INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

ANALYTICAL CHEMISTRY DIVISION
COMMISSION ON SOLUBILITY DATA

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## SOLUBILITY DATA SERIES

Volume 61

# ALKALI METAL AND AMMONIUM PERCHLORATES
# PART I: LITHIUM AND SODIUM PERCHLORATES

# SOLUBILITY DATA SERIES



INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY
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# ALKALI METAL AND AMMONIUM PERCHLORATES
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OXFORD UNIVERSITY PRESS

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The IUPAC Solubility Data Series (ISSN 0191-5622) is published quarterly in March, June, September and December by Oxford University Press, Oxford, UK. Annual subscription price is US$395. The IUPAC Solubility Data Series is distributed by Mercury Airfreight International Ltd., 10 Camptown Road, Irvington, New Jersey 07111-1105, USA. Second Class postage paid at Newark, New Jersey, USA and additional entry points.

US POSTMASTER: send address corrections to IUPAC Solubility Data Series, c/o Mercury Airfreight International Ltd., 10 Camptown Road, Irvington, New Jersey 07111-1105, USA.
This issue date is December 1995.

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Printed and bound in Great Britain by Antony Rowe Ltd, Chippenham, Wiltshire

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## OBITUARY: IVAN NIKONOVICH LEPESHKOV

Ivan Nikonovich Lepeshkov, Professor of Inorganic Chemistry in the Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, died on January 30, 1993 in Moscow. His sudden passing after a brief illness came as a great shock to his friends and colleagues.

Prof. Lepeshkov was born into a peasant family on January 15, 1907 in a small village near Smolensk. His early education began in a village school and continued into secondary school in Smolensk. In 1930 he was graduated from the University of Smolensk with a degree in chemistry. The following year he pursued post-graduate studies under the tutelage of Academician N.S. Kurnakov in the Laboratory of General Chemistry in Leningrad (St Petersburg). His thesis for a Candidate's Degree was on the crystallization of natural salts found in the Inder Lake (Kazakhstan) and was completed in 1935 and submitted to the faculty of the Institute of General and Inorganic Chemistry in Moscow (the Chemical Laboratories of the Academy of Sciences in Leningrad was moved to Moscow in 1934). Prof. Kurnakov became the director of this new institute and Prof. Lepeshkov was appointed Professor of Inorganic Chemistry at this same institute in 1943.

Prof. Lepeshkov spent the whole of his active life at the Institute of General and Inorganic Chemistry. He was Head of the Laboratory of Chemistry and Technology of Natural Salts for more than 40 years, and at the time of the Second World War was promoted to Vice-Director of the institute. At this time he also served as chief of the Division of Physico-Chemical Analysis. He participated in numerous scientific expeditions in the Central Asian Republics, Volga-Ural regions and Siberia. Prof. Lepeshkov and his colleagues discovered industrial deposits of bishoffite in the Volvograd Region, and he was active in the solution of the problems connected with Lake Kara-Bugas, and his solutions are presently accepted as the most practical.

Prof. Lepeshkov is best known for his extensive and numerous studies on water-salt equilibria which found important applications in geochemical and chemical engineering aspects of prospecting and processing of salt deposits. He contributed to chemical technology and production of fertilizers, antifreeze formulations and electronic materials. The results of these studies were published in more than 400 papers and several books. He exerted strong influence on the formation and development of inorganic chemistry in Kazakhstan, Kirgizia,

Turkmenistan and Kabardino-Balkaria where he helped organize the research groups and Laboratories on Natural Salt Chemistry. Prof Lepeshkov continually stressed the fundamental ideas of his teacher, Prof. Kurnakov, on the principles of physico-chemical analysis, and from amongst his more than fifty students, a number have become professors and members of the Academy of Sciences of the Central Asia Republics.

Prof. Lepeshkov's international activities began in the 1950s when he established contacts with German chemists in the former DDR. He visited China, Bulgaria, Spain and other countries. His most fruitfull cooperation was between his laboratory and the Chemical Division of the Freiberg Bergakademie in Germany and the Laboratory of Inorganic Salts of the Bulgarian Academy of Sciences in Sofia. During the last years of his life Prof. Lepeshkov participated in the IUPAC Solubility Data Project and became a close colleague of many members of IUPAC Commission V.8. At he invitation of Prof. A.S. Kertes (founder of the Solubility Data Project and Chairman of Commission V.8 until shortly before his untimely death in 1988), Prof. Lepeshkov became the first Russian chemist to participate in this international project. His first volume on Alkaline Earth Metal Perchlorates, co-edited with Profs C.-Y. Chan and K.H. Khoo from the University of Malaysia, was published as volume 41 in 1989, and this second volume on the solubilities of perchlorates is dedicated to his memory.

Prof. Lepeshkov will be missed by all who knew him. His work and science lives on and we are indeed richer for his contributions and for his friendship.

V.L. Valyashko and V. Danilov

## INTRODUCTION TO THE SOLUBILITY DATA SERIES

## SOLUBILITY OF SOLIDS IN LIQUIDS

### NATURE OF THE PROJECT

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

### COMPILATIONS AND EVALUATIONS

The formats for the compilations and critical evaluations have been standardized for all volumes. A description of these formats follows.

#### Compilations

The format used for the compilations is, for the most part, self-explanatory. Normally, a compilation sheet is divided into boxes, with detailed contents described below.

*Components*: Each component is listed according to IUPAC name, formula, and Chemical Abstracts (CA) Registry Number. The Chemical Abstracts name is also included if this differs from the IUPAC name, as are trivial names if appropriate. IUPAC and common names are cross-referenced to Chemical Abstracts names in the System Index.

The formula is given either in terms of the IUPAC or Hill (1) 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 on a given compilation sheet according to:

(a) saturating components:
(b) non-saturating components;
(c) solvents.

In each of (a), (b) or (c), the components are arranged in order according to the IUPAC 18-column periodic table with two additional rows:

| | |
|---|---|
| Columns 1 and 2: | H, alkali elements, ammonium, alkaline earth elements |
| Columns 3 to 12: | transition elements |
| Columns 13 to 17: | boron, carbon, nitrogen groups; chalcogenides, halogens |
| Column 18: | noble gases |
| Row 1: | Ce to Lu |
| Row 2: | Th to the end of the known elements, in order of atomic number. |

The same order is followed in arranging the compilation sheets within a given volume.

*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. In the case of multiple entries (for example, translations) an asterisk indicates the publication used for compilation of the data.

*Variables*: Ranges of temperature, pressure, etc. are indicated here.

*Prepared by*: The names of all compilers are given here.

*Experimental Values*: Components are described as (1), (2), etc., as defined in the "Components" box. 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. 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 1989 atomic weights (2). Temperatures are expressed as $t$/°C, $t$/°F or $T$/K as in the original; if necessary, conversions to $T$/K are made, sometimes in the compilations and always in the critical evaluation. However, the author's units are expressed according to IUPAC recommendations (3) as far as possible.

Errors in calculations, fitting equations, etc. are noted, and where possible corrected. Material inserted by the compiler is identified by the word "compiler" or by the compiler's name in parentheses or in a footnote. In addition, compiler-calculated values of mole or mass fractions are included if the original data do not use these units. If densities are reported in the original paper,

conversions from concentrations to mole fractions are included, but otherwise this is done in the evaluation, with the values and sources of the densities being quoted and referenced.

