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SOLUBILITY DATA SERIES

Volume 11

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

SOLUBILITY DATA SERIES

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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 is commissioned to recognized authorities, involving a process of careful selection in the best tradition of IUPAC. This selection in turn is the key to the quality of the output. These top experts are expected to view their specific topics dispassionately, paying equal attention to their own contributions and to those of their peers. They digest literature data into a coherent story by weeding out what is wrong from what is believed to be right. To fulfill this task, the evaluator must cover *all* relevant open literature. No reference is excluded by design and every effort is made to detect every bit of relevant primary source. Poor 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;

(ii) a set of recommended numerical data. Whenever possible, the set of recommended data includes weighted average and standard deviations, and a set of smoothing equations derived from the experimental data endorsed by the evaluator;

(iii) a graphical plot of recommended data.

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

The typical data sheet carries the following information:

(i) components - definition of the system - their names, formulas and Chemical Abstracts registry numbers;

(ii) reference to the primary source where the numerical information is reported. In cases when the primary source is a less common periodical or a report document, published though of limited availability, abstract references are also given;

(iii) experimental variables;

(iv) identification of the compiler;

(v) experimental values as they appear in the primary source. Whenever available, the data may be given both in tabular and graphical form. If auxiliary information is 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 responsive 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 project. It should help to dispel confusion in the minds of many authors of what represents a permanently useful bit of information of an archival value, and what does not.

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

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.

(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. Scrosati
C. 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:

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

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

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

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

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

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

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

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

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

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

where M_A is the molar mass of the solvent.

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

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

The terms molarity and molar are not used.

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

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

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

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

Thermodynamics of Solubility

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

Activity Coefficients (1)

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

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

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

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

(b) *Solutions.*

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

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

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

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

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

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

or

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

or

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

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

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

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

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

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

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

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

(ii) *Solvent, A:*

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

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

where μ_A^* is the chemical potential of the pure solvent.

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

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

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:

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

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

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

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

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

where (7)

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

it is found that

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

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

where

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

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

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

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

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

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

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

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

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

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

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

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

where 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

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

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

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

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

and (27) becomes

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

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

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

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

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

$$\ln\left\{\frac{x_B^{\nu}(1-x_B)^n}{1+(\nu-1)x_B{}^{n+\nu}}\right\} = \ln\left\{\frac{n^n}{(n+\nu)^{n+\nu}}\right\} - \left\{\frac{\Delta H^{\infty}_{AB}(T^*)-T^*\Delta C_p{}^*}{R}\right\}\left(\frac{1}{T} - \frac{1}{T^*}\right) + \frac{\Delta C_p^*}{R}\ln(T/T^*) \tag{34}$$

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

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

(b) Solubility as a function of composition.

At constant temperature and pressure, the chemical potential of a saturating solid phase is constant:

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

$$= (n\mu^*_A + \nu_+\mu_+{}^{\infty}+\nu_-\mu_-{}^{\infty}) + nRT \ln f_Ax_A$$

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

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

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

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

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

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

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

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

The Solid Phase

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

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

COMPILATIONS AND EVALUATIONS

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

Guide to the Compilations

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

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

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

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

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

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

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

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

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

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

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

References. See the above description for Original Measurements.

Guide to the Evaluations

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

Components. See the description for the Compilations.

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

Critical Evaluation

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

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

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

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

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

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

References

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2. McGlashan, M.L. *Physicochemical Quantities and Units*. 2nd ed. Royal Institute of Chemistry. London. 1971.
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R. Cohen-Adad, Villeurbanne, France
J.W. Lorimer, London, Canada
M. Salomon, Fair Haven, New Jersey, U.S.A.

August, 1979

COMPONENTS:	EVALUATOR:
(1) Lithium chloride; LiCl; [7447-41-8] (2) Formamide; CH_3NO; [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 LiCl 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. In neither investigation was the recommended method of drying the LiCl (by passing dry HCl) 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:

308 K

211 ± 11 g/kg of solvent

318 K

185 ± 10 g/kg of solvent.

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

COMPONENTS:

(1) Lithium chloride; LiCl; [7447-41-8]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	25.1
35	21.1
45	18.5

AUXILIARY INFORMATION

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 made in triplicate.

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 5×10^{-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

COMPONENTS:

(1) Lithium chloride; LiCl; [7447-41-8]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of lithium chloride in formamide at 25 °C

28.2 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 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:

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 x 10^{-4} S m^{-1}.

ESTIMATED ERROR:

Solubility	± 1%
Temperature	± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:	EVALUATOR:
(1) Lithium bromide; LiBr; [7550-35-8] (2) Formamide; CH_3NO; [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 2×10^{-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.

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.

Tentative values

298 K

600 ± 150 g/kg of solvent

308 K

583 ± 150 g/kg of solvent

318 K

759 ± 150 g/kg of solvent

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

COMPONENTS:

(1) Lithium bromide; LiBr; [7550-35-8]

(2) Formamide, CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Paul, R.C.; Singla, J.P.; Lambda, M.S.; Gill, D.S.; Narula, S.P.

Indian J. Chem., 1973, *11*, 1024-1026

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

$t/^{\circ}C$	g/100 g solvent
25	45.5
35	58.3
45	75.9

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of lithium 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 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade lithium 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 ± 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.

COMPONENTS:

(1) Lithium bromide; LiBr; [7550-35-8]
(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of lithium bromide in formamide at 25 °C

73.8 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 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 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 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}.

ESTIMATED ERROR:

Solubility	± 1%
Temperature	± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:	EVALUATOR:
(1) Sodium fluoride; NaF; [7681-49-4] (2) Formamide; CH_3NO; [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 ± 0.1 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:

(1) Sodium fluoride; NaF; [7681-49-4]
(2) Formamide, CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Colton, E.; Brooker, R.E.
J. Phys. Chem., 1958, *62*, 1595-1596

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium fluoride in formamide at 25 °C

1.1 g/1000 cm^3 of saturated solution.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade sodium fluoride.
Commercial formamide (Fisher 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:

COMPONENTS:	EVALUATOR:
(1) Sodium chloride; NaCl; [7647-14-5] (2) Formamide; CH_3NO; [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 2×10^{-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 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.
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. 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.

COMPONENTS:

(1) Sodium chloride; NaCl; [7647-14-5]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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/ °C	g/100 g solvent
25	9.44
35	8.85
45	8.32

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of sodium 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.

SOURCE AND PURITY OF MATERIALS:

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 5×10^{-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.

COMPONENTS:

(1) Sodium chloride; NaCl; [7647-14-5]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One Temperature: 25 °C

PREPARED BY:

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:

Considerable effort was made to obtain water free solutions and all the solutions were prepared in a dry box. Saturated sodium 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:

Sodium 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. *Ric. Sci.*, 1967, *37*, 1167-1172

COMPONENTS:

(1) Sodium chloride; NaCl; [7647-14-5]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
25	9.32
30	9.05
35	8.91
40	8.71
45	8.55
50	8.42

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of NaCl 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_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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade sodium 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:	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; [7647-14-5] (2) Formamide; CH_3NO; [75-12-7]	Colton, E.; Brooker, R.E. *J. Phys. Chem.*, 1958, *62*, 1595-1596
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium chloride in formamide at 25 °C

99 g/1000 cm^3 of saturated solution.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of sodium 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.

The sodium was determined photometrically after decomposing the formamide. A Beckman DU spectrophotometric was used.

SOURCE AND PURITY OF MATERIALS:

Reagent grade sodium 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 ± 1 °C (author)

REFERENCES:

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) Formamide; CH_3NO; [75-12-7]

EVALUATOR:

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 x 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 x 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 ± 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.

308 K

387 ± 5 g/kg of solvent

318 K

421 ± 6 g/kg of solvent.

(continued)

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) Formamide; CH_3NO; [75-12-7]

EVALUATOR:

C.A. Vincent,
Department of Chemistry,
University of St.Andrews,
St.Andrews, Fife,
Scotland.
December 1978

CRITICAL EVALUATION: (continued)

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. Colton, E.; Brooker, R.E. *J. Phys. Chem.*, 1958, *62*, 1595-1596.

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.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium bromide; NaBr; [7647-15-6] (2) Formamide; CH_3NO; [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
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:

Saturated solutions of sodium 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.

SOURCE AND PURITY OF MATERIALS:

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^{-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 .

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]
(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium bromide in formamide at 25 °C.

35.3 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 sodium 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 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 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}.

ESTIMATED ERROR:

Solubility	±	1%
Temperature	±	0.05 °C (author)

REFERENCES:

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

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

$t/^\circ C$	g/100 g solvent
25	35.8
30	38.0
35	39.1
40	40.1
45	42.4
50	44.1

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of NaBr 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_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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade sodium bromide.
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: (1) Sodium bromide; NaBr; [7647-15-6] (2) Formamide; CH_3NO; [75-12-7]	ORIGINAL MEASUREMENTS: Colton, E.; Brooker, R.E. *J. Phys. Chem.*, 1958, *62*, 1595-1596
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium bromide in formamide at 25 °C
439 g/1000 cm^3 of saturated solution

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of sodium bromide 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 DU spectrophotometer was used.

SOURCE AND PURITY OF MATERIALS:

Reagent grade sodium bromide.
Commercial formamide (Fisher 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:

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Pavlopolous, T.; Strehlow, H.

Z. Phys. Chem. (Wiesbaden), 1954 , *2,* 89-103

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium bromide in formamide at 25 °C

340 g/1000 cm^3 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 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 sodium bromide 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 ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Pavlopoulos, T.; Strehlow, H.
   Z. Phys. Chem. (Leipzig), 1953 , *202,* 474-479.

COMPONENTS:	EVALUATOR:
(1) Sodium iodide; NaI; [7681-82-5] (2) Formamide; CH_3NO; [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 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

Tentative value at 308 K

788 ± 10 g/kg of solvent

Tentative value at 318 K

1038 ± 15 g/kg of solvent.

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. 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.

COMPONENTS:

(1) Sodium iodide; NaI; [7681-82-5]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	65.6
35	80.0
45	102

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 5×10^{-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

COMPONENTS:

(1) Sodium iodide; NaI; [7681-82-5]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Berardelli, M.L.; Pistoia, G.;
Polcaro, A.M.

Ric. Sci., 1968 *38*, 814-819

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium iodide in formamide at 25 °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 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 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 x 10^{-4} S m^{-1}.

ESTIMATED ERROR:

Solubility	± 1%
Temperature	± 0.05 °C (author)

REFERENCES:

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

COMPONENTS: (1) Sodium iodide; NaI; [7681-82-5] (2) Formamide; CH_3NO; [75-12-7]	ORIGINAL MEASUREMENTS: Gopal, R.; Husain, M.M. *J. Ind. Chem. Soc.*, 1963, *40*, 272-274
VARIABLES: Temperature	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
25	56.6
30	67.7
35	78.0
40	89.7
45	103
50	119

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of NaI 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_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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade sodium iodide.

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: (1) Sodium iodide; NaI; [7681-82-5] (2) Formamide; CH_3NO; [75-12-7]	ORIGINAL MEASUREMENTS: Colton, E.; Brooker, R.E. *J. Phys. Chem.*, 1958, *62*, 1595-1596
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium iodide in formamide at 25 °C

752 g/1000 cm^3 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 DU spectrophotometer was used.

SOURCE AND PURITY OF MATERIALS:

Reagent grade sodium iodide.

Commercial formamide (Fisher 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:

COMPONENTS:	EVALUATOR:
(1) Sodium thiocyanate; NaCNS; [540-72-7] (2) Formamide; CH_3NO; [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 NaCNS 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

1435.0 ± 10.0 g/kg of solvent

308 K

891.0 ± 7.0 g/kg of solvent

318 K

575.1 ± 4.0 g/kg of solvent

References

1. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. *Indian J. Chem.*, 1973, *11*, 1024-1026.
2. Noltley, J.M.; Spiro, M. *J. Chem. Soc.(B)*, 1966, 362-366.

COMPONENTS:

(1) Sodium thiocyanate; NaCNS; [540-72-7]
(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
25	144
35	89.1
45	57.5

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of sodium thioazamate 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° 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.

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. *J. Chem. Soc., Dalton Trans.*, 1972, 522-524.

COMPONENTS:	EVALUATOR:
(1) Potassium chloride; KCl; [7447-40-7] (2) Formamide; CH_3NO; [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 purffication 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)

COMPONENTS:	EVALUATOR:
(1) Potassium chloride; KCl; [7447-40-7] (2) Formamide; CH_3NO; [75-12.7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION: (continued)

291 K

61.2 ± 1.0 g/kg of solvent

308 K

64.5 ± 1.0 g/kg of solvent

318 K

66.7 ± 1.0 g/kg of solvent

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. Colton, E.; Brooker, R.E. *J. Phys. Chem.,* 1958, *62,* 1595-1596.
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

COMPONENTS:

(1) Potassium chloride; KCl; [7447-40-7]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	6.29
35	6.48
45	6.78

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of potassium 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade potassium 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., Narula, S.P. *J. Chem. Soc., Dalton Trans.* 1972, 522-524

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KCl; [7447-40-7] (2) Formamide; CH_3NO;[75-12-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. *Ric. Sci.*, 1968, *38*, 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 1.40×10^{-4} S m^{-1}.

ESTIMATED ERROR:

Solubility	±	1%
Temperature	±	0.05 °C (author)

REFERENCES:

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

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KCl; [7447-40-7] (2) Formamide; CH_3NO; [75-12-7]	Gopal, R.; Husain, M.M. *J. Indian Chem. Soc.*, 1963, *40*, 272-274
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
25	6.30
30	6.34
35	6.41
40	6.49
45	6.56
50	6.69

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of KCl 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 withdrawm 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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade potassium 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:

(1) Potassium chloride; KCl; [7447-40-7]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Colton, E.; Brooker, R.E.

J. Phys. Chem., 1958, *62*, 1595-1596

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium chloride in formamide at 25 °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 photometrically 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:

COMPONENTS:

(1) Potassium chloride; KCl; [7447-40-7]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Pavlopoulos, T.; Strehlow, H.

Z. Phys. Chem. (Weisbaden), 1954, *2*, 89-103

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium chloride in formamide at two different temperatures.

t/°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 checked by density measurements.

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_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	± 1%
Temperature	± 0.1 °C (author)

REFERENCES:

1. Pavlopoulos, T.; Strehlow, H. *Z. Phys. Chem. (Leipzig)*, 1953, *202*, 474-479

COMPONENTS:

(1) Potassium bromide; KBr; [7758-02-3]

(2) Formamide; [75-12-7]

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

COMPONENTS:

(1) Potassium bromide; KBr; [7758-02-3]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	21.6
35	21.8
45	22.2

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satured solutions of potassium bromide were prepared by adding excess of the powdered salt to formamide (15-20 g) in Pyrex tubes (15×2.5 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.

SOURCE AND PURITY OF MATERIALS:

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 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.
J. Chem. Soc., Dalton Trans., 1972, 522-524

COMPONENTS:

(1) Potassium bromide; KBr; [7758-02-3]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One Temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium bromide in formamide at 25 °C.

21.6 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 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 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 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}.

ESTIMATED ERROR:

Solubility	± 1%
Temperature	± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:

(1) Potassium bromide; KBr; [7758-02-3]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°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:

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_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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade potassium bromide. 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) Potassium iodide; KI; [7681-11-0] (2) Formamide; CH_3NO; [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 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.

308 K

691 ± g/kg of solvent

318 K

698 ± 10 g/kg of solvent.

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. Colton, E.; Brooker, R.E. *J. Phys. Chem.*, 1958, *62*, 1595-1596.

