATOR: Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979
CRITICAL EVALUATION: The sodium iodide-N,N-dimethylacetamide syst Scrosati (1) and by Fedorov et.al. (2). The result of the latter authors (0.40 mol of purification of the solvent was described an attempted.	lm ⁻³ at 295-298 K) is rejected since no
The solubility determined by Pistoia and Scrosati (1) at 298 K (346 g/kg of solvent) appears to be more accurate since these authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.	

Nevertheless, because of the lack of reliable data from other sources, which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value of the solubility of sodium iodide in N,N-dimethylacetamide at 298K.

Tentative value of solubility of NaI at 298 K 346 g/kg of solvent.

References

1. Pistoia, G.; Scrosati, B. Ric. Sci., 1967, 37, 1173-1177.

 Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. Zh. Prikl. Khim., 1971, 44(10), 2361-2362.

COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Sodium iodide; NaI; 7681-82-5 (2) N,N-dimethylacetamide; C ₄ H ₉ NO; 127-19-5	Pistoia, G.; Scrosati, B. <i>Ric.Sci.</i> , <u>1967</u> , <i>37</i> , 1173-1177.	
VARIABLES: One temperature: 25 [°] C	PREPARED BY: Bruno Scrosati	
EXPERIMENTAL VALUES: Solubility of sodium iodide at 25 ⁰ C.		-

34.6 g/100 g of solvent.

AUXILIARY	INFORMATION

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The method and the procedure for obtaining the saturated solution was described in the course of the compilation of the lithium chloride-N,N-dimethylacetamide system, to which we refer the reader for details. The amount of dissolved NaI in the saturated solution was determined by Volhard titration	Sodium iodide, reagent grade commercial product, was dried under vacuum. N,N-dimethylacetamide was purified by fractional distillation under reduced pressure (2 mmHg).
	ESTIMATED ERROR: Solubility ⁺ 0.4%.
	Solubility - 0.4%. Temperature $\stackrel{+}{=}$ 0.05°C (authors).
	REFERENCES:

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COMPONENTS:	EVALUATOR:
 (1) Sodium thiocyanate; NaSCN; 540-72- (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5 	-7 Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

Very few solubility data of pseudohalides in acetamides are available in the literature. In particular the sodium thiocyanate-N,N-dimethylacetamide system has been investigated only by Fedorov et.al. (1), who reported a value of 1.78 mol dm⁻³ at 295:298 K.

In other systems, where more reliable data from other sources are available, the results of Fedorov et.al. (1) are rejected since no purification of the solvent nor control of temperature was attempted by these authors in their investigation.

However, in the cases like the present one, where the data of Fedorov et.al. (1) are the only available, the results of these authors have been compiled and reported as very approximate values of solubility.

Approximate value of solubility of NaSCN at 298 K

144 g dm⁻³ of saturated solution.

Since the density of the saturated solution is not known it is not possible to convert this datum into molal units.

References

 Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. Z. Prikl. Khim., 1971, 44(10), 2361-2362. N,N-Dimethylacetamide

COMPONENTS:	ORIGINAL MEASUREMENTS:
<pre>(1) Sodium thiocyanate; NaSCN; 540-72-7 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5</pre>	Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. Z. Prikl. Khim., <u>1971</u> , 44(10), 2361-2362.
VARIABLES: One temperature: 22-25 ⁰ C	PREPARED BY: Bruno Scrosati
EXPERIMENTAL VALUES:	

Solubility of sodium thiocyanate at 22-25°C

1.78 mol dm⁻³

	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The saturated solution was prepared at 'ambient' temperature (22-25°C) adding excess of solute to freshly distilled solvent. The solution was kept in a desiccator for three days. The amount of dissolved salt in the solution was the evaluated by titrating the thiocyanate ions with standard mercuric solution.	Sodium thiocyanate was dried under vacuum. No details on the purification of N,N- dimethylacetamide are reported.
	ESTIMATED ERROR:
	Solubility [±] 10% (compiler)
	REFERENCES :

1	COMPONENTS :	EVALUATOR:
	 (1) Potassium chloride; KC1; 7447-40-7 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5 	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

The potassium chloride-N,N-dimethylacetamide system has been investigated by Pistoia and Scrosati (1) and by Fedorov et.al. (2).