Details of smoothing equations (with limits) are included if they are present in the original publication and if the temperature or pressure ranges are wide enough to justify this procedure and if the compiler finds that the equations are consistent with the data.

The precision of the original data is preserved when derived quantities are calculated, if necessary by the inclusion of one additional significant figure. In some cases, compilers note that numerical data have been obtained from published graphs using digitizing techniques. In these cases, the precision of the data can be determined by the quality of the original graph and the limitations of the digitizing technique. In some cases graphs have been included, either to illustrate data more clearly, or if this is the only information in the original. Full grids are not usually inserted as it is not intended that users should read data from the graphs.

*Method*: The apparatus and procedure are mentioned briefly. Abbreviations used in Chemical Abstracts are often used here to save space, reference being made to sources of further detail if these are cited in the original paper.

*Source and Purity of Materials*: For each component, referred to as (1), (2), etc., the following information (in this order and in abbreviated form) is provided if available in the original paper: source and specified method of preparation; properties; degree of purity.

*Estimated Error*: If estimated errors were omitted by the original authors, and if relevant information is available, the compilers have attempted to estimate errors (identified by "compiler" or the compiler's name in parentheses or in a footnote) from the internal consistency of data and type of apparatus used. Methods used by the compilers for estimating and reporting errors are based on Ku and Eisenhart (4).

*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*: The format for these follows the format for the Original Measurements box, except that final page numbers are omitted. References (usually cited in the original paper) are given where relevant to interpretation of the compiled data, or where cross-reference can be made to other compilations.

**Evaluations**

The evaluator's task is 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. There are only three boxes on a typical evaluation sheet, and these are described below.

*Components*: The format is the same as on the Compilation sheets.

*Evaluator*: The name and affiliation of the evaluator(s) and date up to which the literature was checked.

*Critical Evaluation*:

(a) Critical text. The evaluator checks that the compiled data are correct, assesses their reliability and quality, estimates errors where necessary, and recommends numerical values based on all the published data (including theses, patents and reports) for each given system. Thus, the evaluator reviews the merits or shortcomings of the various data. Only published data are considered. Documented rejection of some published data may occur at this stage, and the corresponding compilations may be removed.

The solubility of comparatively few systems is known with sufficient accuracy to enable a set of recommended values to be presented. Although many systems have been studied by at least two workers, the range of temperatures is often sufficiently different to make meaningful comparison impossible.

Occasionally, it is not clear why two groups of workers obtained very different but internally consistent sets of results at the same temperature, although both sets of results were obtained by reliable methods. In such cases, a definitive assessment may not be possible. In some cases, two or more sets of data have been classified as tentative even though the sets are mutually inconsistent.

(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, stating the limits within which it should be used.

(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 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, including all those publications appearing in the accompanying compilation sheets and those which, by virtue of their poor precision, have been rejected and not compiled.

(f) Units. While the original data may be reported in the units used by the investigators, the final recommended values are reported in SI units (3) when the data can be converted accurately.

## QUANTITIES AND UNITS USED IN COMPILATION AND EVALUATION OF SOLUBILITY DATA

### Mixtures, Solutions and Solubilities

A *mixture* (5) describes a gaseous, liquid or solid phase containing more than one substance, where the substances are all treated in the same way.

A *solution* (5) 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 solute 1 (solid, liquid or gas) is the analytical composition of a saturated solution, expressed in terms of the proportion of the designated solute in a designated solvent (6).

"Saturated" implies equilibrium with respect to the processes of dissolution and precipitation; the equilibrium may be stable or metastable. The solubility of a substance in metastable equilibrium is usually greater than that of the same substance in stable equilibrium. (Strictly speaking, it is the activity of the substance in metastable equilibrium 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 reference states used for definition of activities, activity coefficients and osmotic coefficients.

Note that the composition of a 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 proportions of anhydrous salt in solution, rather then the relative proportions of hydrated salt and water.

### Physicochemical Quantities and Units

Solubilities of solids have been the subject of research for a long time, and have been expressed in a great many ways, as described below. In each case, specification of the temperature and either partial or total pressure of the saturating gaseous component is necessary. The nomenclature and units follow, where possible, ref. (3)

*A note on nomenclature.* The nomenclature of the IUPAC Green Book (3) calls the solute component B and the solvent component A. In compilations and evaluations, the first-named component (component 1) is the solute, and the second (component 2 for a two-component system) is the solvent. The reader should bear these distinctions in nomenclature in mind when comparing equations given here with those in the Green Book.

1. *Mole fraction* of substance 1, $x_1$ or $x(1)$for condensed phases, $y_1$ for gaseous phases:

$$x_1 = n_1 / \sum_{s=1}^{c} n_s \qquad [1]$$

where $n_s$ is the amount of substance of $s$, and $c$ is the number of distinct substances present (often

the number of thermodynamic components in the system). *Mole per cent* of substance 1 is 100 $x_1$.

2. *Ionic mole fractions* of salt i, $x_{i+}$, $x_{i-}$:
For a mixture of $s$ binary salts i, each of which ionizes completely into $\nu_{i+}$ cations and $\nu_{i-}$ anions, with $\nu_i = \nu_{+i} + \nu_{-i}$ and a mixture of $p$ non-electrolytes k, of which some may be considered as solvent components, a generalization of the definition in (7) gives:

$$x_{+i} = \frac{\nu_{+i}x_i}{1+\sum_{j=1}^{s}(\nu_j-1)x_j}, \quad x_{-i} = \frac{\nu_{-i}x_{+i}}{\nu_{+i}} \quad i = 1...s \qquad [2]$$

$$x_k' = \frac{x_k}{1+\sum_{j=1}^{s}(\nu_j-1)x_j}, \quad k = (s+1),...,(s+p) \qquad [3]$$

The sum of these mole fractions is unity, so that, with $c = s + p$,

$$\sum_{i=1}^{s}(x_{+i}+x_{-i})+\sum_{i=s+1}^{c}x_i' = 1 \qquad [4]$$

General conversions to other units in multicomponent systems are complicated. For a three-component system containing non-electrolyte 1, electrolyte 2 and solvent 3,

$$x_1 = \frac{\nu_{+2}x_1'}{\nu_{+2}-(\nu_2-1)x_{+2}} \qquad x_2 = \frac{x_{+2}}{\nu_{+2}-(\nu_2-1)x_{+2}} \qquad [5]$$

These relations are used in solubility equations for salts, and for tabulation of salt effects on solubilities of gases.

3. *Mass fraction* of substance 1, $w_1$ or $w(1)$:

$$w_1 = g_1/\sum_{s=1}^{c}g_s \qquad [6]$$

where $g_s$ is the mass of substance $s$. *Mass per cent* of substance 1 is 100 $w_1$. The equivalent terms *weight fraction*, *weight per cent* and *g (1)/100 g solution* are no longer used.

4. *Solute mole fraction* of substance 1, $x_{s,1}$:

$$x_{s,1} = m_1/\sum_{i=1}^{s}m_i = x_1/\sum_{i=1}^{s}x_i \qquad [7]$$

where $c$ is the number of solutes in the mixture. These quantities are sometimes called Jänecke mole (mass) fractions (8, 9). *Solute mass fraction* of substance 1, $w_{s,1}$, is defined analogously.