(continued)

COMPONENTS:	EVALUATOR:
(1) Potassium iodide; KI; [7681-11-0] (2) Formamide; CH_3NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St.Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION: (continued)

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.

COMPONENTS:

(1) Potassium iodide; KI; [7681-11-0]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g Solvent
25	68.5
35	69.2
45	69.8

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of potassium iodide 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 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 potassium 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 5×10^{-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

COMPONENTS:

(1) Potassium iodide; KI; [7681-11-0]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °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 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 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	± 1%
Temperature	± 0.05 °C (author)

REFERENCES:

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

COMPONENTS: (1) Potassium iodide; KI; [7681-11-0] (2) Formamide; CH_3NO; [75-12-7]	ORIGINAL MEASUREMENTS: Gopal, R.; Husain, M.M. *J. Ind. Chem. Soc.*, 1963, *40*, 272-274
VARIABLES: Temperature	PREPARED BY: 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

AUXILIARY INFORMATION

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_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. 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 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 unknown solubility.

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.

ESTIMATED ERROR:

Solubility ± 1%
Temperature ± 0.1 °C (compiler)

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0] (2) Formamide; CH_3NO; [75-12-7]	Colton, E.; Brooker, R.E. *J. Phys. Chem.*, 1958. *62*, 1595-1596
VARIABLES:	**PREPARED BY:**
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium iodide in formamide at 25 °C

628 g/1000 cm^3 of saturated solution.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of potassium 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 potassium was determined photometrically 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 iodide. Commercial formamide (Fisher 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:

COMPONENTS: (1) Potassium iodide; KI; [7681-11-0] (2) Formamide; CH_3NO; [75-12-7]	ORIGINAL MEASUREMENTS: Pavlopoulos, T.; Strehlow, H. *Z. Phys. Chem. (Wiesbaden)*,1954, *2*, 89-103
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium iodide in formamide at 25 °C

616 g/1000 cm^3 of saturated solution.

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 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_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 $\pm$ 1%

Temperature $\pm$ 0.1 °C (author)

REFERENCES:

1. Pavlopoulos, T.; Strehlow, H.; *Z. Phys. Chem. (Leipzig)*, 1953, *202*, 474-479.

COMPONENTS:

(1) Potassium cyanide; KCN; [151-50-8]

(2) Formamide; CH_3NO; [75-12-7]

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 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 2×10^{-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 ± 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:
(1) Potassium cyanide; KCN; [151-50-8] (2) Formamide; CH_3NO; [75-12-7]	Colton, E.; Brooker, R.E. *J. Phys. Chem.*, 1958, *62*, 1595-1596
VARIABLES: One temperature: 25 °C	**PREPARED BY:** J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium cyanide in formamide at 25 °C.

146 g/1000 cm^3 of saturated solution.

AUXILIARY 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.

The potassium was determined photometrically 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 cyanide.
Commercial formamide (Fisher 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:

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

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. Colton, E.; Brooker, R.E. *J. Phys. Chem.*, 1958, *62*, 1595-1596.
4. Becker, B. *J. Chem. Eng. Data*, 1970, *15*, 31-32.

COMPONENTS:

(1) Potassium thiocyanate; KCNS; [333-20-0]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	143.5
35	141.9
45	141.0

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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.

SOURCE AND PURITY OF MATERIALS:

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 5×10^{-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.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium thiocyanate; KCNS;[333-20-0] (2) Formamide; CH_3NO;[75-12-7]	Becker, B. *J. Chem. Eng. Data,* 1970, *15(1),* 31-32
VARIABLES:	**PREPARED BY:**
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium thiocyanate in formamide at 25 °C.

89 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions were prepared by shaking excess potassium thiocyanate with the solvent for at least 48 hours in a thermostatted bath. The formamide was exposed to air only briefly during addition. The mixture was filtered rapidly in air at 25 °C and a weighed aliquot of the filtrate was taken 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 as K_2PtCl_6.

SOURCE AND PURITY OF MATERIALS:

Reagent grade potassium thiocyanate. Formamide (Matheson, Coleman and Bell,99%) was used without further purification. Its melting point of 2.5 °C ± 0.1 °C was in good agreement with the literature value of 2.55 °C.

ESTIMATED ERROR:

Solubility ± 5% (compiler)

Temperature ± 1 °C (author)

REFERENCES:

(1) Perrin, C.H. *Anal. Chem.,* 1949 , *21,* 984

COMPONENTS:

(1) Potassium thiocyanate; KCNS; [333-20-0]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Colton, E.; Brooker, R.E.;
J. Phys. Chem., 1958, *62*, 1595-1596

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J. S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium thiocyanate in formamide at 25 °C

659 g/1000 cm^3 of saturated solution.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of potassium thiocyanate 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 photometrically 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 thiocyanate. Commercial formamide (Fisher 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:

COMPONENTS:	EVALUATOR:
(1) Rubidium bromide; RbBr; [7789-39-1] (2) Formamide; CH_3NO; [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 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.*, 1967, *89*, 3703-3712.

COMPONENTS: (1) Rubidium bromide; RbBr; [7789-39-1] (2) Formamide; CH_3NO; [75-12-7]	ORIGINAL MEASUREMENTS: Pavlopoulos, T.; Strehlow, H. *Z. Phys. Chem. (Wiesbaden),* 1954, *2*, 89-103
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of rubidium bromide in formamide at 25 °C

275 g/1000 cm^3 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 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 rubidium bromide 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 ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Pavlopoulos, T.; Strehlow, H. *Z. Phys. Chem. (Leipzig),* 1953, *202*, 474-479.

COMPONENTS:

(1) Caesium chloride; CsCl; [7647-17-8]

(2) Formamide; CH_3NO; [75-12-7]

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 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 2×10^{-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 CsCl at 298 K is recommended as a tentative value only.

Tentative value at 298 K

91.5 ± 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:	ORIGINAL MEASUREMENTS:
(1) Caesium chloride; CsCl; [7647-17-8] (2) Formamide; CH_3NO; [75-12-7]	Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. *J. Am. Chem. Soc.*, 1967, *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:

$\log (K_s^o/\text{mol dm}^{-3}) = -0.53$

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 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 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^{-3} $AgNO_3$ solution into a half-cell containing the silver wire immersed in 20 cm^3 of 0.01 mol dm^{-3} caesium chloride in formamide. The reference half-cell was a silver wire in 0.01 mol dm^{-3} $AgNO_3$ 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 °C (compiler)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Caesium bromide; CsBr; [7787-69-1] (2) Formamide; CH_3NO; [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.

Tentative value at 298 K

152.4 ± 3.0 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 bromide; CsBr; [7787-69-1]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J.

J. Am. Chem. Soc., 1967, *89*, 3703-3712

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium bromide in formamide at 25 °C

152.4 g/1000 cm^3 of saturated solution.

Value calculated from solubility product data given as:

$\log (K_s o/\text{mol dm}^{-3}) = -0.29$

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 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^{-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^{-3} $AgNO_3$ solution into a half-cell containing the silver wire immersed in 20 cm^3 of 0.01 mol dm^{-3} caesium bromide in formamide. The reference half-cell was a silver wire in 0.01 mol dm^{-3} $AgNO_3$ in the same solvent.

SOURCE AND PURITY OF MATERIALS:

A.R.grade caesium bromide 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 the titration with potassium bromide, was as required by the stoichiometric amount of silver nitrate.

ESTIMATED ERROR:

Solubility ± 2% (compiler)

Temperature ± 0.1 °C (compiler)

REFERENCES:

COMPONENTS:

(1) Caesium iodide; CsI; [7789-17-5]

(2) Formamide; CH_3NO; [75-12-7]

EVALUATOR:

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 Pavlopoulos 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 x 10^{-5} S m^{-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.
2. Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. *J. Am. Chem. Soc.*, 1967, *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_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Alexander R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J.

J. Am. Chem. Soc., 1967, *89*, 3703-3712

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium iodide in formamide at 25 °C

199.4 g/1000 cm^3 of saturated solution.

Value calculated from solubility product data given as:

$$\log (K_s\mathrm{o}/\mathrm{mol\ dm^{-3}}) = -0.23$$

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 °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^{-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 $dm^{-3}AgNO_3$ solution into a half-cell containing the silver wire immersed in 20 cm^3 of 0.01 mol dm^{-3}caesium iodide in formamide. The reference half-cell was a silver wire in 0.01 mol dm^{-3} $AgNO_3$ 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 °C (compiler)

REFERENCES:

COMPONENTS:

(1) Caesium iodide, CsI; [7789-17-5]
(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Pavlopoulos, T.; Strehlow, H.
Z. Phys. Chem. (Wiesbaden), 1954, *2*, 89-103

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium iodide in formamide at 25 °C

431 g/1000 cm^3 of saturated solution.

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 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 ± 1%
Temperature ± 0.1 °C (author)

REFERENCES:

1. Pavlopoulos, T.; Strehlow, H. *Z. Phys. Chem. (Leipzig)*, 1953, *202*, 474-479.

COMPONENTS:	EVALUATOR:
(1) Ammonium chloride; NH_4Cl; [12125-02-9] (2) Formamide; CH_3NO; [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_4Cl 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_4Cl 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)*, 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.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ammonium chloride; NH_4Cl; [12125-02-9] (2) Formamide; CH_3NO; [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
VARIABLES:	PREPARED BY:
Temperature	J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
25	10.9
35	11.6
45	12.3

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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.

SOURCE AND PURITY OF MATERIALS:

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 5×10^{-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.

COMPONENTS:

(1) Ammonium chloride; NH_4Cl; [12125-02-9]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
25	11.0
30	11.4
35	11.7
40	12.1
45	12.4
50	12.8

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of NH_4Cl 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_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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade ammonium 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:

(1) Ammonium bromide; NH_4Br; [12124-97-9]

(2) Formamide; CH_3NO; [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 NH_4Br 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

308 K

384.0 ± 5.0 g/kg of solvent

318 K

394.5 ± 5.0 g/kg of solvent

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.

COMPONENTS:

(1) Ammonium bromide; NH_4Br; [12124-97-9]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	37.2
35	38.9
45	39.5

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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.

SOURCE AND PURITY OF MATERIALS:

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	± 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

COMPONENTS:

(1) Ammonium bromide; NH_4Br; [12124-97-9]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°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:

Saturated solutions of NH_4Br were prepared at 10-15° above the temperature at which the solubility was to be measured before thermostatting at the required temperature. Aliquots (3-4 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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade ammonium bromide

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:

(1) Ammonium iodide; NH_4I; [12027-06-4]

(2) Formamide; CH_3NO; [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 NH_4I 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_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

318 K

1060.5 ± 2.0 g/kg of solvent

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

COMPONENTS:

(1) Ammonium iodide; NH_4I; [12027-06-4]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	104
35	105
45	106

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of ammonium iodide 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 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 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 5×10^{-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

COMPONENTS:

(1) Ammonium iodide; NH_4I; [12027-06-4]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
25	104
30	105
35	105
40	105
45	106
50	106

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of NH_4I 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_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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade ammonium iodide

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:

(1) Ammonium thiocyanate; NH_4CNS; [1762-95-4]

(2) Formamide; CH_3NO; [75-12-7]

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 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

References

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:

(1) Ammonium thiocyanate; NH_4CNS; [1762-95-4]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narual, S.P.

Indian J. Chem., 1973, *11*, 1024-1026

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
25	102
35	110
45	116

AUXILIARY INFORMATION

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 in triplicate.

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 5×10^{-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

COMPONENTS:	EVALUATOR:
(1) Magnesium chloride; $MgCl_2$; [7786-30-3] (2) Formamide; CH_3NO; [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 $CaCl_2$ obtained by the above workers and those of Gopal and Husain (3). However the recommended method of preparing anhydrous $MgCl_2$ (by passing dry HCl over the hydrated double salt, $MgCl_2 . NH_4Cl$) 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 ± 4 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:

(1) Magnesium chloride; $MgCl_2$; [7786-30-3]
(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of magnesium chloride in formamide at 25 °C

8.40 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 magnesium 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 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 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:

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}.

ESTIMATED ERROR:

Solubility	± 1%
Temperature	± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:	EVALUATOR:
(1) Calcium chloride; $CaCl_2$; [10043-52-4] (2) Formamide; CH_3NO; [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 $CaCl_2$ 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}. 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

308 K

224 ± 3 g/kg of solvent

318 K

249 ± 3 g/kg of solvent

328 K

271 ± 3 g/kg of solvent.

References

1. Notley, J.M.; Spiro, M. *J. Chem. Soc.(B)*, 1966, 362-366
2. Colton, E.; Brooker, R.E. *J. Phys. Chem.* 1958, *62*, 1595-1596.
3. Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. *Ric. Sci.*, 1968, *38*, 814-819
4. Gopal, R.; Husain, M.M. *J. Indian Chem. Soc.*, 1963, *40*, 272-274.

COMPONENTS:

(1) Calcium chloride; $CaCl_2$; [10043-52-4]
(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of calcium chloride in formamide at 25 °C.

20.0 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 calcium 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:

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}.

ESTIMATED ERROR:

Solubility	± 1%
Temperature	± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:

(1) Calcium chloride; $CaCl_2$; [10043-52-4]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g
35	22.9
40	23.6
45	24.5
50	25.9
55	27.2

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of $CaCl_2$ 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_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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade calcium 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: (1) Calcium chloride; $CaCl_2$; [10043-52-4] (2) Formamide; CH_3NO; [75-12-7]	**ORIGINAL MEASUREMENTS:** Colton, E.; Brooker, R.E. *J. Phys. Chem.*, 1958, *62*, 1595-1596
VARIABLES: One temperature: 25 °C	**PREPARED BY:** J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of calcium chloride in formamide at 25 °C

222 g/1000 cm^3 of saturated solution

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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.

SOURCE AND PURITY OF MATERIALS:

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 ± 1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Calcium bromide; $CaBr_2$; [7789-41-5] (2) Formamide; CH_3NO; [75-12-7]	C.A. Vincent, Department of Chemistry, University of St.Andrews, St. Andrews, Fife, Scotland. December 1979

CRITICAL EVALUATION:

One investigation of the solubility of $CaBr_2$ 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 $CaCl_2$ obtained by the above workers and those of Gopal and Husain (3).

Recommended value at 298 K

434 ± 4 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:

(1) Calcium bromide; $CaBr_2$; [7789-41-5]
(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

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 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:

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 x 10^{-4} S m^{-1}.

ESTIMATED ERROR:

Solubility	± 1%
Temperature	± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:	EVALUATOR:
(1) Strontium chloride; $SrCl_2$; [10476-85-4] (2) Formamide; CH_3NO; [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 $SrCl_2$ 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 -0.994. On the basis of this analysis the following solubility values are recommended.

Recommended values

T/K	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

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.

COMPONENTS:

(1) Strontium chloride; $SrCl_2$; [10476-85-4]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
20	15.0
25	15.9
30	16.2
35	17.0
40	18.1
45	18.8
50	19.3

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of $SrCl_2$ 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_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. 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 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 unknown solubility.

SOURCE AND PURITY OF MATERIALS:

A.R. grade strontium 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:

(1) Strontium bromide; $SrBr_2$; [10476-81-0]

(2) Formamide; CH_3NO; [75-12-7]

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 $SrBr_2$ 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

T/K	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.

COMPONENTS:

(1) Strontium bromide; $SrBr_2$; [10476-81-0]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

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

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of $SrBr_2$ 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_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. 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) 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 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade strontium bromide.

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 chloride; $BaCl_2$; [10361-37-2] (2) Formamide; CH_3NO; [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

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.