The latter authors stated that potassium chloride is 'not soluble' in N,N-dimethylacetamide. This statement, however, is rejected since no purification of the solvent was described and no control of the temperature was attempted in the investigation of Fedorov et.al. (2).

The solubility determined by Pistoia and Scrosati (1) at 298 K (0.11 g/kg of solvent) appears to be more accurate since these authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

Nevertheless, because of the lack of reliable data from other sources which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value of the solubility of potassium chloride in N,N-dimethylacetamide at 298 K.

Tentative value of solubility of KC1 at 298 K 0.11 g/kg of solvent

References

- 1. Pistoia, G.; Scrosati, B.; Ric.Sci., 1967, 37, 1173-1177
- Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. Z. Prikl. Khim., 1971, 44(10), 2361-2362.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KC1; 7447-40-7	Pistoia, G; Scrosati, B. <i>Ric. Sci</i> ., <u>1967</u> , 37, 1173 - 1177
(2) N,N-dimethylacetamide; C ₄ H ₉ NO;	
127-19-5	
VARIABLES:	PREPARED BY:
One temperature: 25°C	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of potassium chloride at 25 ⁰ C	
0.01 g/100	g of solvent
AUXILIARY	INFORMATION
METHOD / AP PARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS;
The method and the procedure for obtaining	Potassium chloride, reagent grade commercial
the saturated solution and determining the solubility were described in the course of	product, was dried under vacuum.
the compilation of the lithium-chloride-N,	N.N-dimethylacetamide was purified by fractional distillation under reduced
N-dimethylacetamide system, originally measured by Pistoia and Scrosati, to which	pressure (2 mmHg).
we refer the reader for details.	
	ESTIMATED ERROR:
	Solubility ± 0.4%
	Temperature $\stackrel{+}{=}$ 0.05°C (authors)
	REFERENCES:

COMPONENTS:	EVALUATOR:
 (1) Potassium bromide; KBr; 7558-02-3 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5 	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

The potassium bromide-N,N-dimethylacetamide system has been investigated by Pistoia and Scrosati (1) and by Fedorov et.al. (2).

Pistoia and Scrosati (1) reported a solubility at 298 K of 3.9 g/kg of solvent. This value appears reliable since the authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

The datum of Fedorov et.al. (2) at 295-298 K (0.03 mol dm^{-3}) cannot be used as a reliable quantity of comparison and evaluation, since these authors have not attempted to purify the solvent nor to control the temperature.

The datum of Fedorov et.al. (2) is therefore rejected and the result of Pistoia and Scrosati (1) is reported as a tentative value of the solubility of potassium bromide in N,N-dimethylacetamide at 298 K.

Tentative value of solubility of KBr at 298 K 3.9 g/kg of solvent

References

1. Pistoia, G.; Scrosati, B.; Ric. Sci., 1967, 37, 1173-1177.

Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I.
 Prikl. Khim., <u>1971</u>, 44(10), 2361-2362.

COMPONENTS:	ORIGINAL MEASUREMENTS:
 (1) Potassium bromide; KBr; 7558-02-3 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5 	Pistoia, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1173-1177.
VARIABLES: One temperature; 25 ⁰ C	PREPARED BY: Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of potassium bromide at 25 ⁰ C	
0.39 g/100 g	of solvent
AUXILIARY	INFORMATION
METHOD APPARATUS/PROCEDURE: The method and the procedure for obtaining the saturated solution and determining	SOURCE AND PURITY OF MATERIALS: Potassium bromide, reagent grade commercial product, was dried under vacuum.
the solubility were descrived in the course of the compilation of the lithium chloride-N,N-dimethylacetamide system, originally measured by Pistoia and Scrosati, to which we refer the reader for details.	N,N-dimethylacetamide was purified by fractional distillation under reduced pressure (2 mmHg).
	ESTIMATED ERROR:
	Solubility [±] 0.4% Temperature [±] 0.05 [°] C (authors)
	REFERENCES :

į	COMPONENTS:	EVALUATOR:
	<pre>(1) Potassium iodide; KI; 7681-11-0 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5</pre>	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

The potassium iodide-N,N-dimethylacetamide system has been investigated by Pistoia and Scrosati (1) and by Fedorov et.al. (2).