5. *Solvent mole fraction* of substance 1, $x_{v,1}$:

$$x_{v,1} = x_1/\sum_{i=s+1}^{c}x_i \qquad [8]$$

Here, $p$ is the number of solvent components in the mixture. *Solvent mass fraction* of substance 1, $w_{v,1}$, is defined analogously.

6. *Molality* of solute 1 in a solvent 2, $m_1$:

$$m_1 = n_1/n_2\ M_2 \qquad [9]$$

SI base units: mol $kg^{-1}$. Here, $M_2$ is the molar mass of the solvent.

7. *Aquamolality, Solvomolality* of substance 1 in a mixed solvent with components 2, 3 (10), $m_1^{(3)}$:

$$m_1^{(3)} = m_1\overline{M}/M_3 \qquad [10]$$

SI base units: mol $kg^{-1}$. Here, the average molar mass of the solvent is

$$\overline{M} = x_{v,2}M_2+(1-x_{v,2})M_3 \qquad [11]$$

and $x_{v,2}$ is the solvent mole fraction of component 2. This term is used most frequently in discussing comparative solubilities in water (component 2) and heavy water (component 3) and in their mixtures.

8. *Amount concentration* of solute 1 in a solution of volume $V$, $c_1$:

$$c_1 = [\text{formula of solute 1}] = n_1/V \qquad [12]$$

SI base units: mol $m^{-3}$. The symbol $c_1$ is preferred to [formula of solute 1], but both are used. The old terms *molarity*, *molar* and *moles per unit volume* are no longer used.

9. *Mass concentration* of solute 1 in a solution of volume $V$, $\rho_1$:

$$\rho_1 = g_1/V = c_1M_1/V \qquad [13]$$

SI base units: kg $m^{-3}$.

10. *Mole ratio*, $r_{1,2}$ (dimensionless) (11):

$$r_{1,2} = n_1/n_2 \qquad [14]$$

Mass ratio, symbol $\zeta_{1,2}$, may be defined analogously (11).

11. *Partial pressure*, $p_1 = y_1p$ for substance 1, where $y_1$ is the mole fraction of 1 in the vapor phase and $p$ is the total pressure. SI units: Pa; common units: mmHg, Torr. 1 mmHg and 1 Torr differ slightly in definition (3), but are identical numaerically to better than $2 \times 10^{-7}$ Torr.

12. *Ionic strength*, $I_m$ (molality basis), or $I_c$ (concentration basis):

$$I_m = \frac{1}{2}\sum_i m_iz_i^2, \quad I_c = \frac{1}{2}\sum_i c_iz_i^2 \qquad [15]$$

where $z_i$ is the charge number of ion i. While these quantities are not used generally to express solubilities, they are used to express the compositions of non-saturating components. For a single salt i with ions of charge numbers $z_+$, $z_-$,

$$I_m = |z_+z_-|\nu m_i, \quad I_c = |z_+z_-|\nu c_i \qquad [16]$$

Mole and mass fractions and mole ratios are appropriate to either the mixture or the solution point of view. The other quantities are appropriate to the solution point of view only. Conversions between pairs of these quantities can be carried out using the equations given in Table 1 at the end of this Introduction. Other useful quantities will be defined in the prefaces to individual volumes or on specific data sheets.

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 noted carefully the critical evaluation.

Mineralogical names are also quoted, along with their CA Registry Numbers, again usually in in the text, and CA Registry Numbers (where available) are given usually in the critical evaluation.

In addition to the quantities defined above, the following are useful in conversions between concentrations and other quantities.

13. *Density*, $\rho$:

$$\rho = g/V = \sum_{i=1}^{c} \rho_i \qquad [17]$$

SI base units: kg $m^{-3}$. Here $g$ is the total mass of the system.

14. *Relative density*, $d = \rho/\rho^o$: the ratio of the density of a mixture at temperature $t$, pressure $p$ to the density of a reference substance at temperature $t'$, pressure $p'$. For liquid solutions, the reference substance is often water at 4°C, 1 bar. (In some cases 1 atm is used instead of 1 bar.) The term *specific gravity* is no longer used.

*Thermodynamics of Solubility*

Thermodynamic analysis of solubility phenomena provides a rational basis for the construction of functions to represent solubility data, and thus aids in evaluation, and sometimes enables thermodynamic quantities to be extracted. Both these aims are often difficult to achieve because of a lack of experimental or theoretical activity coefficients. Where thermodynamic quantities can be found, they are not evaluated critically, since this task would involve examination of a large body of data that is not directly relevant to solubility. Where possible, procedures for evaluation are based on established thermodynamic methods. Specific procedures used in a particular volume will be described in the Preface to that volume.

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October, 1995

Table 1. Interconversions between Quantities Used as Measures of Solubility
*c*-component Systems Containing *c* - 1 Solutes *i* and Single Solvent *c*

| | $x_i$ | $w_i$ | $m_i$ | $c_i$ |
|---|---|---|---|---|
| $x_i =$ | $x_i$ | $\dfrac{1}{1+\dfrac{M_i}{M_c}\left(\dfrac{1}{w_i}-1+\sum_{j\neq i}^{c-1}\left(\dfrac{M_c}{M_j}-1\right)\dfrac{w_j}{w_i}\right)}$ | $\dfrac{1}{1+\dfrac{1}{m_iM_c}+\sum_{j\neq i}^{c-1}\dfrac{m_j}{m_i}}$ | $\dfrac{1}{1+\dfrac{1}{M_c}\left(\dfrac{\rho}{c_i}-M_i\right)+\sum_{j\neq i}^{c-1}\dfrac{c_j}{c_i}\left(1-\dfrac{M_j}{M_c}\right)}$ |
| $w_i =$ | $\dfrac{1}{1+\dfrac{M_c}{M_i}\left(\dfrac{1}{x_i}-1+\sum_{j\neq i}^{c-1}\left(\dfrac{M_j}{M_c}-1\right)\dfrac{x_j}{x_i}\right)}$ | $w_i$ | $\dfrac{1}{1+\dfrac{1}{m_iM_i}\left(1+\sum_{j\neq i}^{c-1}m_jM_j\right)}$ | $\dfrac{c_iM_i}{\rho}$ |
| $m_i =$ | $\dfrac{1}{M_c\left(\dfrac{1}{x_i}-1-\sum_{j\neq i}^{c-1}\dfrac{x_j}{x_i}\right)}$ | $\dfrac{1}{M_i\left(\dfrac{1}{w_i}-1-\sum_{j\neq i}^{c-1}\dfrac{w_j}{w_i}\right)}$ | $m_i$ | $\dfrac{1}{\dfrac{1}{c_i}\left(\rho-\sum_{j\neq i}^{c-1}c_jM_j\right)-M_i}$ |
| $c_i =$ | $\dfrac{\rho}{M_i+M_c\left(\dfrac{1}{x_i}-1+\sum_{j\neq i}^{c-1}\left(\dfrac{M_j}{M_c}-1\right)\dfrac{x_j}{x_i}\right)}$ | $\dfrac{\rho w_i}{M_i}$ | $\dfrac{\rho}{\dfrac{1}{m_i}\left(1+\sum_{j\neq i}^{c-1}M_jm_j\right)+M_i}$ | $c_i$ |

$\rho$ - density of solution; $M_i$ - molar masses of i. For relations for 2-component systems, set summations to 0.