COMPONENTS:

(1) Barium chloride; $BaCl_2$; [10361-37-2]

(2) Formamide; CH_3NO; [75-12-17]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

t/°C	g/100 g solvent
25	11.8
30	12.5
35	13.1
40	13.0
45	14.6
50	15.4

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of $BaCl_2$ 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_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. 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 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 unknown solubility.

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:

(1) Barium bromide; $BaBr_2$; [10553-31-8]

(2) Formamide; CH_3NO; [75-12-7]

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 $BaBr_2$ 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

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.

COMPONENTS:

(1) Barium bromide; $BaBr_2$; [10553-31-8]

(2) Formamide; CH_3NO; [75-12-7]

ORIGINAL MEASUREMENTS:

Gopal, R.; Husain, M.M.

J. Ind. Chem. Soc., 1963, *40*, 272-274

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

T/°C	g/100 g solvent
25	29.9
30	31.5
35	34.5
40	35.4
45	35.5
50	36.5

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of $BaBr_2$ 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_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. The samples were then cooled and weighed in a dry atmosphere. The process was repeated to obtain a constant weight. All the three samples taken were treated in a similar manner.
(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 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade barium bromide.

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:

(1) Lithium chloride; LiCl; [7447-41-8]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

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 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 LiCl (by passing dry HCl over the salt) so that the purity of the solute is unknown.

A mean value for the solubility of LiCl of 230 ± 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,* 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.; 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.

COMPONENTS:

(1) Lithium chloride; LiCl; [7447-41-8]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	20.6
35	15.1
45	12.9

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of lithium 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade lithium chloride (BDH) was crystallised and dried under vacuum at 110-125 °C.

N-methylformamide was purified by fractionation 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 $3\text{-}4 \times 10^{-5}$ 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) Lithium chloride; LiCl; [7447-41-8] (2) N-methylformamide; C_2H_5NO; [123-39-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. *Ric. Sci.*, 1968, *38*, 814-819
VARIABLES:	**PREPARED BY:**
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

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

23.9 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 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 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 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-methylformamide 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. *Ric. Sci.*, 1967, *37*, 1167-1172

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium chloride; LiCl; [7447-41-8] (2) N-methylformamide; C_2H_5NO; [123-39-7]	Strack, G.A.; Swanda, K.S.; Bahe, L.W. *J. Chem. Eng. Data*, 1964, *9*, 416-418
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

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

24.2 g/100 g solvent.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of lithium chloride and 10 cm^3 of N-methylformamide in a carefully stoppered test tube were placed in a constant temperature bath for 2-4 days. The test tubes were shaken several times each day. Saturation was assumed when samples taken at least one day apart gave identical concentrations. 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 100 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 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. As the densities were used to calculate

(continued)

SOURCE AND PURITY OF MATERIALS:

Anhydrous A.R. grade lithium 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 methylamine 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 10^{-1}

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Berger, C.; Dawson, L.R. *Anal. Chem.*, 1952, *24*, 994-996.

(continued)

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium chloride; LiCl; [7447-41-8] (2) N-methylformamide; C_2H_5NO; [123-39-7]	Strack, G.A.; Swanda, K.S.; Bahe, L.W. *J. Chem. Eng. Data,* 1964, *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 PURITY 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)]; ρ_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_2H_5NO; [123-39-7]	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 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.

References

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

(1) Lithium bromide; LiBr; [7550-35-8]

(2) N-methylformamide; C_2H_5NO;[123-39-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	35.2
35	22.0
45	11.7

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of lithium 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade lithium bromide (BDH) was recrystallised and dried under vacuum at 110-125 °C.

N-methylformamide was purified by fractionation 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:

(1) Lithium bromide; LiBr; [7550-35-8]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

Berardelli, M.L.; Pistoia, G.; Polcaro, A.M

Ric. Sci., 1968, *38*, 814-819

VARIABLES:

One temperature: 25 °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 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 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^{-4}S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:	EVALUATOR:
(1) Sodium chloride; NaCl; [7647-14-5] (2) N-methylformamide; C_2H_5NO; [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 x 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 ± 1.0 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.
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)*, 1966, 212-219.

COMPONENTS: (1) Sodium chloride; NaCl; [7647-14-5] (2) N-methylformamide; C_2H_5NO; [123-39-7]	ORIGINAL MEASUREMENTS: 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/°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 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 chloride (BDH) was recrystallised and dried under vacuum at 110-125 °C.

N-methylformamide was purified by fractionation 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) Sodium chloride; NaCl; [7647-14-5] (2) N-methylformamide; C_2H_5NO; [123-39-7]	Berardelli, M.L.; Pistoia, G.; Polcaro, A.M. *Ric. Sci.*, 1968, *38*, 814-819
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium chloride in N-methylformamide at 25 °C

3.29 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 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 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:

Sodium 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-methylformamide 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. *Ric. Sci.*, 1967, *37*, 1167-1172

COMPONENTS:

(1) Sodium chloride; NaCl; [7647-14-5]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium chloride in N-methylformamide at 25 °C

3.22 g/100 g solvent

Within the temperature rante 0-35 °C the solubilities in g/100 g of solvent, S, are given by:

$$S = -0.011\ T + 6.419$$

where T is the absolute temperature.

AUXILIARY INFORMATION

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 temperature bath for 2-4 days. The test tubes were shaken several times each day. Saturation was assumed when samples taken at least one day apart gave identical concentrations. 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 100 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 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)

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 methylamine 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)

REFERENCES:

1. Berger, C.; Dawson, L.R. *Anal. Chem.*, 1952, *24*, 994-996.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; [7647-14-5] (2) N-methylformamide; C_2H_5NO; [123-39-7]	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 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 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)]; ρ_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", 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:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

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 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 x 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 ± 10 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:

308 K

287 ± 10 g/kg of solvent

318 K

277 ± 10 g/kg of solvent.

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.
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),* 1966, 212-219.

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	29.8
35	28.0
45	27.7

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of sodium 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade sodium bromide (BDH) was recrystallised and dried under vacuum at 110-125 °C.

N-methylformamide was purified by fractionation 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:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

Berardelli, M.L.; Pistoia, G.; Polcaro, A.M.

Ric. Sci., 1968, *38*, 814-819

VARIABLES:

One temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

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

30.8 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 sodium 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 determined by a potentiometric method using a titrimeter (type Radiometer TTT1) and, presumably, standatd 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:

Sodium 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^{-4} S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

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

28.6 g/100 g solvent

Within the temperature range 0-35 °C the solubilities in g/100 g of solvent, S, are given by:

$$S = -0.124\ T + 66.59$$

Where T is the absolute temperature.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sodium bromide and 10 cm^3 of N-methylformamide in a carefully stoppered test tube were placed in a constant temperature bath for 2-4 days. The test tubes were shaken several times each day. Saturation was assumed when samples taken at least one day apart gave identical concentrations. 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 100 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 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. As the densities were used to calculate concentrations on the molar scale the

(continued)

SOURCE AND PURITY OF MATERIALS:

Anhydrous A.R. grade sodium bromide 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 methylamine 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 10^{-1} and 10^{-2} S m^{-1}. It was stored in tightly

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Berger, C.; Dawson, L.R. *Anal. Chem.,* 1952, *24,* 994-996.

(continued)

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) N-methylformamide; C_2H_5NO; [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)

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 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 are within 0.6 - 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.,* 1952, *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 iodide; NaI; [7681-82-5] (2) N-methylformamide; C_2H_5NO; [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 ± 30 g/kg of solvent.

318 K

946 ± 30 g/kg of solvent.

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.
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),* 1966, 212-219.

COMPONENTS:

(1) Sodium iodide; NaI; [7681-82-5]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	79.5
35	87.0
45	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 fractionation 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:

(1) Sodium iodide; NaI; [7681-82-5]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

Berardelli, M.L.; Pistoia, G.; Polcaro, A.M.

Ric. Sci., 1968, *38*, 814-819

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

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

57.5 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 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 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:

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-methylformamide 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. *Ric. Sci.*, 1967, *37*, 1167-1172

COMPONENTS:

(1) Sodium iodide; NaI; [7681-82-5]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

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

77.9 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sodium iodide and 10 cm^3 of N-methylformamide in a carefully stoppered test tube were placed in a constant temperature bath for 2-4 days. The test tubes were shaken several times each day. Saturation was assumed when samples taken at least one day apart gave identical concentrations. 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 100 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 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. As the densities were used to calculate

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SOURCE AND PURITY OF MATERIALS:

Anhydrous A.R. grade sodium iodide 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 methylamine 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 10^{-1}

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Berger, C.; Dawson, L.R. Anal. Chem., 1952, *24,* 994-996.

(continued)

COMPONENTS:

(1) Sodium iodide; NaI; [7681-82-5]

(2) N-methylformamide; C_2H_5NO; [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)

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} 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)]; ρ_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.,* 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) Sodium thiocyanate; NaCNS; [540-72-7] (2) N-methylformamide; C_2H_5NO; [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 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

298 K

503 ± 10 g/kg of solvent

308 K

356 ± 8 g/kg of solvent

318 K

288 ± 6 g/kg of solvent.

References

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.

COMPONENTS:

(1) Sodium thiocyanate; NaCNS; [540-72-7]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	50.3
35	35.6
45	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.
Measurements were made in triplicate.

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 fractionation 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:	EVALUATOR:
(1) Potassium chloride; KCl; [7447-40-7] (2) N-methylformamide; C_2H_5NO; [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 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 KCl.

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.

Recommended value at 298 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.

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.
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)*, 1966, 212-219.

COMPONENTS: (1) Potassium chloride; KCl; [7447-40-7] (2) N-methylformamide; C_2H_5NO; [123-39-7]	ORIGINAL MEASUREMENTS: 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/°C	g/100 g solvent
25	2.54
35	2.64
45	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.

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 fractionation 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:

(1) Potassium chloride; KCl; [7447-40-7]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

Berardelli, M.L.; Pistoia, G.; Polcaro, A.M

Ric. Sci., 1968, *38*, 814-819

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

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

2.14 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 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 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-methylformamide 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. *Ric. Sci.*, 1967, *37*, 1167-1172.

COMPONENTS:

(1) Potassium chloride; KCl; [7447-40-7]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

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

2.04 g/100 g solvent

Within the temperature range 0-35 °C the solubilities in g/100 g of solvent, S, are given by:

$$S = 0.000\,T + 2.036$$

where T is the absolute temperature

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of potassium chloride and 10 cm^3 of N-methylformamide in a carefully stoppered test tube were placed in a constant temperature bath for 2-4 days. The test tubes were shaken several times each day. Saturation was assumed when samples taken at least one day apart gave identical concentrations. 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 100 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 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. As the densities were used to calculate concentrations on the molar scale the results are estimated to be accurate to within 2% on

(continued)

SOURCE AND PURITY OF MATERIALS:

Anhydrous A.R. grade potassium 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 methylamine 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 10^{-1} and 10^{-2} S m^{-1}. It was stored in tightly-

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Berger, C.; Dawson, L.R. *Anal. Chem.*, 1952, *24*, 994-996.

(continued)

COMPONENTS:

(1) Potassium chloride; KCl; [7447-40-7]

(2) N-methylformamide; C_2H_5NO; [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)

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 are within 0.6 to 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)]; ρ_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.,* 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) Potassium bromide; KBr; [7758-02-3] (2) N-methylformamide; C_2H_5NO; [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 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.

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.
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:

(1) Potassium bromide; KBr; [7758-02-3]

(2) N-methylformamide; C_2H_5NO;[123-39-7]

ORIGINAL MEASUREMENTS:

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/°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 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 bromide (BDH) was recrystallised and dried under vacuum at 110-125 °C.

N-methylformamide was purified by fractionation 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:

(1) Potassium bromide; KBr; [7758-02-3]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium bromide in N-methylformamide at 25 °C
10.2 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 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 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 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^{-4} $S\ m^{-1}$.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:

(1) Potassium bromide; KBr; [7758-02-3]

(2) N-methylformamide; C_2H_5NO; [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 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 are within 0.6 to 2.5% of those derived by direct measurement.

SOURCE AND PURITY OF MATERIALS: (continuation)

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.*, 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:

1) Potassium bromide; KBr; [7758-02-3]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

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

9.95 g/100 g solvent

Within the temperature range 0-35 °C the solubilities in g/100 g of solvent, S, are given by:

$$S = -0.012\,T + 13.79$$

where T is the absolute temperature.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of potassium bromide and 10 cm^3 of N-methylformamide in a carefully stoppered test tube were placed in a constant temperature bath for 2-4 days. The test tubes were shaken several times each day. Saturation was assumed when samples taken at least one day apart gave identical concentrations. 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 100 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 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)

SOURCE AND PURITY OF MATERIALS:

Anhydrous A.R. grade potassium bromide 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 methylamine 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 10^{-1} and 10^{-2} S m^{-1}. It was stored

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Berger, C.; Dawson, L.R. *Anal. Chem.*, 1952, *24*, 994-996.

(continued)

COMPONENTS:	EVALUATOR:
(1) Potassium iodide; KI; [7681-11-0] (2) N-methylformamide; C_2H_5NO; [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 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.

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
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),* 1966, 212-219.

COMPONENTS:

(1) Potassium iodide; KI; [7681-11-0]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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/ °C	g/100 g solvent
25	44.2
35	45.9
45	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 fractionation 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:

(1) Potassium iodide; KI; [7681-11-0]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

Berardelli, M.L.; Pistoia, G.; Polcaro,A.M.

Ric. Sci., 1968, *38*, 814-819

VARIABLES:

One temperature: 25 °C

PREPARED BY:

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 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 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 °C (author)

REFERENCES:

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

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0] (2) N-methylformamide; C_2H_5NO; [123-39-7]	Strack, G.A.; Swanda, K.S.; Bahe, L.W. *J. Chem. Eng. Data*, 1964, *9*, 416-418
VARIABLES:	**PREPARED BY:**
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

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

48.6 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of potassium iodide and 10 cm^3 of N-methylformamide in a carefully stoppered test tube were placed in a constant temperature bath for 2-4 days. The test tubes were shaken several times each day. Saturation was assumed when samples taken at least one day apart gave identical concentrations. 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 100 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 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)

SOURCE AND PURITY OF MATERIALS:

Anhydrous A.R. grade potassium iodide 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 methylamine 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 10^{-1} and 10^{-2} S m^{-1}. It was stored in tightly

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Berger, C.; Dawson, L.R. *Anal. Chem.*, 1952, *24*, 994-996.

(continued)

COMPONENTS:

(1) Potassium iodide; KI; [7681-11-0]

(2) N-methylformamide; C_2H_5NO; [23-39-7]

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 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^{25} = 1.4305 [1.4300(4), 1.4310(2)]; ρ_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.*, 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) Potassium thiocyanate; KCNS; [333-20-0] (2) N-methylformamide; C_2H_5NO; [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 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 ± 7 g/kg of solvent.

References

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.

COMPONENTS: (1) Potassium thiocyanate; KCNS;[333-20-0] (2) N-methylformamide; C_2H_5NO;[123-39-7]	ORIGINAL MEASUREMENTS: 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/°C	g/100 g solvent
25	80.9
35	77.3
45	73.1

AUXILIARY INFORMATION

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.

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 fractionation 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:	EVALUATOR:
(1) Ammonium chloride; NH_4Cl; [12125-02-9] (2) N-methylformamide; C_2H_5NO; [123-39-7]	C.A. Vincent, Department of Chemistry, University of St. Andrews, St. Andrews, Fife, Scotland. December 1978.

CRITICAL EVALUATION:

Two investigations of the solubility of NH_4Cl 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.