The latter authors reported a solubility at 295-298 K of 0.05 mol dm^{-3} . However, this datum is rejected since no purification of the solvent nor control of the temperature was attempted in the investigation of Fedorov et.al. (2).

The solubility determined by Pistoia and Scrosati (1) at 298 K (15.4 g/kg of solvent) appears to be more accurate since these authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

Nevertheless, because of the lack of reliable data from other sources, which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value of the solubility of potassium iodide in N,N-dimethylacetamide at 298 K.

Tentative value of solubility of KI at 298 K

15.4 g/kg of solvent.

References

- 1. Pistoia, G.; Scrosati, B.; Ric. Sci., 1967, 37, 1173-1177.
- Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I.
 Prikl. Khim., 1971, 44(10), 2361-2362.

330 N, N-Dimeth	nylacetamide
COMPONENTS :	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; 7681-11-0 (2) N,N-dimethylacetamide; C ₄ H ₉ NO; 127-19-5	Pistoia, G.; Scrosati, B. <i>Ric. Sci</i> ., <u>1967</u> , 37, 1173-1177.
VARIABLES:	PREPARED BY:
One temperature: 25 [°] C	Bruno Scrosati
EXPERIMENTAL VALUES:	<u> </u>
Solubility of potassium iodide at 25 ⁰ C	
1.54 g/100	g of solvent.
AUXILIARY INFORMATION	
METHOD / APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
The method and the procedure for obtaining the saturated solution were described in	Potassium iodide, reagant grade commercial product, was dried under vacuum.
the course of the compilation of the lithium chloride-N,N-dimethylacetamide system, originally measured by Pistoia and Scrosati, to which we refer the reader for details.	N,N-dimethylacetamide was purified by fractional distillation under reduced pressure (2 mmHg).
The amount of dissolved KI in the saturated solution was determined by Volhard titration.	
	ESTIMATED ERROR:
	Solubility [±] 0.4%
	Temperature $\pm 0.05^{\circ}$ C (authors)

REFERENCES:

COMPONENTS:	EVALUATOR:
 (1) Potassium thiocyanate; KSCN; 333-20-0 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5 	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

Very few solubility data of pseudohalides in acetamides are available from the literature. In particular the potassium thiocyanate-N,N-dimethylacetamide system has been investigated only by Fedorov et.al. (1), who reported a value of 1.10 mol dm^{-3} at 295-298 K.

In other systems, where more reliable data from other sources are available, the results of Fedorov et.al. (1) are rejected since no purification of the solvent nor control of the temperature was attempted by these authors in their investigation.

However, in the cases like the present one, where only the data of Fedorov et.al. (1) are available, the results of these authors have been compiled and reported as very approximate values of solubility.

Approximate value of solubility of KSCN at 298 K

107 g dm⁻³ of saturated solution.

Since the density of the saturated solution is not known it is not possible to convert this datum into molal units.

References

Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shalionskaya, M.A.; Filatova, N.I.
 2. Prikl. Khim., <u>1971</u>, 44(10), 2361-2362.

332 <i>N, N</i> -Dim	nethylacetamide
COMPONENTS :	ORIGINAL MEASUREMENTS:
 Potassium thiocyanate; KSCN; 333-20-0 	Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M. Shilionskaya, M.A.; Filatova, N.I.
<pre>(2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5</pre>	Z. Prikl. Khim., <u>1971</u> , 44(10), 2361-2362.
VARIABLES :	PREPARED BY:
One temperature: 22:25°C	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of potassium thiocyanate at 22-	-25 [°] C
1.10	mol dm^{-3}
	ARY INFORMATION
METHOD /APPARATUS / PROCEDURE :	SOURCE AND PURITY OF MATERIALS:
The saturated solution was prepared at 'ambient' temperature (22:25°C) adding excess of solute to freshly distilled	Potassium thiocyanate was dried under vacuum. No details on the purification of N,N-dimethylacetamide are reported.