# PREFACE

The compilation and evaluation work on solubility data for alkali metal and ammonium perchlorates in aqueous, non-aqueous and mixed solvents resulted in two volumes which have been prepared in accordance with the objectives and format guidelines of the IUPAC Solubility Data Series. The first of these volumes, Part I, covers solubility data for lithium and sodium perchlorates and Part II, the second volume, covers those for potassium, rubidium, cesium and ammonium perchlorates.

The first comprehensive review, and probably the only one of its kind available, on the manufacture, properties, uses and analytical chemistry of perchloric acid, its salts and derivative compounds appeared as an excellent monograph edited by Schumacher (6) in 1960. It also included a brief account of the history and manufacture of perchloric acid and its salts. Potassium perchlorate was the first salt of perchloric acid to be prepared. Its discovery, together with the preparation of perchloric acid, was reported by von Stadion (1) as early as 1816. During the period between 1816 and 1831 the term "oxychlorate" was used to describe salts of perchloric acid. Serullas (2), who reported in 1830 another method of perchloric acid preparation and also the preparation of ammonium perchlorate and most of the more common inorganic metal perchlorates, helped to popularise the use of the term "perchlorate", which has been the preferred term since then. The main industrial and patent interests in the alkali metal and ammonium perchlorates appear to lie in the use of both potassium perchlorate and ammonium perchlorate in the manufacture of explosives and solid rocket propellants, in the use of lithium perchlorate as battery electrolyte, and in the use of sodium perchlorate as starting material in the manufacture of ammonium perchlorate and other perchlorates. Rubidium and cesium perchlorate attract relatively little commercial interest.

The unusual stability of the perchlorate ion is well-known but so is its ability to act as a powerful oxidant which can react explosively with organic materials (6). The alkali metal perchlorates together with ammonium perchlorate form an interesting group in that the lithium and sodium salts are very soluble in water and are hydrated while the potassium, rubidium, cesium and ammonium salts are unhydrated and have low solubilities. Lithium and sodium perchlorates are also much more soluble in alcohols and other non-aqueous solventshan the potassium, rubidium, cesium and ammonium salts. All of them exist as colourless crystalline solids. Reliable solubility data for these perchlorates in water, alcohols

(continued)

and other solvents were first published in 1923 by Willard and Smith (4). After that, up until the 1960's, more interest appeared to be focused on the solubility systems of potassium perchlorate in both aqueous and non-aqueous solvents than those of other perchlorates. The period between 1960 and 1980 saw prolific publication of solubility data on ammonium, alkali metal and alkaline earth metal perchlorates in binary and multi-component systems in aqueous, non-aqueous and mixed solvents. The majority of such work was carried out in the Commonwealth of Independent States (formerly USSR) and found only in Russian publications. After 1980 much fewer reports of solubility data for these perchlorates were found.

The primary sources used in the literature survey for relevant information were *Chemical Abstracts* from 1907 to 1990 and the volumes on *Solubilities of Inorganic and Metal Organic Compounds* by Linke (5). Other sources include the monographs by Schumacher (6) and Schilt (8), the article by Carlson (3), and the comprehensive treatise by Mellor (7). While the compilers have made their best effort to compile on all relevant and available data published up to 1990, it is possible that certain pertinent articles published in obscure journals have missed their attention. No compilations have been prepared for data presented only in graphic forms and they involved only a few articles, published in Russian. It is practically impossible to communicate with the authors of the original measurements to obtain the numerical data.

This work is the result of many years of collaboration between the Russian and Malaysian scientists involved in the Solubility Data Project, which has already resulted in volume 41 in the Series (on alcaline earth metal perchlorates, published in 1990). It is worth noting that much of the compilations in the present two Volumes, just as in Volume 41, are on data published in Russian Journals which are not readily available outside the C.I.S. The editors thank the University of Malaya and the Kurnakov Institute of General and Inorganic Chemistry, Moscow, for providing facilities used in the preparation of these volumes. They also wish to acknowledge the much appreciated help and advice from Dr. M. Salomon (U.S.A.), Prof. J.W. Lorimer (Canada), the late Prof. A.S. Kertes (Israel) and Prof. Yagodin (Russia), given in various ways, including liaison and literature search. They also thank all those colleagues in IUPAC Commission V.8 who have given help in one way or another.

(continued)

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| COMPONENTS | EVALUATORS |
|---|---|
| (1) Lithium Perchlorate; $LiClO_4$; [7791-03-9] | S. Gryzlova; *Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Moscow, Russia |
| 2) Water ; $H_2O$ ; [7732-18-5] | Cohen-Adad; Université Claude Bernard Lyon I Villeurbanne, France |
| (3) Other Solvents | |

## CRITICAL EVALUATION

Solubility data for fifty-eight binary systems $LiClO_4$ - solvents are presented in eleven publications (5, 7, 12, 14, 15, 20, 25, 32, 39, 46, 57). There are also data for twenty-four ternary (5, 17, 21-26, 28-30, 36, 38, 42-45, 47, 48, 50, 51, 54, 55, 58) and seven quaternary systems (6, 22, 31, 34, 37, 50, 52).

## EXPERIMENTAL METHODS

The solubility of lithium perchlorate has been measured in most cases by the isothermal method. Simmons and Ropp (7) used a visual heating method in sealed tubes; a cooling method is also mentioned in ref. (46).

The solid phase was characterized by evaporation to dryness and, for ternary or quaternary systems, by the Schreinemakers' wet residues method (1). Thermographic and X-ray powder analysis were also employed (38).

## ANALYSIS OF SOLUTIONS

Lithium perchlorate in saturated solution was determined by evaporation to dryness or by chemical analysis : $Li^+$ was titrated gravimetrically by precipitation as sulfate (17) or lithium zinc uranyl acetate (22, 30, 31, 37) or in solution, using the periodate method (21, 30), atomic absorption spectrophotometry (25) or flame photometry (12).

Perchlorate ion was determined gravimetrically as nitron perchlorate (14, 20, 21, 23, 29, 43, 47, 54) or by conversion to chloride and titration as AgCl (14).

## SOURCE AND PURITY OF MATERIALS

In most cases lithium perchlorate was prepared by the methods described in references (2, 3, 4) and analysed as nitron perchlorate. It was also obtained by reacting 70 % $HClO_4$ (aq.) with $Li_2CO_3$, recrystallized twice (20). Keller and Foster used a commercial product

(continued)

CRITICAL EVALUATION (continued)

(Atomergic Chemicals Co) dried at 90-120 °C under vacuum. Lithium perchlorate was also dried at 200-250 °C under vacuum (20) or at 150-160 °C for 20-25 hours (15).

The solvents were "Reagent grade products" (14, 25) purified by fractional distillation after drying over anhydrous calcium sulfate (14), metallic lithium (15), zeolites of type NaA (58) or by recrystallization (12). Alcohols were purified by refluxing with calcium and fractional distillation (5). Acetonitrile, ethyl acetate, ether were redistilled from their mixture with $P_2O_5$ (25). Acetone was refluxed with powdered KOH and purified by the bisulfite process (5). Propylene carbonate (Matheson, Coleman & Bell) spectropure grade was fractionally distilled in the presence of $CaH_2$ and dry $N_2$ (25).