References

1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. *J. Chem. Eng. Data,* 1964, *9,* 416-418.
2. 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.

COMPONENTS:

(1) Ammonium chloride; NH_4Cl; [12125-02-9]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	6.59
35	5.57
45	3.81

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of ammonium 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade ammonium chloride (BDH) was recrystallised and dried under vacuum at 110-125 °C.

N-methylformamide was purified by fractionation 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:

(1) Ammonium chloride; NH_4Cl; [12125-02-9]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of ammonium chloride in N-methylformamide at 25 °C

5.03 g/100 g solvent

Within the temperature range 0-35 °C the solubilities in g/100 g of solvent, S, are given by:

$$S = 0.020\,T \quad -0.977$$

where T is the absolute temperature.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of ammonium chloride and 10 cm^3 of N-methylformamide in a carefully stoppered test tube were placed in a constant temperature bath for 2-4 days. The test tubes were shaken several times each day. Saturation was assumed when samples taken at least one day apart gave identical concentrations. 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 100 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 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. As the densities were used to calculate concentrations on the molar scale the

(continued)

SOURCE AND PURITY OF MATERIALS:

Anhydrous A.R. grade ammonium 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 methylamine 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 10^{-1} and 10^{-2} S m^{-1}. It was stored in tightly-

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Berger, C.; Dawson, L.R. *Anal. Chem.*, 1952, *24*, 994-996.

(continued)

COMPONENTS:

(1) Ammonium chloride; NH_4Cl; [12125-02-9]

(2) N-methylformamide; C_2H_5NO; [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)

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 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 are within 0.6 - 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)]; ρ_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

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:

(1) Ammonium bromide; NH_4Br; [12124-97-9]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

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 NH_4Br 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 $\pm$ 44 g/kg or $\pm$15% to encompass the two experimental values. The only reasonable conclusion that may be drawn from these results is that the minimum solubility of NH_4Br at 298 K is likely to be $\gtrsim$ 237 g/kg of solvent.

References

1. Strack, G.A.; Swanda, K.S.; Bahe, L.W. *J. Chem. Eng. Data,* 1964, *9,* 416-418.
2. 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.

COMPONENTS:

(1) Ammonium bromide; NH_4Br; [12124-97-9]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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/°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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade ammonium bromide (BDH) was recrystallised and dried under vacuum at 110-125 °C.

N-methylformamide was purified by fractionation 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:

(1) Ammonium bromide; NH_4Br; [12124-97-9]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

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

23.7 g/100 g solvent

Within the temperature range 0-35 °C the solubilities in g/100 g of solvent, S, are given by:

$$S = 0.000\,T + 23.99$$

where T is the absolute temperature.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of ammonium bromide and 10 cm^3 of N-methylformamide in a carefully stoppered test tube were placed in a constant temperature bath for 2-4 days. The test tubes were shaken several times each day. Saturation was assumed when samples taken at least one day apart gave identical concentrations. 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 100 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 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. As the densities were used to calculate

(continued)

SOURCE AND PURITY OF MATERIALS:

Anhydrous A.R. grade ammonium bromide 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 methylamine 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 10^{-1}

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

1. Berger, C.; Dawson, L.R. *Anal. Chem.*, 1952, *24*, 994-996.

(continued)

COMPONENTS:

(1) Ammonium bromide;NH_4Br; [12124-97-9]

(2) N-methylformamide; C_2H_5NO; [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)

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 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 are within 0.6 - 2.5% of those derived by direct measurement.

SOURCE AND PURITY 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)]; ρ_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.*, 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_4I; [12027-06-4] (2) N-methylformamide; C_2H_5NO; [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 solutio
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.

318 K

821 ± 25 g/kg of solvent.

References

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

COMPONENTS:

(1) Ammonium iodide; NH_4I; [12027-06-4]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	38.0
35	58.5
45	82.1

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of ammonium 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 ammonium iodide (BDH) was recrystallised and dried under vacuum at 110-125 °C.

N-methylformamide was purified by fractionation 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:	EVALUATOR:
(1) Ammonium thiocyanate; NH_4CNS; [1762-95-4] (2) N-methylformamide; C_2H_5NO; [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 K

744 ± 15 g/kg of solvent

308 K

717 ± 14 g/kg of solvent

318 K

652 ± 13 g/kg of solvent.

References

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.

COMPONENTS:

(1) Ammonium thiocyanate; NH_4CNS; [1762-95-4]

(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	74.4
35	71.7
45	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 fractionation 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:	EVALUATOR:
(1) Magnesium chloride; $MgCl_2$; [7786-30-3] (2) N-methylformamide; C_2H_5NO; [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 $MgCl_2$ 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. An additional problem is that the investigators did not follow the recommended method for preparing anhydrous $MgCl_2$ (by passing dry HCl over the hydrated double salt, $MgCl_2 \cdot NH_4Cl$), 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 ± 2 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.

COMPONENTS:

(1) Magnesium chloride; $MgCl_2$; [7786-30-3]
(2) N-methylformamide; C_2H_5NO; [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 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of magnesium chloride in N-methylformamide at 25 °C

8.80 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 magnesium 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 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^{-4}S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%
Temperature ± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:	EVALUATOR:
(1) Calcium chloride; $CaCl_2$; [10043-52-4] (2) N-methylformamide; C_2H_5NO; [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 $CaCl_2$ 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.

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.

COMPONENTS:

(1) Calcium chloride; $CaCl_2$; [10043-52-4]
(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

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

18.6 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 calcium 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 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 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-methylformamide 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. *Ric. Sci.*, 1967, *37*, 1167-1172

COMPONENTS:	EVALUATOR:
(1) Calcium bromide; $CaBr_2$; [7789-41-5] (2) N-methylformamide; C_2H_5NO; [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 ± 9 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.

COMPONENTS:

(1) Calcium bromide; $CaBr_2$; [7789-41-5]
(2) N-methylformamide; C_2H_5NO; [123-39-7]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

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 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 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^{-4}S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

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

COMPONENTS:	EVALUATOR:
(1) Lithium fluoride; LiF; [7789-24-4] (2) N,N-dimethylformamide; C_3H_7NO; [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 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 x 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. $2H_2O$. 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

$(1.39 \pm 0.18) \times 10^{-3}$ 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] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Criss, C.M.; Luksha, E. *J. Phys. Chem.*, 1968, *72*, 2966-2970.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of lithium fluoride in N,N-dimethylformamide at 25 °C

1.39×10^{-4} g/100 g solvent

Value calculated from solubility data given as:

$5.34 \pm 0.69 \times 10^{-5}$ mol/kg of N,N-dimethylformamide.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of lithium 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 supersaturation. 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%.

SOURCE AND PURITY OF MATERIALS:

Lithium fluoride was prepared by the dropwise addition of aqueous HF to an aqueous solution of lithium chloride in a polyethylene beaker. When precipitation was complete, the LiF was filtered and washed repeatedly with conductivity water, before drying at 150 °C under vacuum for 24 hours and storing in sealed vials until ready for use.

(continued)

ESTIMATED ERROR:

Solubility ± 13%

Temperature ± 0.05 °C (author)

REFERENCES:

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

(continued)

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium fluoride; LiF; [7789-24-4] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	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 (1). The specific conductance was in the range 0.32 - 1.5 x 10^{-5} S m^{-1}, which is in good agreement with the lowest values reported in the literature (0.6 - 18.3 x 10^{-5} 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:
(1) Lithium chloride; LiCl; [7447-41-8] (2) N,N-dimethylformamide; C_3H_7NO; [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 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.$2H_2O$. 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.

References

1. Paul, R.C.; Sreenathan, B.R. *Indian J. Chem.*, 1966, *4*, 382-386.
2. Pistoia, G.; Pecci, G.; Scorsati, 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.*, 1973, *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: (1) Lithium chloride; LiCl; [7447-41-8] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	ORIGINAL MEASUREMENTS: Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of lithium chloride in N,N-dimethylformamide at 25 °C

11.40 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: Not stated	SOURCE AND PURITY OF MATERIALS: Not stated
	ESTIMATED ERROR:
	REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium chloride; LiCl; [7447-41-8] (2) N,N-dimethylformamide; C_3H_7NO; [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/°C	g/100 g solvent
25	13.8
35	12.3
45	11.6

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of lithium 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade lithium chloride (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:

COMPONENTS:

(1) Lithium chloride; LiCl; [7447-41-8]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Pistoia, G.; Pecci, G.; Scrosati, B.

Ric. Sci., 1967, *37*, 1167-1172

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of lithium chloride in N,N-dimethylformamide at 25 °C

11.3 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 the dry box. Saturated lithium 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 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.

SOURCE AND PURITY OF MATERIALS:

Lithium 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 ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:

(1) Lithium chloride; LiCl; [7447-41-8]
(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of lithium chloride in N,N-dimethylformamide at 25 °C

27.5 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of lithium 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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

Purity of lithium 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 3×10^{-5} S m^{-1} at 25 °C.

ESTIMATED ERROR:

Solubility ± 1%
Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Lithium bromide; LiBr; [7550-35-8] (2) N,N-dimethylformamide; C_3H_7NO; [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 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.$2H_2O$. 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.
2. 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.

COMPONENTS:

(1) Lithium bromide; LiBr; [7550-35-8]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	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 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 lithium 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:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium bromide; LiBr; [7550-35-8] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1167-1172
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of lithium bromide in N,N-dimethylformamide at 25 °C

16.6 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 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 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.

SOURCE AND PURITY OF MATERIALS:

Lithium 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 3×10^{-5} S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium fluoride; NaF; [7681-49-4] (2) N,N-dimethylformamide; C_3H_7NO; [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 x 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 $DMF.2H_2O$. 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

$(1.96 \pm 0.14) \times 10^{-3}$ 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:

(1) Sodium fluoride; NaF; [7681-49-4]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium fluoride in N,N-dimethylformamide at 25 °C

1.96×10^{-4} g/100 g solvent

Value calculated from solubility data given as:

$4.68 \pm 0.34 \times 10^{-5}$ mol/kg N,N-dimethylformamide

AUXILIARY 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 supersaturation. 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%.

SOURCE AND PURITY OF MATERIALS:

Sodium fluoride was prepared by the dropwise addition of aqueous HF to an aqueous solution of sodium chloride in a polyethylene beaker. When precipitation was complete, the NaF was filtered and washed repeatedly with conductivity water, before drying at 150 °C under vacuum for 24 hours and storing in sealed vials until ready for use.

(continued)

ESTIMATED ERROR:

Solubility ± 7%

Temperature ± 0.05 °C (author)

REFERENCES:

(1) Prue, J.E.; Sherrington, P.J.
Trans. Faraday Soc., 1961, *57*, 1795-180

(continued)

COMPONENTS:

(1) Sodium fluoride; NaF; [7681-49-4]

(2) N,N-dimethylformamide; C_3H_7NO; [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 x 10^{-5} S m^{-1}, which is in good agreement with the lowest values reported in the literature (0.6 - 18.3 x 10^{-5} 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:
(1) Sodium chloride; NaCl; [7647-14-5] (2) N,N-dimethylformamide; C_3H_7NO; [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 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 x 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 $DMF.2H_2O$. 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 ≤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.

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. Criss, C.M.; Luksha, E. *J. Phys. Chem.*, 1968, *72*, 2966-2970.
4. "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:	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; [7647-14-5] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Properties and uses of DIMETHYLFORMAMIDE (DMF) Du Pont Information Booklet, 1976.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium chloride in N,N-dimethylformamide at 25 °C

0.05 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:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; [7647-14-5] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Criss, C.M.; Luksha, E. *J. Phys. Chem.*, 1968, *72*, 2966-2970.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium chloride in N,N-dimethylformamide at 25 °C

0.036 g/100 g solvent

Value calculated from solubility data given as:

$6.08 \pm 0.06 \times 10^{-3}$ mol/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 supersaturation. 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%.

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)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

(1) Ives, D.J.G.; Janz, G.J.
Reference Electrodes- Theory and Practice,
Academic Press, Inc., New York, N.Y. 1961.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium chloride; NaCl; [7647-14-5] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	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.

COMPONENTS:

(1) Sodium chloride; NaCl; [7647-14-5]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Pistoia, G.; Pecci, G.; Scrosati, B.

Ric. Sci., 1967, *37*,1167-1172.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium chloride in N,N-dimethylformamide at 25 °C

0.042 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 sodium 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 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.

SOURCE AND PURITY OF MATERIALS:

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 3×10^{-5} S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:

(1) Sodium chloride; NaCl; [7647-14-5]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Paul, R.C.; Sreenathan, B.R.

Indian J. Chem., 1966, *4*, 382-386.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium chloride in N,N-dimethylformamide at 25°C

0.05 g/100 g solvent

AUXILIARY 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 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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

Purity of sodium 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.

ESTIMATED ERROR:

Solubility ± 10%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium bromide; NaBr; [7647-15-6] (2) N,N-dimethylformamide; C_3H_7NO; [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 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 $DMF.2H_2O$. 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.

Values at 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.

References

1. Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1167-1172.
2. 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.

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	13.3
35	10.9
45	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:

COMPONENTS:

(1) Sodium bromide; NaBr; [7647-15-6]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Pistoia, G.; Pecci, G.; Scrosati, B.

Ric. Sci., 1967, *37*, 1167-1172.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium bromide in N,N-dimethylformamide at 25 °C

10.3 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 the dry box. Saturated sodium 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.

SOURCE AND PURITY OF MATERIALS:

Sodium 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 3×10^{-5} S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium iodide; NaI; [7681-82-5] (2) N,N-dimethylformamide; C_3H_7NO; [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 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 $DMF.2H_2O$. 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.*, 1973, *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.
6. Alexander, R.; Ko, E.C.F.; Mac. Y.C.; Parker, A.J. *J. Am. Chem. Soc.*, 1967, *89*, 3703-3712.

COMPONENTS: (1) Sodium iodide; NaI; [7681-82-5] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	ORIGINAL MEASUREMENTS: Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium iodide in N,N-dimethylformamide at 25 °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: Not stated	SOURCE AND PURITY OF MATERIALS: Not stated
	ESTIMATED ERROR:
	REFERENCES:

COMPONENTS:

(1) Sodium iodide; NaI; [7681-11-0]

(2) N,N-dimethylformamide; C_3H_7NO;[68-12-2]

ORIGINAL MEASUREMENTS:

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/°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 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,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:

COMPONENTS:

(1) Sodium iodide; NaI; [7681-82-5]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Pistoia, G.; Pecci, G.; Scrosati, B.

Ric. Sci., 1967, *37*, 1167-1172

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium iodide in N,N-dimethylformamide at 25 °C

6.35 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 sodium 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.

SOURCE AND PURITY OF MATERIALS:

Sodium iodide, 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 ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium iodide; NaI; [7681-82-5] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Paul, R.C.; Sreenathan, B.R. *Indian J. Chem.*, 1966, *4*, 382-386.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium iodide in N,N-dimethylformamide at 25°C

3.72 g/100 g solvent

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 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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

Purity of sodium iodide 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 3×10^{-5} S m^{-1} at 25 °C.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium cyanide; NaCN; [143-33-9] (2) N,N-dimethylformamide; C_3H_7NO; [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 x 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.$2H_2O$. 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:

(1) Sodium cyanide; NaCN; [143-33-9]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium cyanide in N,N-dimethylformamide at 25 °C

0.76 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:

Not stated

SOURCE AND PURITY OF MATERIALS:

Not stated

ESTIMATED ERROR:

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Sodium cyanide; NaCN; [143-33-9] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Paul, R.C.; Sreenathan, B.R. *Indian J. Chem.*, 1966 *4*, 382-386.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium cyanide in N,N-dimethylformamide at 25 °C

18.8 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of sodium cyanide 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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

Purity of sodium cyanide 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 3×10^{-5} S m^{-1} at 25 °C.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium cyanate; NaCNO; (2) N,N-dimethylformamide; C_3H_7NO; [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 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.