'ambient' temperature $(22\div25^{\circ}C)$ adding excess of solute to freshly distilled solvent. The solution was kept in a desiccator for three days. The amount of dissolved salt in the solution was then evaluated by titrating the thiocyanate ions with standard mercuric solution.

> ESTIMATED ERROR: Solubility ⁺10% (compiler)

REFERENCES:

COMPONENTS :	EVALUATOR:
<pre>(1) Magnesium chloride; MgCl₂; 7786-30-3 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5</pre>	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

The magnesium chloride-N,N-dimethylacetamide system has been investigated by Pistoia and Scrosati (1) and by Fedorov et.al. (2).

The latter authors reported a solubility at 295-298 K of 0.25 mol dm⁻³. However, this datum is rejected since no purification of the solvent nor control of the temperature was attempted in the investigation of Fedorov et.al. (2).

The solubility determined by Pistoia and Scrosati (1) at 298 K (35.4 g/kg of solvent) appears to be more accurate since these authors have used precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

Nevertheless, because of the absence of reliable data from other sources, which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value of the solubility of magnesium chloride in N,N-dimethylacetamide at 298 K.

Tentative value of solubility of MgCl, at 298 K

35.4 g/kg of solvent.

References

1. Pistoia, G.; Scrosati, B.; Ric. Sci., 1967, 37, 1173-1177.

Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I.
 Z. Prikl. Khim., 1971, 44(10), 2361-2362.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Magnesium chloride; MgCl ₂ ; 7786-30-3	Pistoia, G.; Scrosati, B.
(2) N,N-dimethylacetamide; $C_{L}^{2}H_{Q}NO;$	Ric. Sci., <u>1967</u> , 37, 1173–1177
127-19-5	
127-19-5	
VARIABLES:	PREPARED BY:
One temperature: 25°C	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of magnesium chloride at 25°C	
3.54 g/100	g of solvent
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
me maked and the suscedure for sharing	
The method and the procedure for obtaining the saturated solution were described in the	Magnesium chloride, reagent grade commercial product, was dried under vacuum.
course of the compilation of the lithium	
chloride-N,N-dimethylacetamide system,	N,N-dimethylacetamide was purified by fractional distillation under reduced
originally measured by Pistoia and Scrosati	pressure (2mmHg).
to which we refer the reader for details.	
The amount of dissolved MgCl in the	
saturated solution was determined by Fajans titration, using dichloro-	
fluorescein as indicator.	
	ESTIMATED ERROR:
	Solubility [±] 0.4%
	Temperature $\stackrel{+}{=}$ 0.05°C (authors)
	Temperature 0.05 C (authors)
	REFERENCES :
]	

COMPONENTS:	EVALUATOR:
<pre>(1) Magnesium iodide; MgI₂; 10377-58-9 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5</pre>	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

Very few solubility data of halides in acetamides are available in the literature. In particular the system magnesium iodide-N,N-dimethylacetamide has been investigated only by Pistoia and Scrosati (1) who have reported a solubility value at 298 K of 570 g/kg of solvent.

The result appears reliable since the authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature. However, because of the lack of data from other sources, which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value of the solubility of magnesium iodide in N,N-dimethylacetamide at 298 K.

Tentative value of solubility of MgI, at 298 K

570 g/kg of solvent.

References

1. Pistoia, G.; Scrosati, B. Ric. Sci., 1967, 37, 1173-1177

336 N, N-Dimethylacetamide			
COMPONENTS: (1) Magnesium iodide; MgI ₂ ; 10377-58-9 (2) N,N-dimethylacetamide; C ₄ H ₉ NO; 127-19-5	ORIGINAL MEASUREMENTS: Pistoia, G.; Scrosati, B. <i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1173-1177		
VARIABLES: One temperature: 25 [°] C	PREPARED BY: Bruno Scrosati		
EXPERIMENTAL VALUES: Solubility of magnesium iodide at 25 ⁰ C			
57.0 g/100	g of solvent		
AUXILIARY INFORMATION			
METHOD/APPARATUS/PROCEDURE: The method and the procedure for obtaining the saturated solution were described in the course of the compilation of the lithium chloride-N,N-dimethylacetamide system, originally measured by Pistoia and Scrosati, to which we refer the reader for details. The amount of dissolved MgI ₂ in the saturated solution was determined by Volhard titration.	SOURCE AND PURITY OF MATERIALS: Magnesium iodide, reagent grade commercial product, was dried under vacuum. N,N-dimethylacetamide was purified by fractional distillation under reduced pressure (2 mmHg).		
	ESTIMATED ERROR: Solubility [±] 0.4% Temperature [±] 0.05 [°] C (authors)		
	REFERENCES :		