Anhydrous perchloric acid was distilled from a mixture of oleum and perchloric acid dihydrate at 100 °C under vacuum (20). Nickel perchlorate was synthetized from nickel carbonate and perchloric acid, followed by recrystallization (29).

Terbium perchlorate was prepared by heating terbium nitrate and dissolving the resulting terbium oxide in 56 % perchloric acid. The salt was recrystallized from aqueous solution and washed with chloroform (42).

## I BINARY SYSTEMS

When possible a statistical treatment of data was performed in order to make a critical evaluation or to check the coherence of data

### System $LiClO_4$-$H_2O$

The solubility of lithium perchlorate in aqueous solutions has been studied over a wide temperature range, from 273 K to 445 K and is presented in twenty-three publications

Within the accuracy of the analyses, the solid phases in equilibrium with saturated solutions are stoichiometric. Three solubility curves can be identified, involving anhydrous salt and two congruent melting hydrates $LiClO_4 \cdot H_2O$, $LiClO_4 \cdot 3H_2O$.

*Fitting equations*

The data of compilation sheets have been analyzed following the procedure described in the preface of volume 47 (59). The curves are represented by equation :

$$Y = f(T) = A/T + B \ln T + CT + D + ... \qquad [1]$$

where $T$ is the temperature (K) and $Y$ is the natural logarithm of solubility for the liquidus curve of ice, and is related to the solubility constant for the salts (table 1).

(continued)

CRITICAL EVALUATION (continued)

Table 1 $LiClO_4$-H2O

| | Expression of Y |
|---|---|
| $LiClO_4$ | $Y = \ln(4x^2/(1+x)^2)$ |
| $LiClO_4 \cdot H2O$ | $Y = \ln(27x^2(1-x)/(1+x)^3)$ |
| $LiClO_4 \cdot 3H2O$ | $Y = \ln(5^5x^2(1-x)^3/(27(1+x)^5)$ |

Four coefficients, deduced from experimental data by linear regression, were necessary to represent the data. Their values are given in table 2.

Table 2 System $LiClO_4$-H2O

Coefficients of fitting equations

| Solid Phase | A | B | C | D |
|---|---|---|---|---|
| $LiClO_4$ | -226579.46 | -1022.4424 | 1.1542909 | 6229.9317 |
| $LiClO_4 \cdot H_2O$ | 87977.65 | 446.3249 | 0.5600779 | -2670.172 |
| $LiClO_4 \cdot 3H_2O$ | -1943.37 | 0.503708 | -8.49846E-3 | 5.4328 |

*Solubility of anhydrous $LiClO_4$*

Only four data are available so that the coefficients A, B, C, D have been calculated by simple resolution of a system of 4 linear equations and the data above 172 °C must be considered as tentative data. The value 509 K (236 °C) given by Richards and Willard (2) for the melting point of lithium perchlorate has been adopted. The comparison between experimental and calculated values of solubility is presented in table 3.

Table 3 System $LiClO_4$-H2O

Liquidus of anhydrous $LiClO_4$

| *t* °C | exp *100w1* | calc | exp *100x1* | calc | ref |
|---|---|---|---|---|---|
| 144.2 | 90.0 | 90.04 | 60.4 | 60.5 | 7 |
| 167.5 | 91.04 | 91.06 | 63.24 | 63.3 | 7 |
| 172.0 | 91.11 | 91.13 | 63.44 | 63.5 | 7 |
| 236 | 100 | 100 | 100 | 100 | 2 |

The solid phase in equilibrium with liquid at 144.2 °C is probably anhydrous lithium perchlorate instead of the monohydrate claimed by the authors

(continued)

CRITICAL EVALUATION (continued)

*Solubility Curve of $LiClO_4 H_2O$*

The solubility Curve of $LiClO_4 \cdot H_2O$ has been determined only by Simmons and Ropp (7). The coherence of experimental results is good except for the data close to the melting point. The congruent melting point is given by solution of the equation :

$$D = - A/T_m - B\ln(T_m) - CT_m \qquad [2]$$

The calculated value is 152.2 °C, the experimental value is 149 °C (5).

Table 4 Liquidus of monohydrate $LiClO_4 \cdot H_2O$

| *t* °C | exp100w1 | calc | exp100x1 | calc | deviat.x1000 | ev. | ref |
|---|---|---|---|---|---|---|---|
| 93.2 | 70.5 | 70.53 | 28.8 | 28.84 | 0 | R | 7 |
| 97.3 | 71.0 | 70.95 | 29.3 | 29.26 | 0 | R | 7 |
| 108.9 | 72.8 | 72.74 | 31.2 | 31.12 | 3.2 | R | 7 |
| 120.7 | 75.0 | 75.23 | 33.7 | 33.96 | - 8.8 | R | 7 |
| 136.9 | 80.0 | 79.38 | 40.4 | 39.46 | 22.7 | T | 7 |
| 144.0 | 82.5 | 81.47 | 44.4 | 42.68 | 39.8 | T | 7 |
| 144.2 | 81.53 | | 42.78 | | | | ** |
| 148.5 | 85.0 | 83.02 | 49.0 | 45.29 | 81.7 | A | 7 |
| 149.3 | 87.5 | 83.34 | | 45.87 | | | ** |
| 149.3 | | 87.48 | 54.2 | 54.2 | 0 | R | 7 |
| 148.5 | | 87.74 | | 54.8 | | | ** |
| 144.2 | 90.0 | 88.85 | 60.4 | 57.45 | 50.5 | A | 7* |
| 136.9 | | 89.87 | | 60.03 | | | ** |
| 120.7 | | 92.37 | | 67.2 | | | ** |
| 108.9 | | 93.38 | | 70.48 | | | ** |
| 97.3 | | 94.01 | | 72.66 | | | ** |
| 93.2 | | 94.15 | | 73.15 | | | ** |

* belongs probably to the liquidus of anhydrous $LiClO_4$ ** calculated by evaluator
R = recommended, T = tentative, A = aberrant value

A comparison between experimental and calculated values of solubility is presented in table 4. The deviation is the quantity $100(x_{exp}-x_{calc})/x_{calc}$. The data have been recommended when dev < 2 % , considered as tentative when 2 % < dev < 5 % and aberrant above 5 %

*Solubility Curve of $LiClO_4 3H_2O$*

The range of solubility of trihydrate has been determined in totality by Simmons and Ropp above 0 °C, but in most papers the data are reported at 25 °C. Lithium perchlorate trihydrate crystallizes from aqueous solutions as short prismatic or needle-like crystals of the hexagonal form. The critical evaluation is given in table 5. The coherence of experimental results is good except for the data close to the melting point. The calculated congruent melting point is 95 °C.