References

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:

(1) Sodium cyanate; NaCNO

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium cyanate in N,N-dimethylformamide at 25 °C

0.05 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:

Not stated

SOURCE AND PURITY OF MATERIALS:

Not stated

ESTIMATED ERROR:

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium thiocyanate; NaCNS; [540-72-7] (2) N,N-dimethylformamide; C_3H_7NO; [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 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 $DMF.2H_2O$. 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.

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.*, 1973, *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) Sodium thiocyanate; NaCNS;[540-72-7] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of soidum thiocyanate in N,N-dimethylformamide at 25 °C

29.2 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:

COMPONENTS:

(1) Sodium thiocyanate; NaCNS; [540-72-7]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	29.5
35	20.9
45	14.9

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of sodium thiocyanate 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 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 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:

COMPONENTS:

(1) Sodium thiocyanate; NaCNS; [540-72-7]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Pistoia, G.; Pecci, G.; Scrosati, B.

Ric. Sci., 1967, *37*, 1167-1172

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium thiocyanate in N,N-dimethylformamide at 25 °C

29.9 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 sodium thiocyanate 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 presumably by Mohr titration using dichlorofluorescein 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.

SOURCE AND PURITY OF MATERIALS:

Sodium thiocyanate, 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 ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:

(1) Sodium thiocyanate; NaCNS; [540-72-7]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Paul, R.C.; Sreenathan, B.R.

Indian J. Chem., 1966, *4*, 382-386

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of sodium thiocyanate in N,N-dimethylformamide at 25 °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 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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

Purity of sodium thiocyanate 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 3×10^{-5} S m^{-1} at 25 °C.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Potassium chloride; KCl; [7447-40-7] (2) N,N-dimethylformamide; C_3H_7NO; [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 KCl 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 x 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.$2H_2O$. 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 ≤ 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.

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. Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. *J. Am. Chem. Soc.*, 1967, *89*, 3703-3712.
4. Criss, C.M.; Luksha, E. *J. Phys. Chem.*, 1968, *72*, 2966-2970.
5. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. *Indian J. Chem.*, 1973, *11*, 1024-1026.
6. "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.*, 1957, *79*, 1843-1848.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KCl; [7447-40-7] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet,1976.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubilities of potassium chloride in N,N-dimethylformamide at 25 °C

0.05 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:

COMPONENTS:

(1) Potassium chloride; KCl; [7447-40-7]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	0.05
35	0.04
45	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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade potassium chloride (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:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KCl; [7447-40-7] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Criss, C.M.; Luksha, E.. *J. Phys. Chem.*, <u>1968</u>, *72*, 2966-2970.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium chloride in N,N-dimethylformamide at 25 °C

0.017 g/100 g solvent

Value calculated from solubility data given as:

$2.28 \pm 0.07 \times 10^{-3}$ mol/kg N,N-dimethylformamide

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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 which oscillated 200 times/minute through an arc of about 30°.

Solubilities were obtained by approaching equilibrium from both under- and supersaturation. 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%.

SOURCE AND PURITY OF MATERIALS:

Reagent grade potassium chloride was further purified by double recrystallisation from conductivity water. The salt was stored at 400 °C until ready for use.

(continued)

ESTIMATED ERROR:

Solubility ± 3%

Temperature ± 0.05 °C (author)

REFERENCES:

(1) Prue, J.E.; Sherrington, P.J. *Trans. Faraday Soc.*, <u>1961</u>, *57*, 1795-1808

(continued)

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KCl; [7447-40-7] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	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 (1). The specific conductance was in the range 0.32 - 1.5 x 10^{-5} S m^{-1}, which is in good agreement with the lowest values reported in the literature (0.6 - 18.3 x 10^{-5} 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) Potassium chloride; KCl; [7447-40-7]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Pistoia, G.; Pecci, G.; Scrosati, B.

Ric. Sci., 1967, *37*, 1167-1172

VARIABLES:

One temperature: 25 °C

PREPARED BY:

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:

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 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.

SOURCE AND PURITY OF MATERIALS:

Potassium 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}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:

(1) Potassium chloride; KCl; [7447-40-7]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium chloride in N,N-dimethylformamide at 25 °C

0.15 g/1000 cm^3 of saturated solution.

Value calculated from solubility product data given as:

$\log(K_s o/\text{mol dm}^{-3}) = -5.4$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of potassium 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 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 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^{-3} $AgNO_3$ solution into a half-cell containing the silver were immersed in 20 cm^3 of 0.01 mol dm^{-3} potassium chloride in DMF. The reference half-cell was a silver wire in 0.01 mol dm^{-3} $AgNO_3$ in the same solvent.

SOURCE AND PURITY OF MATERIALS:

A.R. grade potassium chloride was used withou 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 °C (compiler)

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium chloride; KCl; [7447-40-7] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Paul, R.C.; Sreenathan, B.R. *Indian J. Chem.*, 1966, *4*, 382-386
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium chloride in N,N-dimethylformamide at 25 °C

0.05 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

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 3×10^{-5} $S\ m^{-1}$ at 25 °C.

ESTIMATED ERROR:

Solubility ± 10%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Potassium bromide; KBr; [7758-02-3] (2) N,N-dimethylformamide; C_3H_7NO; [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 x 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.$2H_2O$. 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

References

1. Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1167-1172.
2. Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. *J. Am. Chem. Soc.*, 1967, *89*, 3703-3712.
3. 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.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium bromide; KBr; [7758-02-3] (2) N,N-dimethylformamide; C_3H_7NO; [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/°C	g/100 g solvent
25	0.12
35	0.10
45	0.09

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of potassium 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 potassium 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:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium bromide; KBr; [7758-02-3] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1167-1172.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium bromide in N,N-dimethylformamide at 25 °C

0.82 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 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.

SOURCE AND PURITY OF MATERIALS:

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 3×10^{-5} S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:

(1) Potassium bromide; KBr; [7758-02-3]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie.

EXPERIMENTAL VALUES:

Solubility of potassium bromide in N,N-dimethylformamide at 25 °C

7.51 g/1000 cm^3 of saturated solution

Value calculated from solubility product data given as:

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

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of potassium 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 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^{-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^{-3} $AgNO_3$ solution into a half-cell containing 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} $AgNO_3$ in the same solvent.

SOURCE AND PURITY OF MATERIALS:

A.R. grade potassium 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} $AgNO_3$ for the potentiometric titrations was also stored in a light proof container.

ESTIMATED ERROR:

Solubility ± 2%

Temperature ± 0.1 °C (compiler).

REFERENCES:

COMPONENTS:

(1) Potassium iodide; KI; [7681-11-0]

(2) N,N-dimethylformamide; C_3H_7NO;[68-12-2]

EVALUATOR:

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 $DMF.2H_2O$. 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/dm^3 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

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. Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. *J. Am. Chem. Soc.*, 1967, *89*, 3703-3712.
4. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. *Indian J. Chem.*, 1973, *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.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0] (2) N,N-dimethylformamide; C_3H_7NO;[68-12-2]	Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium iodide in N,N-dimethylformamide at 25 °C

25 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:

COMPONENTS:

(1) Potassium iodide; KI; [7681-11-0]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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/°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,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 ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Pistoia, G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1167-1172
VARIABLES: One temperature: 25 °C	PREPARED BY: 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^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.

SOURCE AND PURITY OF MATERIALS:

Potassium iodide, reagent grade, was dried under vacuum before use.

Reagent grade N,N-dimethylformamide (C. Erba RP) was purified by fractional distillation under reduced pressure (2mm Hg). The final product had a specific conductance of 3×10^{-5} S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium iodide; KI; [7681-11-0] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. *J. Am. Chem. Soc.*, 1967, *89*, 3703-3712.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium iodide in N,N-dimethylformamide at 25 °C

295.21 g/1000 cm^3 of saturated solution

Value calculated from solubility product data given as:

$\log (K_s o/\text{mol dm}^{-3}) = 0.5$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of potassium 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 °C. The liquid phase was analysed for halide potentionmetrically 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^{-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^{-3} $AgNO_3$ solution into a half-cell containing the silver wire immersed in 20 cm^3 of 0.01 mol dm^{-3} potassium iodide in DMF. The reference half-cell was a silver wire in 0.01 mol dm^{-3} $AgNO_3$ in the same solvent.

SOURCE AND PURITY OF MATERIALS:

A.R. grade potassium iodide 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} $AgNO_3$ for the potentiometric titrations was also stored in a light proof container.

ESTIMATED ERROR:

Solubility ± 2%

Temperature ± 0.1 °C (compiler)

REFERENCES:

COMPONENTS:

(1) Potassium iodide; KI; [7681-82-5]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Paul, R.C.; Sreenathan, B.R.

Indian J. Chem., 1966, *4*, 382-386.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium iodide in N,N-dimethylformamide at 25 °C

13.7 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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 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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

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 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 3×10^{-5} S m^{-1} at 25 °C.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Potassium cyanide; KCN; [151-50-8] (2) N,N-dimethylformamide; C_3H_7NO; [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 ± 0.2 g/kg of solvent

References

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: (1) Potassium cyanide; KCN; [151-50-8] (2) N,N-dimethylformamide; C_3H_7NO;[68-12-2]	ORIGINAL MEASUREMENTS: Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.
VARIABLES: One temperature: 25 °C	PREPARED BY: 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-quantitative solubilities of inorganic materials in DMF at 25 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: Not stated	SOURCE AND PURITY OF MATERIALS: Not stated
	ESTIMATED ERROR:
	REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Potassium cyanate; KCNO; (2) N,N-dimethylformamide; C_3H_7NO; [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 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 ± 0.2 g/kg of solvent

References

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: (1) Potassium cyanate; KCNO; (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	ORIGINAL MEASUREMENTS: Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium cyanate in N,N-dimethylformamide at 25 °C

0.12 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: Not stated	SOURCE AND PURITY OF MATERIALS: Not stated.
	ESTIMATED ERROR:
	REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Potassium thiocyanate; KCNS; [333-20-2] (2) N,N-dimethylformamide; C_3H_7NO; [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 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 x 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 $DMF.2H_2O$. 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.

References

1. Paul, R.C.; Sreenathan, B.R. *Indian J. Chem.*, 1966, *4*, 382-386.
2. 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.

COMPONENTS:

(1) Potassium thiocyanate; KCNS; [333-20-0]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

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-quantitative solubilities of inorganic materials in DMF at 25 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Not stated

SOURCE AND PURITY OF MATERIALS:

Not stated

ESTIMATED ERROR:

REFERENCES:

COMPONENTS:

(1) Potassium thiocyanate; KCNS; [333-20-0]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	40.1
35	29.8
45	27.9

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of potassium thiocyanate 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 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 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 2.5×10^{-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:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium thiocyanate; KCNS; [333-20-0] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Paul, R.C.; Sreenathan, B.R. *Indian J. Chem.*, 1966, *4*, 382-386.
VARIABLES:	**PREPARED BY:**
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of potassium thiocyanate in N,N-dimethylformamide at 25 °C

16.0 g /100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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 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.

SOURCE AND PURITY OF MATERIALS:

Purity of potassium thiocyanate was notstated 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.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Caesium chloride; CsCl; [7647-17-8] (2) N,N-dimethylformamide; C_3H_7NO; [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 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 $DMF.2H_2O$. 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.

References

1. 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.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Caesium chloride; CsCl; [7647-17-8] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Criss, C.M.; Luksha, E. *J. Phys. Chem.*, 1968, *72*, 2966-2970.
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium chloride in N,N-dimethylformamide at 25 °C

0.052 g/100 g solvent

Value calculated from solubility date given as:

$3.06 \pm 0.02 \times 10^{-3}$ mol/kg N,N-dimethylformamide

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of caesium 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 supersaturation. 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%.

SOURCE AND PURITY OF MATERIALS:

Caesium chloride (Henley and Co., 99.95% pure) was used without further purification, except for the removal of water by drying in an air oven with the temperature being gradually raised to about 600 °C over a period of 24 hours.

The salt was stored at 400 °C until ready for use.

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

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

(continued)

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Caesium chloride; CsCl; [7647-17-8] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	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 (1). The specific conductance was in the range 0.32 - 1.5 x 10^{-5} S m^{-1}, which is in good agreement with the lowest values reported in the literature (0.6 - 18.3 x 10^{-5} 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) Caesium chloride; CsCl; [7647-17-8]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium chloride in N,N-dimethylformamide at 25 °C

0.60 g/1000 cm^3 of saturated solution

Value calculated from solubility product data given as:

$\log(K_{so}/\text{mol dm}^{-3}) = -4.9$

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 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 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^{-3} $AgNO_3$ solution into a half-cell containing the silver wire immersed in 20 cm^3 of 0.01 mol dm^{-3} caesium chloride in DMF.

The reference half-cell was a silver wire in 0.01 mol dm^{-3} $AgNO_3$ in the same solvent.

SOURCE AND PURITY OF MATERIALS:

A.R. grade caesium chloride 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} $AgNO_3$ for the potentiometric titrations was also stored in a light proof container.

ESTIMATED ERROR:

Solubility ± 2%

Temperature ± 0.1 °C (compiler)

REFERENCES:

COMPONENTS:

(1) Caesium bromide; CsBr; [7787-69-1]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

EVALUATOR:

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 $DMF.2H_2O$. 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.

References.

1. 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.

COMPONENTS:

(1) Caesium bromide; CsBr; [7787-69-1]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium bromide in N,N-dimethylformamide at 25 °C

0.56 g/100 g solvent

Value calculated from solubility data given as:

$2.62 \pm 0.01 \times 10^{-2}$ mol/kg N,N-dimethylformamide

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of caesium bromide 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 supersaturation. 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%.

SOURCE AND PURITY OF MATERIALS:

Caesium bromide (Henley and Co., 99.9% pure) was used without further purification, except that it was dried by heating at 400 °C.

(continued)

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C

REFERENCES:

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

(continued)

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Caesium bromide; CsBr; [7787-69-1] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	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 (1). The specific conductance was in the range 0.32 - 1.5 x 10^{-5} S m^{-1}, which is in good agreement with the lowest values reported in the literature (0.6 - 18.3 x 10^{-5} 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) Caesium bromide; CsBr; [7787-69-1]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium bromide in N,N-dimethylformamide at 25 °C

4.76 g/1000 cm^3 of saturated solution

Value calculated from solubility product data given as:

$\log(K_s o/\text{mol dm}^{-3}) = -3.3$

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 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^{-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^{-3} $AgNO_3$ solution into a half-cell containing the silver wire immersed in 20 cm^3 of 0.01 mol dm^{-3} caesium bromide in DMF.
The reference half-cell was a silver wire in 0.01 mol dm^{-3} $AgNO_3$ 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} $AgNO_3$ for the potentiometric titrations was also stored in a light proof container.

ESTIMATED ERROR:

Solubility ± 2%

Temperature ± 0.1 °C (compiler)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Caesium iodide; CsI; [7789-17-5] (2) N,N-dimethylformamide; C_3H_7NO; [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.2H_2O. 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 ± 4.0 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. Thomas, A.B.; Rochow, E.G. *J. Am. Chem. Soc.*, 1957, *79*, 1843-1848.