COMPONENTS:	EVALUATOR:
(1) Calcium chloride; CaCl ₂ ; 10043-52-4	Bruno Scrosa
(2) N,N-dimethylacetamide; C ₄ H ₉ NO;	Bruno Scrosa Istituto di University o
127-19-5	Mar. 1070

Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

CRITICAL EVALUATION:

The calcium chloride-N,N-dimethylacetamide system has been investigated by Pistoia and Scrosati (1) and by Fedorov et.al. (2).

The latter authors reported a solubility at 295-298 K of 0.25 mol dm⁻³. However, this datum is rejected since no purification of the solvent nor control of the temperature was attempted in the investigation of Fedorov et.al. (2).

The solubility determined by Pistoia and Scrosati (1) at 298 K (41.3 g/kg of solvent) appears to be more accurate since these authors have taken precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

Nevertheless, because of the lack of reliable data from other sources, which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value of the solubility of calcium chloride in N,N-dimethylacetamide at 298 K.

Tentative value of solubility of CaCl, at 298 K

41.3 g/kg of solvent

References

- 1. Pistoia, G.; Scrosati, B. Ric. Sci., 1967, 37, 1173-1177.
- Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I.
 Z. Prikl. Khim., 1971, 44(10), 2361-2362.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Calcium chloride; CaCl ₂ ; 10043-52-4	Pistoia, G.; Scrosati, B.
(2) N,N-dimethylacetamide; C ₄ H ₉ NO;	<i>Ric. Sci.</i> , <u>1967</u> , <i>37</i> , 1173–1177.
127-19-5	
VARIABLES:	
	PREPARED BY:
One temperature: 25 [°] C	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of calcium chloride at 25°C	
4.13 g/100	g of solvent
	5
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS:	
The method and the procedure for obtaining the saturated solution were described in	Calcium chloride, reagent grade commercial product, was dried under vacuum.
the course of the compilation of the lithium	
chloride-N,N-dimethylacetamide system,	fractional distillation under reduced
originally measured by Pistoia and Scrosati, to which we refer the reader for details.	pressure (2 mmHg).
The amount of dissolved CaCl ₂ in the	
saturated solution was determined by Fajans	
titration, using dichlorofluorescein as	
indicator.	
	ESTIMATED ERROR:
	Solubility ± 0.4%
	-
	Temperature $\stackrel{+}{=}$ 0.05 [°] C (authors)
	REFERENCES :

COMPONENTS: (1) Calcium bromide; CaBr₂; 7789-41-5

(2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5 EVALUATOR: Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

CRITICAL EVALUATION:

The calcium bromide-N,N-dimethylacetamide system has been investigated by Pistoia and Scrosati (1) and by Fedorov et.al. (2).

The latter authors have reported a solubility at 295-298 K of 0.32 mol dm⁻³. However, this datum is rejected since no purification of the solvent nor control of the temperature was attempted in the investigation of Fedorov et.al. (2).

The solubility determined by Pistoia and Scrosati (1) at 298 K, (99.5 g/kg of solvent) appears to be more accurate since these authors have used precautions to prepare pure solute and solvent, to operate in dry conditions and to control the temperature.

Nevertheless, because of the lack of reliable data from other sources, which may serve as quantities of comparison and evaluation, the result of Pistoia and Scrosati (1) may only be reported as a tentative value of the solubility of calcium bromide in N,N-dimethylacetamide at 298 K.

Tentative value of solubility of CaBr, at 298 K

99.5 g/kg of solvent.

References

- 1. Pistoia, G; Scrosati, B. Ric. Sci., 1967, 37, 1173-1177.
- Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I.
 Prikl. Khim., <u>1971</u>, 44(10), 2361-2362.