(continued

CRITICAL EVALUATION (continued)

Table 5 Liquidus of trihydrate $LiClO_4 \cdot 3H_2O$

| $t$°C | exp $100w_1$ | calc | exp $100x_1$ | calc | devx1000 | ev. | ref |
|---|---|---|---|---|---|---|---|
| 0.0 | 29.90 | 29.91 | 6.736 | 6.740 | - 0.59 | R | 7 |
| 10.0 | 32.88 | 32.90 | 7.660 | 7.665 | - 0.65 | R | 7 |
| 20.0 | 35.95 | 35.91 | 8.679 | 8.665 | 1.62 | R | 7 |
| 25 | 37.30 | 37.44 | 9.170 | 9.152 | - 3.26 | R | 23 |
| 25 | 37.34 | 37.44 | 9.166 | 9.200 | - 3.70 | R | 30 |
| 25 | 37.34 | 37.44 | 9.166 | 9.200 | - 3.70 | R | 28 |
| 25 | 37.34 | 37.44 | 9.166 | 9.200 | - 3.70 | R | 26 |
| 25 | 37.34 | 37.44 | 9.166 | 9.200 | - 3.70 | R | 51 |
| 25 | 37.34 | 37.44 | 9.166 | 9.200 | - 3.70 | R | 48 |
| 25 | 37.34 | 37.44 | 9.166 | 9.200 | - 3.70 | R | 54 |
| 25 | 37.34 | 37.44 | 9.166 | 9.200 | - 3.70 | R | 47 |
| 25 | 37.38 | 37.44 | 9.188 | 9.200 | - 1.30 | R | 5 |
| 25 | 37.46 | 37.44 | 9.209 | 9.200 | 0.98 | R | 55 |
| 25.0 | 37.48 | 37.44 | 9.216 | 9.200 | 1.74 | R | 7 |
| 25 | 37.50 | 37.44 | 9.223 | 9.200 | 2.50 | R | 45 |
| 25 | 37.52 | 37.44 | 9.230 | 9.200 | 3.26 | R | 36 |
| 25 | 37.53 | 37.44 | 9.231 | 9.200 | 3.26 | R | 29 |
| 25 | 37.55 | 37.44 | 9.241 | 9.200 | 4.49 | R | 44 |
| 25 | 37.58 | 37.44 | 9.252 | 9.200 | 5.65 | R | 43 |
| 25 | 37.58 | 37.44 | 9.252 | 9.200 | 5.65 | R | 29 |
| 25 | 37.62 | 37.44 | 9.266 | 9.200 | 7.27 | R | 21 |
| 25 | 37.66 | 37.44 | 9.280 | 9.200 | 8.70 | R | 42 |
| 25 | 37.70 | 37.44 | 9.295 | 9.200 | 10.33 | T | 24 |
| 25 | 37.78 | 37.44 | 9.323 | 9.200 | 13.37 | T | 38 |
| 30.0 | 38.87 | 38.96 | 9.721 | 9.755 | - 3.49 | R | 17 |
| 35 | 41.09 | 40.49 | 10.56 | 10.330 | 22.27 | A | 22 |
| 40.0 | 41.97 | 42.04 | 10.91 | 10.940 | - 2.65 | R | 7 |
| 50 | 45.28 | 45.18 | 12.29 | 12.249 | 3.67 | R | 40 |
| 64.6 | 50.0 | 49.96 | 14.48 | 14.460 | 1.38 | R | 7 |
| 77.9 | 55.0 | 54.79 | 17.15 | 17.03 | 7.05 | R | 7 |
| 89.2 | 60.0 | 60.02 | 20.26 | 20.27 | - 0.49 | R | 7 |
| 92.3 | 62.5 | 62.12 | 22.01 | 21.735 | 12.65 | T | 7 |
| 94.3 | 65.0 | 64.21 | 23.92 | 23.30 | 26.61 | A | 7 |
| 95.1* | 66.32 | | 25.01 | | | | 7 |
| 94.8 | 66.67 | 67.41 | 25.30 | 25.94 | - 24.67 | A | 7 |
| 93.2 | 70.0 | 69.53 | 28.3 | 27.87 | 15.43 | T | 7 |
| 92.7 | 70.3 | 69.93 | 28.6 | 28.25 | - 58.41 | A | 7 |
| 92.5 | 70.33 | 70.08 | 28.64 | 28.40 | 8.45 | R | 7 |

R = recommended , T = tentative, A = aberrant value
* The calculated melting point is 95.0 °C

*Double saturation points*

Two eutectic points can be observed above 0 °C :

liq. $\leftrightarrow LiClO_4 \cdot 3H_2O + LiClO_4 \cdot H_2O$ T = 92.4 °C , $x_1 = 0.284$

(continued)

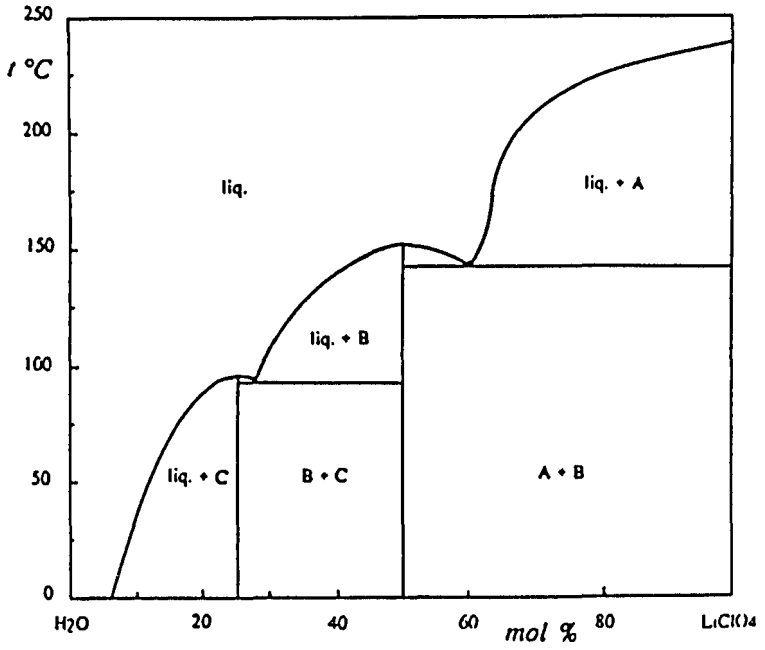
CRITICAL EVALUATION (continued)

liq. ⇌ $LiClO_4 \cdot H_2O$ + $LiClO_4$ $T = 140$ °C , $x_1 = 0.592$

*Phase diagram*

The phase diagram $LiClO_4$ - $H_2O$ between 0 °C and the melting point is presented in figure 1. The calculated solubility of mono and trihydrate are in good agreement with experimental data, the liquidus curve of anhydrous salt must be considered as tentative due to the small number of experimental data.

Figure 1 Solubility of lithium perchlorate in water




A = $LiClO_4$ B = $LiClO_4 \cdot H_2O$ C = $LiClO_4 \cdot 3H_2O$

Solubility of Lithium Perchlorate in other solvents

Due to the small number of papers it was not possible to perform a critical evaluation, so that all data must be considered as tentative.

The solubility of lithium perchlorate in tetrahydrofuran was 2.2 mol $dm^{-3}$ [35] at 298.2 K and could not be converted in mass units.

In most cases the solubility was measured at 298 K and the data at this temperature are collected in table 6.