COMPONENTS: (1) Caesium iodide; CsI; [7789-17-5] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	ORIGINAL MEASUREMENTS: Alexander, R.; Ko, E.C.F.; Mac, Y.C.; Parker, A.J. *J. Am. Chem. Soc.*, 1967, *89*, 3703-3712.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of caesium iodide in N,N-dimethylformamide at 25 °C

36.70 g/1000 cm^3 of saturated solution

Value calculated from solubility product data given as:

$\log(K_s o/\text{mol dm}^{-3}) = -1.7$

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 °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^{-3} silver nitrate was added. The emf was measured on a Radiometer pH meter (type PHM 22r). It was estabilshed 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^{-3} $AgNO_3$ solution into a half-cell containing the silver wire immersed in 20 cm^3 of 0.01 mol dm^{-3} caesium iodide in DMF.
The reference half-cell was a silver wire in 0.01 mol dm^{-3} $AgNO_3$ in the same solvent.

SOURCE AND PURITY OF MATERIALS:

A.R. grade caesium iodide 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} $AgNO_3$ for the potentiometric titrations was also stored in a light proof container.

ESTIMATED ERROR:

Solubility ± 2%

Temperature ± 0.1 °C (compiler)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Ammonium chloride; NH_4Cl; [12125-02-9] (2) N,N-dimethylformamide; C_3H_7NO; [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_4Cl 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 $DMF.2H_2O$. 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.

References.

1. Paul, R.C.; Sreenathan, B.R. *Indian J. Chem.*, 1966, *4*, 382-386.
2. 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.

COMPONENTS:

(1) Ammonium chloride; NH_4Cl; [12125-02-9]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of ammonium chloride in N,N-dimethylformamide at 25 °C

0.1 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:

Not stated

SOURCE AND PURITY OF MATERIALS:

Not stated

ESTIMATED ERROR:

REFERENCES:

COMPONENTS:

(1) Ammonium chloride; NH_4Cl; [12125-02-9]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	0.37
35	0.30
45	0.28

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of ammonium 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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade ammonium chloride (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 2.5×10^{-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:

COMPONENTS:

(1) Ammonium chloride; NH_4Cl; [12125-02-9]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Paul, R.C.; Sreenathan, B.R.

Indian J. Chem., 1966, *4*, 382-386.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of ammonium chloride in N,N-dimethylformamide at 25 °C

0.05 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of ammonium 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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

Purity of ammonium 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 3×10^{-5} S m^{-1} at 25 °C.

ESTIMATED ERROR:

Solubility ± 10%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Ammonium bromide; NH_4Br; [12124-97-9] (2) N,N-dimethylformamide; C_3H_7NO; [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 $DMF.2H_2O$. 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 NH_4Br it has therefore been decided to recommend as a tentative value the mean between the two reported solubilities.

Tentative value at 298 K

147 ± 20 g/kg of solvent.

References

1. Paul, R.C.; Singla, J.P.; Lamba, M.S.; Gill, D.S.; Narula, S.P. *Indian J. Chem.*, 1973, *11*, 1024-1026.
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) Ammonium bromide; NH_4Br; [12124-97-9] (2) N,N-dimethylformamide; C_3H_7NO; [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 bromide in N,N-dimethylformamide at 25 °C

12.7 g/100 g solvent (1)

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: (1) Jones, G.D. British Patent 654,855, July 4, 1951.

COMPONENTS:

(1) Ammonium bromide; NH_4Br; [12124-97-9]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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/°C	g/100 g solvent
25	16.6
35	15.7
45	13.8

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of ammonium 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 ammonium 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 2.5×10^{-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:

COMPONENTS:	EVALUATOR:
(1) Ammonium iodide; NH_4I; [12027-06-4] (2) N,N-dimethylformamide; C_3H_7NO; [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_4I 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.$2H_2O$. 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

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

COMPONENTS:

(1) Ammonium iodide; NH_4I; [12027-06-4]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

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/°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.

SOURCE AND PURITY OF MATERIALS:

A.R. grade ammonium 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)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Ammonium thiocyanate; NH_4CNS; [1762-95-4] (2) N,N-dimethylformamide; C_3H_7NO; [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 x 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 $DMF.2H_2O$. 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 NH_4CNS 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 $NH_4CNS.DMF$ (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 ± 2 g/kg of solvent.

References

1. Paul, R.C.; Sreenathan, B.R. *Indian J. Chem.*, 1966, *4*, 382-386.
2. 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.

COMPONENTS:

(1) Ammonium thiocyanate; NH_4CNS; [1762-95-4]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Properties and uses of DIMETHYLFORMAMIDE (DMF), Du Pont Information Booklet, 1976.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

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-quantitative solubilities of inorganic materials in DMF at 25 °C

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Not stated

SOURCE AND PURITY OF MATERIALS:

Not stated

ESTIMATED ERROR:

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ammonium thiocyanate; NH_4CNS; [1762-95-4] (2) N,N-dimethylformamide; C_3H_7NO; [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/°C	g/100 g solvent
25	15.6
35	17.0
45	18.6

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of ammonium thiocyanate 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 ammonium 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 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:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Ammonium thiocyanate; NH_4CNS;[1762-95-4] (2) N,N-dimethylformamide; C_3H_7NO; 68-12-2	Paul, R.C.; Sreenathan, B.R. *Indian J. Chem.*,1966, *4*, 382-386.
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of ammonium thiocyanate in N,N-dimethylformamide at 25 °C

13.4 g/100 g solvent.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of ammonium 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 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.

SOURCE AND PURITY OF MATERIALS:

Purity of ammonium thiocyanate 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.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Magnesium chloride; $MgCl_2$; [7786-30-3] (2) N,N-dimethylformamide; C_3H_7NO; [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 $MgCl_2$ 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 $MgCl_2$ 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 $MgCl_2$. 2 DMF and $MgCl_2$. 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.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Magnesium chloride; $MgCl_2$; [7786-30-3] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Pistoia. G.; Pecci, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1167-1172
VARIABLES: One temperature: 25 °C	PREPARED BY: J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of magnesium chloride in N,N-dimethylformamide at 25 °C

12.5 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 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 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 dichlorofluorescein 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.

SOURCE AND PURITY OF MATERIALS:

Magnesium 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 ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:

(1) Magnesium chloride; $MgCl_2$[7786-30-3]

(2) N,N-dimethylformamide: C_3H_7NO;[68-12-2]

ORIGINAL MEASUREMENTS:

Paul, R.C.; Sreenathan, B.R.

Indian J. Chem., 1966, *4*, 382-386.

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of magnesium chloride in N, N-dimethylformamide at 25 °C

8.04 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

Purity of magnesium 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.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Magnesium iodide; MgI_2; [10377-58-9] (2) N,N-dimethylformamide; C_3H_7NO; [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 MgI_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 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.$2H_2O$. 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.

COMPONENTS:

(1) Magnesium iodide; MgI_2 ; [10377-58-9]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Pistoia, G.; Pecci, G.; Scrosati, B.

Ric. Sci., 1967, *37*, 1167-1172

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of magnesium iodide in N,N-dimethylformamide at 25 °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.

SOURCE AND PURITY OF MATERIALS:

Magnesium iodide, 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 ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Calcium chloride; $CaCl_2$; [10043-52-4] (2) N,N-dimethylformamide; C_3H_7NO; [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 $CaCl_2$ 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 $CaCl_2$ 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 $CaCl_2$. 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:

(1) Calcium chloride; $CaCl_2$; [10043-52-4]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Pistoia, G.; Pecci, G.; Scrosati, B.

Ric. Sci., 1967, *37*, 1167-1172

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of calcium chloride in N,N-dimethylformamide at 25 °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 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 Fajan's titration using dichlorofluorescein 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.

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 ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Calcium chloride; $CaCl_2$; [10043-52-4] (2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]	Paul, R.C.; Sreenathan, B.R., *Indian J. Chem.*, 1966, *4*, 382-386
VARIABLES:	PREPARED BY:
One temperature: 25 °C	J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of calcium chloride in N,N-dimethylformamide at 25 °C

1.98 g/100 g solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions of calcium 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 solution the metal was estimated and the solubility of the salt determined. No details of the analytical methods used were given.

SOURCE AND PURITY OF MATERIALS:

Purity of calcium 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.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.1 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Calcium bromide; $CaBr_2$; [7789-41-5] (2) N,N-dimethylformamide; C_3H_7NO; [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 $CaBr_2$ 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 $DMF.2H_2O$. 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 ± 2 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:

(1) Calcium bromide; $CaBr_2$; [7789-41-5]

(2) N,N-dimethylformamide; C_3H_7NO; [68-12-2]

ORIGINAL MEASUREMENTS:

Pistoia, G.; Pecci, G.; Scrosati, B.

Ric. Sci., 1967, *37*, 1167-1172

VARIABLES:

One temperature: 25 °C

PREPARED BY:

J.S. McKechnie

EXPERIMENTAL VALUES:

Solubility of calcium bromide in N,N-dimethylformamide at 25 °C

19.2 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 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 Fajan's titration using tetrabromofluorescein 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.

SOURCE AND PURITY OF MATERIALS:

Calcium 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 3×10^{-5} S m^{-1}.

ESTIMATED ERROR:

Solubility ± 1%

Temperature ± 0.05 °C (author)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium iodide; NaI; 7681-82-5 (2) Acetamide; C_2H_5NO; 60-35-5	Bruno Scrosati Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION:

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

T/K	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

COMPONENTS:

(1) Sodium iodide; NaI; 7681-82-5

(2) Acetamide; C_2H_5NO; 60-35-5

ORIGINAL MEASUREMENTS:

Sarkisov, A.G; Melamud, S.F.; Sakharova, N.F. *Khimiya,* 1969, 3-11

VARIABLES:

Temperatures

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of sodium oidide at three temperatures

t/°C	g/100 g of solution
70	34.0
80	35.9
90	37.2

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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 to a crucible. After evaporation of the solvent the residue was weighed and the concentration determined.

SOURCE AND PURITY OF MATERIALS:

Anhydrous and chemically pure potassium iodide was used. Acetamide was synthesised from acetic anhydride dissolved in ether and saturated with ammonia. Upon evaporation of the solvent, white crystals of acetamide precipitated. The product was then recrystallised 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 ± 5% (compiler)

REFERENCES:

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

COMPONENTS:

(1) Potassium chloride, KCl, 7447-40-7

(2) Acetamide, C_2H_5NO, 60-35-5

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy

March 1979

CRITICAL EVALUATION:

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.

COMPONENTS:

(1) Potassium chloride; KCl; 7447-40-7

(2) Acetamide; C_2H_5NO; 60-35-5

ORIGINAL MEASUREMENTS:

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

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of potassium chloride at various temperatures

$t/^{\circ}C$	g/100g of solvent
80	2.45
86	2.52
103	2.58
115	2.62
120	2.68
130	2.74

The solubitility data may be represented as a function of temperature by the following equation

$$\log c\ (g/100\ g) = 9.50 \times 10^{-4}t + 0.313$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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 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^3 flask. The solution was then weighed, dissolved in water, and titrated using the Volhard method.

SOURCE AND PURITY OF MATERIALS:

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 $8.5 \times 10^{-4}\ Sm^{-1}$ at 94°C.

ESTIMATED ERROR:

Temperature ± 0.1°C (author)

Solubility ± 1% (compiler)

REFERENCES:

COMPONENTS:

(1) Potassium bromide; KBr; 7558-02-3

(2) Acetamide; C_2H_5NO; 60-35-5

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy

March 1979

CRITICAL EVALUATION:

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.

COMPONENTS:

(1) Potassium bromide; KBr; 7558-02-3

(2) Acetamide; C_2H_5NO; 60-35-5

ORIGINAL MEASUREMENTS:

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

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of potassium bromide at various temperatures

t/$^\circ$C	g/100 g of solvent
81	10.3
90	10.5
100	10.8
110	11.0
120	11.2
130	11.3

The solubility data may be represented as a function of temperature by the following equation

$$\log c \text{ (g/100g)} = 9.40 \times 10^{-4}t + 0.937$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Purified acetamide containing an excess of potassium bromide 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 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^3 flask. The solution was then weighed, dissolved in water, and titrated using the Volhard method.

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 8.5×10^{-4} Sm^{-1} at 94°C.

ESTIMATED ERROR:

Temperature $\pm$ 0.1°C (author)

Solubility $\pm$ 1% (compiler)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Potassium iodide; KI; 7681-11-0 (2) Acetamide; C_2H_5NO; 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

COMPONENTS:	EVALUATOR:
(1) Potassium iodide; KI; 7681-11-0 (2) Acetamide; C_2H_5NO; 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.

COMPONENTS:

(1) Potassium iodide; KI: 7681-11-0

(2) Acetamide; C_2H_5NO; 60-35-5

ORIGINAL MEASUREMENTS:

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

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of potassium iodide at various temperatures

$t/^{\circ}C$	g/100 g of solvent
80	32.3
85	32.5
97	32.8
118	33.2
130	33.6

The solubility data may be represented as a function of temperature by the following equation

$$\log c\ (g/100g) = 3.33 \times 10^{-4} t + 1.484$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Purified acetamide containing an excess of potassium iodide 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 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^3 flask. The solution was then weighed, dissolved in water, and titrated using the Volhard method.

SOURCE AND PURITY OF MATERIALS:

Analytical grade potassium iodide 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 $8.5 \times 10^{-4}\ Sm^{-1}$ at 94°C.

ESTIMATED ERROR:

Temperature ± 0.1°C (author)

Solubility ± 1% (compiler)

REFERENCES:

COMPONENTS:

(1) Potassium iodide; KI; 7681-11-0

(2) Acetamide; C_2H_5NO; 60-35-5

ORIGINAL MEASUREMENTS:

Sarkisov, A.G.; Melamud, S.F.; Sakharova, N.K. *Khimiya,* 1969, 3-11.

VARIABLES:

Temperature

PREPARED BY:

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:

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 residual was weighed and the concentration determined.

SOURCE AND PURITY OF MATERIALS:

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 acetamide 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 ± 5% (compiler)

REFERENCES:

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

COMPONENTS:

(1) Lithium chloride; LiCl; 7447-41-8

(2) N-methylacetamide; C_3H_7NO; 79-16-3

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy.

March 1979.

CRITICAL EVALUATION:

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 LiCl 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

1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C.
J. Phys. Chem., 1963, *67*, 281-283.

COMPONENTS:

(1) Lithium chloride; LiCl; 7447-41-8

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 lithium chloride at 40°C.

$0.413 \text{ mol dm}^{-3}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined by preparing solutions containing 25 cm^3 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:

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

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lithium chloride; LiCl; 7447-41-8 (2) N-methylacetamide; C_3H_7NO; 79-16-3	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. *J. Phys. Chem.*, 1963, *67*, 281-283

METHOD/APPARATUS/PROCEDURE: (Continuation)

where $\underline{c}$ is the concentration of the solution, L the experimentally measured specific conductance and $\underline{a}$ and $\underline{b}$ 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:

(1) Sodium chloride; $NaCl$; 7647-14-5

(2) N-methylacetamide; C_3H_7NO; 79-16-3

EVALUATOR:

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:

(1) Sodium chloride; NaCl; 7647-14-5

(2) N-methylacetamide; C_3H_7NO; 79-16-3

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica 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

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. *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.
5. Gopal, R.; Bhatnagar, O.N. *J. Phys. Chem.*, 1965, *69*, 2382-2385

COMPONENTS:

(1) Sodium chloride; NaCl; 7647-14-5

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.

J. Indian Chem. Soc., 1968, *45(4)*, 351-353

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of sodium chloride at five temperatures

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

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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 suitable quantity of N-methylacetamide contained in a flask. The flask was shaken continuously and then placed in a thermostat where the temperature was controlled within ± 0.05 °C in the lower range (35-40°C) and within ± 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 solution determined gravimetrically.