COMPONENTS :	ORIGINAL MEASUREMENTS:
	Pistoia, G.; Scrosati, B.
2	<i>Ric. Sci.</i> , <u>1967</u> , 37, 1173-1177.
(2) N,N-dimethylacetamide; C ₄ H ₉ NO;	
127-19-5	
VARIABLES:	PREPARED BY:
One temperature: 25°C	Bruno Scrosati
EXPERIMENTAL VALUES:	
Solubility of calcium bromide at 25 ⁰ C	
9.95 g/100 g	g of solvent.
	· · · · · · · · · · · · · · · · · · ·
	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The method and the procedure for obtaining the saturated solution were described in	Calcium bromide, reagent grade commercial product, was dried under vacuum.
the course of the compilation of the lithium	N.N-dimethylacetamide was purified by
chloride-N,N-dimethylacetamide system, originally measured by Pistoia and Scrosati,	fractional distillation under reduced
to which we refer the reader for details.	pressure (2 mmHg).
The amount of dissolved CaBr, in the	
saturated solution was determined by Fajans	
titration, using tetrabromofluorescein as indicator.	
	ESTIMATED ERROR:
	Solubility ± 0.4%
	Temperature $\stackrel{+}{=} 0.05^{\circ}$ C (authors)
	remperature 0.05 0 (authors)
	REFERENCES:

;

N, N-Dimethylacetamide	
COMPONENTS: (1) Calcium iodide; CaI ₂ ; 10102-68-8 (2) N,N-dimethylacetamide; C ₄ H ₉ NO; 127-19-5	EVALUATOR: Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.
CRITICAL EVALUATION: Very few solubility data of halides in aceta particular the system calcium iodide-N,N-dir Fedorov et.al. (1), who reported a value of	nethylacetamide has been examined only by
In other system, where more reliable data for of Fedorov et.al. are rejected since no pure temperature was attempted by these authors is	
· · ·	where the data of Fedorov et.al. (1) are the have been compiled and reported as very appro
Approximate value of solubility of Cal ₂ at 2 26 g dm ⁻³ of	298 K Saturated solution
Since the density of the saturated solution this datum into molal units.	is not known it is not possible to convert
References 1. Fedorov, A.A.; Sokolov, L.B.; Savinov, Z. Prikl. Khim., 1971, 44(10), 2361-236	

34Z /// Di	mourflasotarmas
COMPONENTS: (1) Calcium iodide; CaI ₂ ; 10102-68-8 (2) N,N-dimethylacetamide; C ₄ H ₉ NO; 127-19-5	ORIGINAL MEASUREMENTS: Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M. Shilionskaya, M.A.; Filatova, N.I. Z. Prikl. Khim., <u>1971</u> , 44(10), 2361-2362
VARIABLES: One temperature: 22-25 ⁰ C	PREPARED BY: Bruno Scrosati
Solubility of calcium iodide at 22-25 ⁰ C 0.09 mc	ol dm ⁻³

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: The saturated solution was prepared at 'ambient' temperature (22-25°C) adding excess of solute to freshly distilled solvent. The solution was kept in a desiccator for three days. The amount of dissolved salt in the solution was then evaluated by titrating the anion concentration with the mercurimetric method.	SOURCE AND PURITY OF MATERIALS: Calcium iodide was dried under vacuum. No details on the purification of N,N-dimethyl- acetamide are reported.
	ESTIMATED ERROR: Solubility [±] 10% compiler. REFERENCES:

COMPONENTS :	EVALUATOR:
(1) Strontium chloride; SrCl ₂ ; 10476-85-4	Bruno Scrosati,
 (1) Strontium chloride; SrCl₂; 10476-85-4 (2) N,N-dimethylacetamide; C₄H₉NO; 127-19-5 	University of Rome, Italy.
127-19-5	May 1979

Very few solubility data of halides in acetamides are available in the literature. In particular the strontium chloride-N,N-dimethylacetamide system has been examined only by Fedorov et.al. (1), who reported a value of 0.17 mol dm⁻³ at 295-298 K.

In other systems where more reliable data from other sources are available, the results of Fedorov et.al. (1) are rejected since no purification of the solvent nor control of the temperature was attempted by these authors in their investigation.