(continued)

CRITICAL EVALUATION (continued)

Table 6 Solubility of LiClO4 in various solvents

| Solvent | | | | g % solvent | *xl* | ref |
|---|---|---|---|---|---|---|
| $CH_3NO$ | [75-12-7] | | formamide | 142.1±0.2 | 37.6 | 14 |
| $CH_4O$ | [67-56-1] | methanol | methyl alcohol | 182.25 | 35.44 | 5 |
| $C_2HF_3O_2$ | [76-05-1] | 2-trifluoroethanoic acid | trifluoroacetic acid | 11.7 | 11.1 | 14 |
| $C_2H_3N$ | [75-05-8] | acetonitrile | | 12.99 | 5.44 | 25 |
| | | | | 14.46 | 6.12 | 46 |
| | | | | 13.6±0,1 | 6.98 | 14 |
| $C_2H_4O_2$ | [64-19-7] | ethanoic acid | acetic acid | 108.7±0.1 | 38.0 | 14 |
| $C_2H_6O$ | [64-17-5] | ethanol | ethyl alcohol | 151.76 | 39.66 | 5 |
| $C_2H_6O_2$ | [107-21-1] | 1-2-ethanediol | ethyleneglycol | 96.7±0.4 | 36.1 | 14 |
| $C_2H_6OS$ | [67-68-5] | sulfinylbis-methane | dimethylsulphoxide | 21.1* | 17.4* | 12 |
| $C_2H_7NO$ | [141-43-5] | 2-aminoethanol | | 78.9±0.9 | 31.2 | 14 |
| $C_2H_8N_2$ | [107-15-3] | 1,2-ethanediamine | ethylenediamine | 48.0±0.3 | 21.3 | 14 |
| $C_3H_6O$ | [123-38-6] | propionaldehyde | propanal | 110.5±0.7 | 37.6 | 14 |
| $C_3H_6O$ | [75-56-9] | methyloxirane | propylene oxide | 91.4± 0.6 | 33.3 | 14 |
| $C_3H_6O$ | [67-64-1] | | acetone | 136.52 | 42.70 | 5 |
| $C_3H_7NO$ | [68-12-2] | N,N-dimethyl-form amide | | 7.5±0.2 | 34.0 | 14 |
| $C_3H_8O$ | [71-23-8] | 1-propanol | n-propyl alcohol | 105.0 | 37.23 | 5 |
| $C_3H_8O$ | [67-63-0] | 2-propanol | isopropyl alcohol | 112.1±0.1 | 38.8 | 14 |
| $C_3H_9N$ | [107-10-8] | 1-propanamine | n-propylamine | 59.1±0.3 | 24.7 | 14 |
| $C_4H_6O_3$ | [108-24-7] | | acetic anhydride | 8.1 | 7.2 | 14 |
| $C_4H_8O$ | [109-99-9] | | tetrahydrofurane | 27.1±0.2 | 15.5 | 14 |
| $C_4H_8O_2$ | [141-78-6] | | ethyl acetate | 95.12 | 44.1 | 5 |
| | | | | 95.1 | 44.1 | 14 |
| $C_4H_8O_2$ | [107-92-6] | butanoic acid | butyric acid | 60.0±0.3 | 33.2 | 14 |
| $C_4H_9NO_2$ | [544-16-1] | 1-butyl nitrite | n-butyl nitrite | 3.4 | 3.2 | 14 |
| $C_4H_{10}O$ | [60-29-7] | 1,1'-oxybis-ethane | diethylether | 113.72 | 44.21 | 5 |
| $C_4H_{10}O$ | [71-36-3] | 1-butanol | n-butyl alcohol | 79.31 | 35.59 | 5 |
| $C_4H_{10}O$ | [78-92-2] | 2-butanol | sec-butyl alcohol | 77.1±0.1 | 34.9 | 14 |
| $C_4H_{10}O$ | [78-83-1] | 2-methyl-1-propanol | isobutylalcohol | 58.05 | 28.8 | 5 |
| $C_4H_{10}O$ | [75-65-0] | 2-methyl-2-propanol | t-butyl alcohol | 0.6 | 0.4 | 14 |
| $C_4H_{10}O_2$ | [110-80-5] | 2-ethoxyethanol | | 136.6±0.4 | 53.6 | 14 |
| $C_4H_{11}N$ | [109-73-9] | 1-butanamine | n-butylamine | 45.7±0.3 | 23.9 | 14 |
| $C_4H_{11}N$ | [13952-84-6] | 2-butanamine | sec-butylamine | 45.7±0.2 | 23.9 | 14 |
| $C_4H_{11}N$ | [75-64-9] | 2-methyl-2-propananamine | t-butylamine | 10.7± 0.3 | 6.8 | 14 |
| $C_5H_5N$ | [110-86-1] | | pyridine | 8.7±0.1 | 6.1 | 14 |
| $C_5H_8O$ | [120-92-3] | | cyclopentanone | 63.8±0.2 | 33.5 | 14 |
| $C_5H_{10}O_3$ | [105-58-9] | | diethyl carbonate | 52.6±0.1 | 36.9 | 14 |
| $C_6H_7N$ | [62-53-3] | benzenamine | aniline | 6.1±0.2 | 5.1 | 14 |
| $C_6H_{10}O$ | [108-94-1] | cyclohexanone | | 54.0±0.3 | 33.2 | 14 |
| $C_6H_{10}O_3$ | [141-97-9] | ethyl-3-oxobuta noate | ethyl acetoacetate | 76.7±0.1 | 48.4 | 14 |

(continued)

CRITICAL EVALUATION (continued)

Table 6 (continued)

Solubility of $LiClO_4$ in various solvents at 298 K

| Solvent | | | | mass % solvent | $x_1$ | ref |
|---|---|---|---|---|---|---|
| $C_6H_{12}O$ | [108-93-0] | cyclohexanol | | 5.9±0.1 | 5.3 | 14 |
| $C_6H_{12}N$ | [108-91-8] | cyclohexanamine | cyclohexylamine | 16.9 | 13.6 | 14 |
| $C_7H_5N$ | [100-47-0] | benzonitrile | | 21.9±0.2 | 17.5 | 14 |
| $C_7H_6O$ | [100-52-7] | benzaldehyde | | 51.5±0.3 | 33.9 | 14 |
| $C_7H_8O$ | [100-51-6] | benzenemethanol | benzyl alcohol | 49.8±0.3 | 33.6 | 14 |
| $C_7H_8O$ | [108-39-4] | 3-methylphenol | | 142.1±0.2 | 37.6 | 14 |
| $C_7H_9N$ | [100-61-8] | n-methyl benzenamine | n-methylaniline | 1.4±0.1 | 1.4 | 14 |
| $C_8H_{16}O_2$ | [124-07-2] | 1-octanoic acid | n-octanoic acid | 32.1±0.3 | 30.3 | 14 |
| $C_8H_{18}O$ | [111-87-5] | 1-octanol | | 43.8±0.3 | 34.9 | 14 |
| $C_8H_{18}O$ | [123-96-6] | 2-octanol | | 44.7±0.3 | 35.4 | 14 |
| $C_8H_{18}O$ | [142-96-1] | 1-1'-oxybis-butane | n-butylether | 13.6±0.2 | 14.3 | 14 |
| $C_8H_{19}N$ | [111-92-2] | n-butyl-1-butanamine | dibutylamine | 45.6±0.4 | 35.6 | 14 |
| $C_9H_{10}O_2$ | [93-89-0] | | ethyl benzoate | 29.2±0.2 | 29.2 | 14 |
| $C_9H_{10}O_2$ | [140-11-4] | | benzyl acetate | 50.1±0.4 | 41.4 | 14 |
| $C_{12}H_{14}O_4$ | [84-66-2] | diethyl-1,2-benzene dicarboxylate | diethylphtalate | 5.5 | 1.2 | 14 |
| $C_{14}H_{26}O_4$ | [110-40-7] | diethyldecanedioate | diethylsebacate | 21.3±0.1 | 34.0 | 14 |
| $N_2H_4$ | [302-01-2] | | hydrazine | 54.4 | 14.1 | 39 |

* the solid phase is the solvate $2LiClO_4 \cdot 7(CH_3)_2SO$

Some solubilities have been measured at 273 K (table 7)

| Solvent | | | mol% solvent | $x_1$ | ref |
|---|---|---|---|---|---|
| $HClO_4$ | [7601-90-3] | perchloric acid | 0.107 | 0.1 | 20 |
| $H_2O_2$ | [7722-84-1] | hydrogen peroxide | 62.3 | 16.61 | 57 |
| $N_2H_4$* | [302-01-2] | hydrazine | 47.1 | 12.4 | 32 |

* The solid phase was $LiClO_4 \cdot 2N_2H_4$

Sometimes the measurements have been performed at several temperatures, so that a fitting equation has been derived in order to allow interpolation.