SOURCE AND PURITY OF MATERIALS:

Sodium chloride was purified by repeated 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)

REFERENCES:

1. Gopal, R.; Hussain, M.M. *J. Indian Chem. Soc.*, 1963, *40(4)*, 272-274.

COMPONENTS:

(1) Sodium chloride; NaCl; 7647-14-5

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 sodium chloride at 40°C

0.339 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined conductometrically.

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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade NaCl 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:

(1) Sodium bromide; NaBr; 7647-15-6

(2) N-methylacetamide; C_3H_7NO; 79-16-3

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy.

March 1979.

CRITICAL EVALUATION:

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 ± 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

T/K	g/kg of Solvent
305	190.8
308	191.6
313	192.3
318	193.1
323	194.2

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.

COMPONENTS:

(1) Sodium bromide; NaBr; 7647-15-6

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.

J. Indian Chem. Soc., 1968, *45(4)*, 351-353.

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of sodium bromide at five temperatures.

t/°C	g/100 g of solvent
32	19.08
35	19.16
40	19.23
45	19.31
50	19.42

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined gravimetrically

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.

SOURCE AND PURITY OF MATERIALS:

Sodium bromide 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)°C (authors)

REFERENCES:

COMPONENTS:

(1) Sodium bromide; NaBr; 7647-15-6

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 sodium bromide at 40°C

1.85 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An 'approximate'(authors) solubility value was determined by the use of a large test tube, fitted with a stopper and covered with aluminium foil, into which was placed 10 cm^3 of solvent. The solute was added in small increments with constant shaking.

When the solution appeared to be saturated, the test tube was heated to 60°C in a water bath with frequent shaking. It was assumed that the solution had indeed reached saturation if solute precipitated upon cooling to 40°C.

The approximate solubility value, which the authors consider to have a precision of 5%, was determined on the basis of the salt added.

SOURCE AND PURITY OF MATERIALS:

Reagent grade sodium bromide was dried in a vacuum desiccator over anhydrous magnesium perchlorate and used without further treatment. As described in another paper from the same laboratory (1), N-methylacetamide was prepared by a reaction of monomethylamine with glacial acetic acid and subsequent heating to crack out and distil off water.

The product was purified by fractional distillation followed by five or more

(Continued)

ESTIMATED ERROR:

Solubility ± 5% (authors)

REFERENCES:

1. Dawson, L.R.; Sears, P.G.; Groves, R.H. *J. Am. Chem. Soc.*, 1955, *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_3H_7NO; 79-16-3	Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. *J. Phys. Chem.*, 1963, *67*, 281-283

SOURCE AND PURITY OF MATERIALS: (Continuation)

fractional freezing cycles, using a procedure described in detail by Berger and Dawson (2).

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

COMPONENTS:	EVALUATOR:
(1) Sodium iodide; NaI; 7681-82-5 (2) N-methylacetamide; C_3H_7NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION:

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 ± 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

T/K	g/kg of solvent
305	514.8
308	533.0
313	562.7
318	592.3
323	623.2

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.

COMPONENTS:

(1) Sodium Iodide; NaI; 7681-82-5

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D; Gopal, R.

J. Indian Chem. Soc., 1968, *45(4)*, 351-353.

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of sodium iodide at five temperatures

t/°C	g/100 g of solvent
32	51.48
35	53.30
40	56.27
45	59.23*
50	62.32

* In the original paper this value is reported as 29.230 g/100. It was however assumed by the compiler in the logic of the sequence that the number 2 was a printing mistake.

AUXILIARY INFORMATION

METHOD/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 details.

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%

Temperature ± (0.05-0.1)°C (authors).

REFERENCES:

COMPONENTS: (1) Sodium iodide; NaI; 7681-82-5 (2) N-methylacetamide; C_3H_7NO; 79-16-3	ORIGINAL MEASUREMENTS: Dawson, R.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 sodium iodide at 40°C

2.86 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An 'approximate' (authors) value of solubility 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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade NaI 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 ± 5% (authors)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium thiocyanate; NaSCN; 540-72-7 (2) N-methylacetamide; C_3H_7NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION:

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 ± 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

1. Dawson, R.L.: Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C.
J. Phys. Chem., 1963, *67*, 281-283.

COMPONENTS:

(1) Sodium thiocyanate; NaSCN; 540-72-7

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 sodium thiocyanate at 40°C

3.64 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An 'approximate' (authors) value of solubility 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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade NaSCN 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 ± 5% (authors)

REFERENCES:

COMPONENTS:

(1) Potassium chloride; KCl; 7447-40-7

(2) N-methylacetamide; C_3H_7NO; 79-16-3

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy.

March 1979

CRITICAL EVALUATION:

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 KCl 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 KCl 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

T/K	g/kg of solvent
305	8.65
308	8.99
313	9.58
318	10.07
323	10.71

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.

COMPONENTS:

(1) Potassium chloride; KCl; 7447-40-7

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.

J. Indian Chem. Soc., 1968, *45(4)*, 351-353

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of potassium chloride at five temperatures.

t/°C	g/100 g of solvent
32	0.865
35	0.899
40	0.958
45	1.007
50	1.071

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined gravimetrically.

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.

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 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 ± (0.05-0.1)°C (authors).

REFERENCES:

COMPONENTS:

(1) Potassium chloride; KCl; 7447-40-7

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 chloride at 40°C

0.120 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined conductimetrically.

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:

(1) Potassium bromide; KBr; 7558-02-3

(2) N-methylacetamide; C_3H_7NO; 79-16-3

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy.

March 1979

CRITICAL EVALUATION:

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-methylacetamide 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

T/K	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_3H_7NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION: (Continuation)

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. Gopal, R.; Bhatnagar, O.N. *J. Phys. Chem.*, 1965 *69*, 2382-2385.

COMPONENTS:

(1) Potassium bromide; KBr; 7558-02-3

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R

J. Indian Chem. Soc., 1968, *45(4)* , 351-353

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of potassium bromide at five temperatures

t/°C	g/100 g of solvent
32	4.773
35	4.862
40	5.003
45	5.142
50	5.278

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined gravimetrically.

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.

SOURCE AND PURITY OF MATERIALS:

Potassium bromide 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% (authors)

Temperature ± (0.05-0.1) °C (authors)

REFERENCES:

COMPONENTS:

(1) Potassium bromide; KBr; 7558-02-3

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 bromide at 40°C

0.429 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined conductometrically.

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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade KBr 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:

(1) Potassium iodide; KI; 7681-11-0

(2) N-methylacetamide; C_3H_7NO; 79-16-3

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy.

March 1979

CRITICAL EVALUATION;

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

T/K	g/kg of solvent
305	304.3
308	306.8
313	316.6
318	316.9
323	322.8

(Continued)

COMPONENTS:	EVALUATOR:
(1) Potassium iodide; KI; 7681-11-0 (2) N-methylacetamide; C_3H_7NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION: (Continuation)

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. Gopal, R.; Bhatnagar, O.N. *J. Phys. Chem.*, 1965, *69*, 2382-2385.

COMPONENTS:

(1) Potassium iodide; KI; 7681-11-0

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.

J. Indian Chem. Soc., 1968, *45(4)*, 351-353

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of KI at five temperatures

t/°C	g/100 g of solvent
32	30.43
35	30.68
40	31.16
45	31.69
50	32.28

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined gravimetrically.

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.

SOURCE AND PURITY OF MATERIALS:

Potassium 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% (authors)

Temperature ± (0.05-0.1)°C (authors)

REFERENCES:

COMPONENTS:

(1) Potassium iodide; KI; 7681-11-0

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 iodide at 40°C

$1.356 \text{ mol dm}^{-3}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined conductometrically.

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.

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_3H_7NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION:

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

1. 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_3H_7NO; 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 conductometrically.

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; NH_4Cl; 12125-02-9 (2) N-methylacetamide; C_3H_7NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION:

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_4Cl 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 NH_4Cl 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 NH_4Cl in N-methylacetamide of 313 K.

Tentative value of solubility of NH_4Cl at 313 K

47.93 g dm^{-3} of saturated solution

Solubility of NH_4Cl as a function of temperature

For temperatures other than 313 K, only the data of Chandra and Gopal (2) are available and these are

(Continued)

COMPONENTS:	EVALUATOR:
(1) Ammonium chloride; NH_4Cl; 12125-02-9 (2) N-methylacetamide; C_3H_7NO; 79-16-3	Bruno Scrosati. Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION: (continuation)

T/K	g/kg of solvent
305	48.83
308	49.67
313	50.93
318	52.21
323	53.53

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. Gopal, R.; Bhatnagar, O.N. *J. Phys. Chem.*, 1965, *69*, 2382-2385.

COMPONENTS:

(1) Ammonium chloride; NH_4Cl; 12125-02-9

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.

J. Indian Chem. Soc., 1968, *45(4)*, 351-353.

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of NH_4Cl at five temperatures

t/°C	g/100 g of solvent
32	4.883
35	4.967
40	5.093
45	5.221
50	5.353

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined gravimetrically.

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.

SOURCE AND PURITY OF MATERIALS:

Ammonium chloride 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% (authors)

Temperature ± (0.05-0.1)°C (authors.

REFERENCES:

COMPONENTS:

(1) Ammonium chloride; NH_4Cl;
12125-02-9

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 ammonium chloride at 40°C

$0.896 \text{ mol dm}^{-3}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined conductometrically.

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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade NH_4Cl 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) Ammonium bromide; NH_4Br; 12124-97-9 (2) N-methylacetamide; C_3H_7NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION:

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 NH_4Br 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 NH_4Br at 313 K

170.92 g dm^{-3} of saturated solution

Solubility of NH_4Br as a function of temperature

For temperatures different than 313 K, only the data of Chandra and Gopal (2) are available and these are

(Continued)

COMPONENTS:

(1) Ammonium bromide; NH_4Br; 12124-97-9

(2) N-methylacetamide; C_3H_7NO; 79-16-3

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy.

March 1979

CRITICAL EVALUATION: (Continuation)

T/K	g/kg of solvent
305	218.6
308	219.6
313	221.1
318	222.6
323	224.3

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. Gopal, R.; Bhatnagar, O.N. *J. Phys. Chem.*, 1965, *69*, 2382-2385.

COMPONENTS:

(1) Ammonium bromide; NH_4Br; 12124-97-9

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.

J. Indian Chem. Soc., 1968, *45(4)*, 351-353

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of NH_4Br at five temperatures

t/°C	g/100 g of solvent
32	21.86
35	21.96
40	22.11
45	22.26
50	22.43

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined gravimetrically.

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.

SOURCE AND PURITY OF MATERIALS:

Ammonium bromide 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% (authors)

Temperature ± (0.05-0.1)°C (authors)

REFERENCES:

COMPONENTS:

(1) Ammonium bromide; NH_4Br; 12124-97-9

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 ammonium bromide at 40°C

$1.745 \text{ mol dm}^{-3}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined conductometrically.

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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade NH_4Br 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:

(1) Ammonium iodide; NH_4I; 12027-06-4

(2) N-methylacetamide; C_3H_7NO; 79-16-3

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy.

March 1979

CRITICAL EVALUATION:

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 NH_4I 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

1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C.
J. Phys. Chem., 1963, *67*, 281-283.

COMPONENTS: (1) Ammonium iodide; NH_4I; 12027-06-4 (2) N-methylacetamide; C_3H_7NO; 79-16-3	ORIGINAL MEASUREMENTS: Dawson, R.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

2.47 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined conductometrically.

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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade NH_4I 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 thiocyanate; NH_4SCN; 1762-95-4 (2) N-methylacetamide; C_3H_7NO; 79-16-3	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. March 1979

CRITICAL EVALUATION:

Very few solubility data of pseudohalides in amides are available from the literature. In particular, the ammonium thiocyanate-N-methylacetamide system has been investigated only by Dawson, Berger, Vaughn and Eckstrom (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_4SCN 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.

References.

1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. *J. Phys. Chem.*, 1963, *67*, 281-283.

COMPONENTS:

(1) Ammonium thiocyanate; NH_4SCN; 1762-95-4

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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

4.626 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined conductometrically.

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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade NH_4SCN 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:

(1) Calcium chloride; $CaCl_2$; 10043-52-4

(2) N-methylacetamide; C_3H_7NO; 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 $CaCl_2$ 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 ± 5% to their datum.

Therefore only an approximate value of the solubility of calcium chloride in N-methylacetamide may be reported.

Approximate value of solubility of $CaCl_2$ 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

1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C. *J. Phys. Chem.*, 1963, *67*, 281-283.

COMPONENTS:

(1) Calcium chloride; $CaCl_2$; 10043-52-4

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 calcium chloride at 40°C

0.27 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An 'approximate' (authors) value of solubility 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.

SOURCE AND PURITY OF MATERIALS:

Reagent and grade $CaCl_2$ 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 described by Dawson, Berger, Vaughn and Eckstrom to which we refer the reader for details.

ESTIMATED ERROR:

Solubility ± 5% (authors)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Calcium bromide; $CaBr_2$; 7789-41-5 (2) N-methylacetamide; C_3H_7NO; 79-16-3	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 $CaBr_2$ 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 ± 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 $CaBr_2$ 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

1. Dawson, R.L.; Berger, J.E.; Vaughn, J.W.; Eckstrom, H.C.
J. Phys. Chem., 1963, *67*, 281-283.

COMPONENTS:

(1) Calcium bromide; $CaBr_2$; 7789-41-5

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 calcium bromide at 40°C

0.40 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An 'approximate' (authors) value of solubility 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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade $CaBr_2$ was dried in a vacuum desiccator over anyhdrous 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 ± 5% (authors)

REFERENCES:

COMPONENTS:

(1) Strontium chloride; $SrCl_2$; 10476-85-4

(2) N-methylacetamide; C_3H_7NO; 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 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 $SrCl_2$ 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.

COMPONENTS:

(1) Strontium chloride; $SrCl_2$; 10476-85-4

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.
J. Indian Chem. Soc., 1968, *45(4)*, 351-353

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of strontium chloride at five temperatures

t/°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:

The solubility was determined gravimetrically.

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.

SOURCE AND PURITY OF MATERIALS:

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)°C (authors)

REFERENCES:

COMPONENTS:

(1) Strontium bromide; $SrBr_2$; 10476-81-0

(2) N-methylacetamide; C_3H_7NO; 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 ± 5% to their approximate data.

Chandra and Gopal (2) have determined gravimetrically the solubility of $SrBr_2$ in N-methylacetamide 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 $SrBr_2$ at 313K

430.0 g/kg of solvent.

Solubility of $SrBr_2$ as a function of temperature.

For temperatures different than 313K, only the data of Chandra and Gopal (2) are available and these are

T/K	g/kg of solvent
305	415.0
308	421.2
313	430.4
318	439.5
323	449.0

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.

COMPONENTS:

(1) Strontium bromide; $SrBr_2$; 10476-81-0

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.
J. Indian Chem. Soc., 1968, *45(4)*, 351-353

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of strontium bromide at five temperatures

t/°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 gravimetrically.

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.

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 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 ± (0.05-0.1)°C (authors)

REFERENCES:

COMPONENTS:

(1) Strontium bromide; $SrBr_2$; 10476-81-0

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Dawson, R.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 strontium bromide at 40°C

1.64 mol dm^{-3}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An 'approximate' (authors) value of solubility 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.