However, in the cases like the present one, where only the data of Fedorov et.al. (1) are available, the results of these authors have been compiled and reported as very approximate values of solubility.

Approximate value of solubility of SrCl, at 298 K

 27 g dm^{-3} of saturated solution

Since the density of the saturated solution is not known it is not possible to convert this datum into molal units.

References

 Fedorov, A.A.; Sokolov, L.B.; Savinvov, V.M.; Shilionskaya, M.A.; Filatova, N.I. Z. Prikl. Khim., <u>1971</u>, 44(10), 2361-2362.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Strontium chloride; SrC1 ₂ ;	Fedorov, A.A.; Sokolov, L.B.;
10476-85-4	Savinov, V.M.; Shilionskaya, M.A.;
(2) N,N-dimethylacetamide; C ₄ H ₉ NO;	Filatova, N.I.
127-19-5	Z. Prikl. Khim., 1971, 44(10), 2361-2362
VARIABLES:	PREPARED BY:
One temperature: 22-25 [°] C	Bruno Scrosati
EXPERIMENTAL VALUES: Solubility of strontium chloride at 22-25 [°] C. 0.17 mol	dm ⁻³

AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: The saturated solution was prepared at 'ambient' temperature (22:25°C) adding excess of solute to freshly distilled solvent The solution was kept in a desiccator for three days. The amount of dissolved salt in the solution was then evaluated by titrating the chloride ion with standard mercuric solution.	SOURCE AND PURITY OF MATERIALS: Strontium chloride was dried under vacuum. No details on the purification of N,N-dime- thylacetamide are reported.
	ESTIMATED ERROR: Solubility ⁺ 10% (compiler)
	REFERENCES :

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SYSTEM INDEX

Underlined page numbers refer to the evaluation text and those not underlined to the compiled tables for that system. The compounds are listed in the order as used in the Chemical Abstracts indexes, for example, N-methylformamide is listed as formamide,N-methyl- and sodium thiocyanate as thiocyanic acid, sodium salt.

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Acetamide,N-methyl + ammonium bromide + ammonium chloride + ammonium iodide + barium bromide + barium chloride + calcium bromide + calcium chloride		285,	290, 286, 292, <u>307</u> , <u>305</u> , <u>298</u> , <u>296</u> ,	287 293 308 306 299 297
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Acetamide,N,N-dimethyl + calcium bromide + calcium chloride + calcium iodide + lithium bromide + lithium bromide + lithium chloride + magnesium chloride + magnesium iodide + potassium bromide + potassium chloride + sodium bromide + sodium bromide + sodium chloride + sodium chloride + strontium chloride + thiocyanic acid,potassium salt	<u>309</u> ,	<u>310</u> ,	$\frac{339}{337}, \frac{341}{313}, \frac{313}{313}, \frac{339}{313}, 3$	338 342 314 312 316 334 326 330 320 318 322 344
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Ammonium chloride + acetamide,N-methyl- + formamide + formamide,N-methyl- + formamide,N,N-dimethyl-	<u>284</u> ,	$\frac{61}{130}$,	286, 62, 131 - 223 -	63 133
Ammonium iodide + acetamide,N-methyl- + formamide + formamide,N-methyl- + formamide,N,N-dimethyl-		<u>67</u> ,	$\frac{292}{68},$ $\frac{138}{229},$	293 69 139 230
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Caesium iodide + formamide + formamide,N,N-dimethyl-	$\frac{58}{220}, 59 - 60$ $\frac{220}{221}$
Calcium bromide + acetamide,N-methyl- + acetamide,N,N-dimethyl- + formamide + formamide,N-methyl- + formamide,N,N-dimethyl-	$\begin{array}{r} 298, 299\\ \underline{339}, 340\\ \overline{78}, 79\\ \underline{146}, 147\\ \underline{243}, 244 \end{array}$
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+ caesium chloride + caesium iodide + calcium bromide + calcium chloride + cyanic acid, potassium salt + lithium bromide + lithium chloride	$\begin{array}{cccccccccccccccccccccccccccccccccccc$
+ magnesium chloride + potassium bromide + potassium chloride + potassium iodide	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$