SOURCE AND PURITY OF MATERIALS:

Reagent grade $SrBr_2$ 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 ± 5% (authors)

REFERENCES:

COMPONENTS:

(1) Barium chloride; $BaCl_2$; 10361-37-2

(2) N-methylacetamide; C_3H_7NO; 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 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 $BaCl_2$ 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

COMPONENTS:

(1) Barium chloride; $BaCl_2$; 10361-37-2

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.
J.Indian Chem. Soc., 1968, *45(4)*, 351-353.

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of barium chloride at five temperatures

t/°C	g/100 g of solvent
32	12.47
35	12.73
40	13.23
45	13.73
50	14.17

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined gravimetrically.

The method and the procedure have been described 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.

SOURCE AND PURITY OF MATERIALS:

Barium chloride 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 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)°C (authors)

REFERENCES:

COMPONENTS:

(1) Barium bromide; $BaBr_2$ 10553-31-8

(2) N-methylacetamide; C_3H_7NO; 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 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 $BaBr_2$ 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.

COMPONENTS:

(1) Barium bromide; $BaBr_2$; 10553-31-8

(2) N-methylacetamide; C_3H_7NO; 79-16-3

ORIGINAL MEASUREMENTS:

Chandra, D.; Gopal, R.
J. Indian Chem. Soc., 1968, *45(4)*, 351-353.

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of barium bromide at five temperatures

t/°C	g/100 g of solvent
32	38.50
35	38.81
40	39.43
45	40.06
50	40.67

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined gravimetrically.

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.

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 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 ± (0.05-0.1)°C (authors)

REFERENCES:

COMPONENTS:

(1) Lithium chloride; LiCl; 7447-41-8

(2) N, N-dimethylacetamide; C_4H_9NO; 127-19-5

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy.

May 1979.

CRITICAL EVALUATION:

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-dimethylacetamide-water 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.

T/K	mol/kg
293	2.03
313	2.52
333	2.92

The solubility data may be represented as function of temperature by the equation (evaluator)

$$\log c\ (\text{mol/kg}) = -386.522/(T/K) + 1.629$$

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)

COMPONENTS:

(1) Lithium chloride: LiCl; 7447-41-8

(2) N, N-dimethylacetamide; C_4H_9NO; 127-19-5

EVALUATOR:

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

T/K	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.*, 1971, *44(10)*, 2361-2362.

COMPONENTS:

(1) Lithium chloride; LiCl; 7447-41-8

(2) N, N-dimethylacetamide; C_4H_9NO; 127-19-5

ORIGINAL MEASUREMENTS:

Lesteva, T.M.: Chernaya, V.I.
Russ.J.Inorg.Chem., 1978, *23(9)*, 1416-1417.

Translated from
Z.Neorg.Khim., 1978, *23*, 2556-2558.

VARIABLES:

Temperature

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of LiCl at three temperatures.

$t/^{o}C$	mol/kg of solvent
20.0	2.03
40.0	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.

Specimen of the liquid phase were analysed for lithium chloride argentometrically.

The composition of the solid phase was determined from the Schreinemakers rays (1).

SOURCE AND PURITY OF MATERIALS:

Not reported.

ESTIMATED ERROR:

Solubility ± 0.2-0.5%

Temperature ± 0.05 K (authors)

REFERENCES:

(1) Schreinemakers, F.A.H., *J.Phys.Chem.* 1906, *55*, 73-75

COMPONENTS:

(1) Lithium chloride; LiCl; 7447-41-8

(2) N, N-dimethylacetamide; C_4H_9NO;
127-19-5

ORIGINAL MEASUREMENTS:

Pistoia, G.; Scrosati, B.
Ric.Sci., 1967, *37*, 1173-1177

VARIABLES:

One temperature: 25°C

PREPARED BY:

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 cm^3 of solvent contained in a 50 cm^3 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 conductance of 2.10^{-5} S m^{-1}, in good agreement with the value reported by Lester, Gover and Sears (2), i.e. (0.8-2), 10^{-5} S m^{-1}.

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.*, 1956, *60* 1076-1079.

COMPONENTS:	EVALUATOR:
(1) Lithium bromide; LiBr; 7550-35-8 (2) N, N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

CRITICAL EVALUATION:

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 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.
2. Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Zh.Prikl.Khim.* 1971, *44(10)*, 2361-2362.

COMPONENTS:

(1) Lithium bromide; LiBr; 7550-35-8

(2) N, N-dimethylacetamide; C_4H_9NO; 127-19-5

ORIGINAL MEASUREMENTS:

Pistoia G.; Scrosati, B.
Ric.Sci., 1967, *37* 1173-1177.

VARIABLES:

One temperature: 25°C

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of lithium bromide at 25°C

26.2 g/100 g of solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method and the apparatus for obtaining the saturated solution and determining the solubility was described in the course of th compilation of the lithium chloride-N, N-dimethylacetamide system, originally measured by Pistoia and Scrosati, to which we refer the reader for details.

SOURCE AND PURITY OF MATERIALS:

Lithium bromide, reagent grade commercial product, was dried under vacuum.

N,N-dimethylacetamide was purified by fractional distillation under reduced pressure (2mm Hg).

ESTIMATED ERROR:

Solubility ± 0.4%

Temperature ± 0.05 °C (authors).

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Lithium iodide; LiI; 10377-51-2 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

CRITICAL EVALUATION:

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

1. Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Zh.Prikl.Khim.*, 1971, *44 (10)*, 2361-2362

COMPONENTS:

(1) Lithium iodide; LiI; 10377-51-2

(2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5

ORIGINAL MEASUREMENTS:

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; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

CRITICAL EVALUATION:

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.
2. Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Zh.Prikl Khim.*, 1971, *44(10)*, 2361-2362.

COMPONENTS:

(1) Sodium chloride; NaCl; 7647-14-5

(2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5

ORIGINAL MEASUREMENTS:

Pistoia, G.; Scrosati, B.
Ric.Sci., 1967, *37*, 1173-1177.

VARIABLES:

One temperature: 25°C

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of sodium chloride at 25°C

0.02 g/100 g of solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method and the apparatus for obtaining the saturated solution and determining 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.

SOURCE AND PURITY OF MATERIALS:

Sodium chloride, reagent grade commercial product, was dried under vacuum.

N,N-dimethylacetamide was purified by fractional distillation under reduced pressure (2 mm Hg).

ESTIMATED ERROR:

Solubility ± 0.4%

Temperature ± 0.05°C (authors)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium bromide; NaBr; 7647-15-6 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

CRITICAL EVALUATION:

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:

(1) Sodium bromide; NaBr; 7647-15-6

(2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5

ORIGINAL MEASUREMENTS:

Pistoia, G.; Scrosati, B.,
Ric.Sci., 1967, *37*, 1173-1177

VARIABLES:

One temperature: 25°C

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of sodium bromide at 25°C.

6.51 g/100 g of solvent.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method and the procedure for obtaining the saturated solution and determining the solubility was described in the course of the compilation of the lithium chloride-N, N-dimetylacetamide system, originally measured by Pistoia and Scrosati, to which we refer the reader for details.

SOURCE AND PURITY OF MATERIALS:

Sodium bromide, 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:

(1) Sodium iodide; NaI; 7681-82-5

(2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5

EVALUATOR:

Bruno Scrosati,
Istituto di Chimica Fisica,
University of Rome, Italy.

May 1979

CRITICAL EVALUATION:

The sodium iodide-N,N-dimethylacetamide system has been investigated by Pistoia and Scrosati (1) and by Fedorov et.al. (2).

The result of the latter authors (0.40 mol dm^{-3} at 295-298 K) is rejected since no purification of the solvent was described and no control of the temperature was attempted.

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.
2. 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_4H_9NO; 127-19-5	Pistoia, G.; Scrosati, B. *Ric.Sci.*, 1967, *37*, 1173-1177.
VARIABLES:	**PREPARED BY:**
One temperature: 25°C	Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of sodium iodide at 25°C.

34.6 g/100 g of solvent.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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

SOURCE AND PURITY OF MATERIALS:

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%.

Temperature ± 0.05°C (authors).

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Sodium thiocyanate; NaSCN; 540-72-7 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

CRITICAL EVALUATION:

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

1. 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) Sodium thiocyanate; NaSCN; 540-72-7 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	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 sodium thiocyanate at 22-25 °C

1.78 mol dm^{-3}

AUXILIARY 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:

COMPONENTS:	EVALUATOR:
(1) Potassium chloride; KCl; 7447-40-7 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

CRITICAL EVALUATION:

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 KCl at 298 K

0.11 g/kg of solvent

References

1. Pistoia, G.; Scrosati, B.; *Ric.Sci.*, 1967, *37*, 1173-1177
2. Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Z.Prikl.Khim.*, 1971, *44(10)*, 2361-2362.

COMPONENTS: (1) Potassium chloride; KCl; 7447-40-7 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	ORIGINAL MEASUREMENTS: Pistoia, G; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1173-1177
VARIABLES: One temperature: 25°C	PREPARED BY: Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of potassium chloride at 25°C

0.01 g/100 g of solvent

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method and the procedure for obtaining the saturated solution and determining the solubility 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.

SOURCE AND PURITY OF MATERIALS:

Potassium chloride, 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) Potassium bromide; KBr; 7558-02-3 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

CRITICAL EVALUATION:

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.
2. Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Z. Prikl. Khim.*, 1971, *44(10)*, 2361-2362.

COMPONENTS: (1) Potassium bromide; KBr; 7558-02-3 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	ORIGINAL MEASUREMENTS: Pistoia, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1173-1177.
VARIABLES: One temperature; 25°C	PREPARED BY: Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of potassium bromide at 25°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 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.

SOURCE AND PURITY OF MATERIALS:

Potassium bromide, 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) Potassium iodide; KI; 7681-11-0 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

CRITICAL EVALUATION:

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.
2. 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 iodide; KI; 7681-11-0 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Pistoia, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1173-1177.
VARIABLES: One temperature: 25°C	PREPARED BY: Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of potassium iodide at 25°C

1.54 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 KI in the saturated solution was determined by Volhard titration.

SOURCE AND PURITY OF MATERIALS:

Potassium iodide, reagant 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) Potassium thiocyanate; KSCN; 333-20-0 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

CRITICAL EVALUATION:

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

1. Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shalionskaya, M.A.; Filatova, N.I. *Z. Prikl. Khim.*, 1971, *44(10)*, 2361-2362.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Potassium thiocyanate; KSCN; 333-20-0 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Z. Prikl. Khim.*, 1971, *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}

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 thiocyanate ions with standard mercuric solution.

SOURCE AND PURITY OF MATERIALS:

Potassium thiocyanate was dried under vacuum. No details on the purification of N,N-dimethylacetamide are reported.

ESTIMATED ERROR:

Solubility ±10% (compiler)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Magnesium chloride; $MgCl_2$; 7786-30-3 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

CRITICAL EVALUATION:

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 $MgCl_2$ at 298 K

35.4 g/kg of solvent.

References

1. Pistoia, G.; Scrosati, B.; *Ric. Sci.*, 1967, *37*, 1173-1177.
2. 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 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Pistoia, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1173-1177
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:

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 $MgCl_2$ in the saturated solution was determined by Fajans titration, using dichlorofluorescein as indicator.

SOURCE AND PURITY OF MATERIALS:

Magnesium chloride, reagent grade commercial product, was dried under vacuum.

N,N-dimethylacetamide was purified by fractional distillation under reduced pressure (2mmHg).

ESTIMATED ERROR:

Solubility ± 0.4%

Temperature ± 0.05°C (authors)

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Magnesium iodide; MgI_2; 10377-58-9 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

CRITICAL EVALUATION:

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_2 at 298 K

570 g/kg of solvent.

References

1. Pistoia, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1173-1177

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Magnesium iodide; MgI_2; 10377-58-9 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Pistoia, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1173-1177
VARIABLES:	**PREPARED BY:**
One temperature: 25°C	Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of magnesium iodide at 25°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 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	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 $CaCl_2$ at 298 K

41.3 g/kg of solvent

References

1. Pistoia, G.; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1173-1177.
2. Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Z. Prikl. Khim.*, 1971, *44(10)*, 2361-2362.

COMPONENTS:

(1) Calcium chloride; $CaCl_2$; 10043-52-4

(2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5

ORIGINAL MEASUREMENTS:

Pistoia, G.; Scrosati, B.
Ric. Sci., 1967, *37*, 1173-1177.

VARIABLES:

One temperature: 25°C

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of calcium chloride at 25°C

4.13 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 $CaCl_2$ in the saturated solution was determined by Fajans titration, using dichlorofluorescein as indicator.

SOURCE AND PURITY OF MATERIALS:

Calcium chloride, 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 bromide; $CaBr_2$; 7789-41-5 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	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 $CaBr_2$ at 298 K

99.5 g/kg of solvent.

References

1. Pistoia, G; Scrosati, B. *Ric. Sci.*, 1967, *37*, 1173-1177.
2. Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Z. Prikl. Khim.*, 1971, *44(10)*, 2361-2362.

COMPONENTS:

(1) Calcium bromide; $CaBr_2$; 7789-41-5

(2) N,N-dimethylacetamide; C_4H_9NO;
127-19-5

ORIGINAL MEASUREMENTS:

Pistoia, G.; Scrosati, B.
Ric. Sci., 1967, *37*, 1173-1177.

VARIABLES:

One temperature: 25°C

PREPARED BY:

Bruno Scrosati

EXPERIMENTAL VALUES:

Solubility of calcium bromide at 25°C

9.95 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 $CaBr_2$ in the saturated solution was determined by Fajans titration, using tetrabromofluorescein as indicator.

SOURCE AND PURITY OF MATERIALS:

Calcium bromide, 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 iodide; CaI_2; 10102-68-8 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979.

CRITICAL EVALUATION:

Very few solubility data of halides in acetamides are available in the literature. In particular the system calcium iodide-N,N-dimethylacetamide has been examined only by Fedorov et.al. (1), who reported a value of 0.09 mol dm^{-3} at 295-398 K.

In other system, where more reliable data from other sources are available, the results of Fedorov et.al. 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 the data of Fedorov et.al. (1) are the only available, the result of these authors have been compiled and reported as very approximate values of solubility.

Approximate value of solubility of CaI_2 at 298 K

26 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

1. Fedorov, A.A.; Sokolov, L.B.; Savinov, V.M.; Shilionskaya, M.A.; Filatova, N.I. Z. *Prikl. Khim.*, 1971, *44(10)*, 2361-2362.

COMPONENTS:

(1) Calcium iodide; CaI_2; 10102-68-8

(2) N,N-dimethylacetamide; C_4H_9NO;
127-19-5

ORIGINAL MEASUREMENTS:

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 calcium iodide at 22-25°C

0.09 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 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-dimethylacetamide are reported.

ESTIMATED ERROR:

Solubility ±10% compiler.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Strontium chloride; $SrCl_2$; 10476-85-4 (2) N,N-dimethylacetamide; C_4H_9NO; 127-19-5	Bruno Scrosati, Istituto di Chimica Fisica, University of Rome, Italy. May 1979

CRITICAL EVALUATION:

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 $SrCl_2$ 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

1. Fedorov, A.A.; Sokolov, L.B.; Savinvov, V.M.; Shilionskaya, M.A.; Filatova, N.I. *Z. Prikl. Khim.*, 1971, *44(10)*, 2361-2362.

COMPONENTS:

(1) Strontium chloride; $SrCl_2$;
10476-85-4

(2) N,N-dimethylacetamide; C_4H_9NO;
127-19-5

ORIGINAL MEASUREMENTS:

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 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-dimethylacetamide are reported.

ESTIMATED ERROR:

Solubility ±10% (compiler)

REFERENCES:

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.

REGISTRY NUMBER INDEX

Underlined page numbers refer to evaluation text and those not underlined to compiled tables.