r	
<pre>Formamide + rubidium bromide + sodium bromide + sodium fluoride + sodium bromide + strontium bromide + thiocyanic acid, potassium salt + thiocyanic acid, sodium chloride + ammonium chloride + angonsium chloride + calcium chloride + lithium chloride + potassium chloride + potassium chloride + potassium chloride + potassium chloride + potassium chloride + sodium chloride + sodium chloride + sodium chloride + sodium chloride + sodium chloride + thiocyanic acid, sodium salt + caesium chloride + caesium chloride + caesium chloride + caesium chloride + caesium chloride + calcium bromide + calcium chloride + calcium chloride + potassium salt + hydrocyanic acid, sodium salt + hydrocyanic acid, sodium salt + hydrocyanic acid, sodium salt + potassium iodide + potassium iodide + potassium iodide + sodium chloride + sodium chloride</pre>	$\begin{array}{c} \underline{14}, \ \underline{15}, \ \overline{16} - 20\\ \underline{9}, \ 10 - 13\\ 7, \ 8\\ \underline{21}, \ 2\overline{2} - 25\\ \underline{82}, \ 83\\ \underline{80}, \ \overline{81} - 83\\ \underline{70}, \ 711\\ \underline{48}, \ 49 - 51\\ \underline{26}, \ 27\\ \underline{134}, \ 135 - 137\\ \underline{130}, \ 131 - 133\\ \underline{138}, \ 139\\ \underline{146}, \ 147\\ \underline{144}, \ 145\\ \underline{93}, \ 94, \ 95\\ \underline{88}, \ 89 - 92\\ \underline{142}, \ 143\\ \underline{118}, \ 119 - 122\\ \underline{113}, \ 114 - 117\\ \underline{123}, \ 124 - 127\\ \underline{101}, \ 102 - 105\\ \underline{96}, \ 97 - 100\\ \underline{106}, \ 107 - 110\\ \underline{140}, \ 141\\ \underline{128}, \ 129\\ \underline{111}, \ 112\\ \underline{226}, \ 227, \ 228\\ \underline{222}, \ 223 - 225\\ \underline{229}, \ 230\\ \underline{231}, \ 232 - 234\\ \underline{216}, \ 217 - 219\\ \underline{212}, \ 213 - 215\\ \underline{220}, \ 221\\ \underline{243}, \ 244\\ \underline{240}, \ 241, \ 242\\ \underline{176} - 177, \ 178\\ \underline{204}, \ 205\\ \underline{206}, \ 207\\ \underline{179}, \ 180\\ \underline{155}, \ 152 - 155\\ \underline{148}, \ 149, \ 150\\ \underline{235}, \ 236, \ 237\\ \underline{238}, \ 239\\ \underline{194}, \ 197\\ \underline{186}, \ 167, \ 193\\ \underline{198}, \ 199 - 203\\ \underline{168}, \ 169, \ 170\\ \underline{162}, \ 163 - 167\\ \underline{159}, \ 160, \ 161\\ \underline{171}, \ 172 - 175\\ \underline{208}, \ 209 - 211\\ \underline{181}, \ 182 - 185\\ \end{array}$
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м			
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P			
Potassium bromide + acetamide + acetamide,N-methyl- + acetamide,N,N-dimethyl- + formamide	<u>274</u> ,	35,	$\begin{array}{r} \underline{249}, \ 250\\ \overline{276}, \ 277\\ \underline{327}, \ 328\\ \overline{36} - \ 38 \end{array}$
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Potassium chloride + acetamide + acetamide,N-methyl- + formamide + formamide,N-methyl- + formamide,N,N-dimethyl-	<u>28</u> ,	29,	247, 248 272, 273 30 - 34 114 - 117 187 - 193
Potassium cyanate, see hydrocyanic acid,potassium salt cyanide, see cyanic acid,potassium salt			
Potassium iodide + acetamide + acetamide,N-methyl- + acetamide,N,N-dimethyl- + formamide + formamide,N-methyl-	$\frac{251}{278},$ $\frac{39}{39},$	<u>279</u> , 40,	253, 254280, 281329, 330 $41 - 45$
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