System $LiClO_4$-Acetonitrile [75-05-8]

Tomkins and Turner [46] have measured the solubility of lithium perchlorate in acetonitrile at temperatures between 24.20 and 50.17 °C. A congruent melting point is observed for $x_1$=0.20. The solid phase is $LiClO_4 \cdot 4CH_3CN$ and the data are fitted by the equation :

(continued)

CRITICAL EVALUATION (continued)

$\ln(x_1(1-x_1)^4) = A/T + B$ [3]

where $A = -2119.1747$, $B = 4.0842$.

The agreement between experimental and calculated solubilities is good except in the vicinity of the dystectic point (table 8)

Table 8 System $LiClO_4$-Acetonitrile

| 100 $x_1$ | $t$ °C exp. | $t$ °C calc | 100 d |
|---|---|---|---|
| 5.953 | 24.20 | 23.19 | 0.3 |
| 7.106 | 29.16 | 28.58 | 0.2 |
| 8.076 | 32.80 | 32,32 | 0.15 |
| 9.178 | 36.33 | 35.87 | 0.15 |
| 10.818 | 40.12 | 40.05 | 0.02 |
| 11.693 | 41.94 | 41.84 | 0.03 |
| 13.923 | 45.67 | 45.26 | 0.1 |
| 15.480 | 48.28 | 46.84 | 0.45 |
| 17.694 | 49.50 | 48.18 | 0.4 |
| 20.146 | 49.41 | 48.60 | 0.25 |
| 20.350 | 50.17 | 48.60 | 0.5 |
| 22.780 | 48.85 | 48.05 | 0.2 |
| 23.750 | 41.21 | 47.62 | - 2 |
| 24.240 | 45.66 | 47.36 | - 0.5 |
| 25.600 | 45.43 | 46.50 | - 0.3 |

The deviation is given by $d = (T_{exp.} - T_{calc.})/T_{calc.}$ The congruent melting point (dystectic point) is located at $x_1 = 0.20$ and $t = 49 \pm 1$°C

System $LiClO_4$-DMSO [67-68-5]

The data of Kenttamaa [12] at 25, 35 and 45 °C are not accurate and all data must be considered as tentative.

System $LiClO_4$-Propionitrile [107-12-0]

The solubility of lithium perchlorate in propionitrile at temperatures between 230 and 285 K has been measured by Tomkins and Turner [46]. A congruent melting point is observed for $x_1 = 0.20$. The solid phase is $LiClO_4 \cdot 4CH_3CN$ and the data are fitted by equation [3]. The values of coefficients are

$A = -628.3486$ $B = -0.17801$

The agreement between experimental and calculated solubilities is good (table 9, figure 2).

(continued)

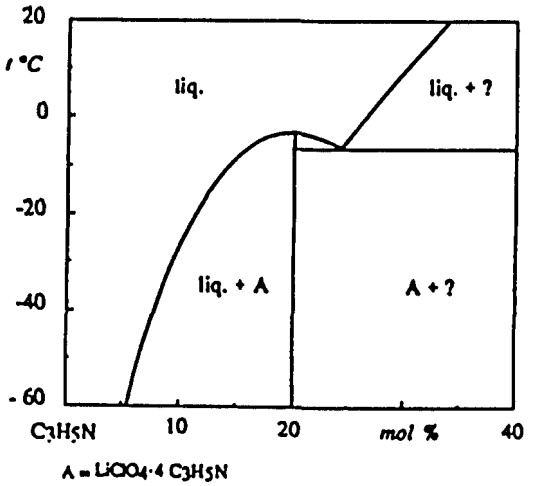
CRITICAL EVALUATION (continued)

Table 9 System $LiClO_4$-Propionitrile

| $100x_1$ | $t$°C exp. | $t$°C calc | 100 d | solid phase |
|---|---|---|---|---|
| 7.23 | - 43 | - 44.935 | 0.8 | $LiClO_4 \cdot 4C_3H_5N$ |
| 12.141 | - 21.3 | - 16.9 | - 1.7 | " |
| 14.218 | - 12.1 | - 10.3 | - 0.7 | " |
| 17.049 | - 0.5 | - 4.9 | - 1.6 | " |
| 18.62 | - 3.9 | - 3.6 | - 0.1 | " |
| 27.378 | 2.1 | | | ? |
| 30.97 | 12 | | | ? |

The deviation is given by $d = (t_{exp.} - t_{calc.})/t_{calc.}$ The congruent melting point is located at $x_1 = 0.20$ and $t = -3.2 \pm 1$°C and the eutectic point are close to $x_1 = 0.243$, $t = -6.3$ °C.

Figure 2 Solubility of Lithium perchlorate in propionitrile




II TERNARY SYSTEMS

Solubility of $LiClO_4 \cdot 3H_2O$ in various solvents at 298.15 K

The values of solubility are presented in table 10, the solid phase was not specified. In some cases it is assumed to be $LiClO_4 \cdot 3H_2O$

Table 10 Solubility of $LiClO_4 \cdot 3H_2O$ in various solvents

| Solvent | | | mass % | ref. |
|---|---|---|---|---|
| $CH_4O$ | [67-56-1] | Methanol | 60.95 | 5 |
| $C_2H_6O$ | [64-17-5] | Ethanol | 42.16 | 5 |
| $C_3H_6O$ | [67-64-1] | Acetone | 49.04 | 5 |
| $C_3H_8O$ | [71-23-8] | 1-Propanol | 26.82 | 5 |
| $C_4H_8O_2$ | [141-78-6] | Ethyl acetate | 27.41 | 5 |
| $C_4H_{10}O$ | [71-36-3] | 1-Butanol | 21.40 | 5 |
| $C_4H_{10}O$ | [78-83-1] | 2-Methyl-1-propanol | 18.85 | 5 |
| $C_4H_{10}O$ | [60-29-7] | dithyl ether | 0.196 | 5 |

(continued)

CRITICAL EVALUATION (continued)

Solubility in mixed solvents

*Solubility in solvents containing a trace of water*

Determinations at 25 and 60 °C have been made by Keller and Foster (25) with two solvents (propylene carbonate $C_4H_6O_3$ and N,N-dimethylformamide $C_3H_7NO$) and containing slight quantities of water.

Table 11 Solubility of lithium perchlorate in solvents containing a trace of water

| Solvent | T / K | $H_2O$ mg $dm^{-3}$ | $LiClO_4$ c/mol $dm^{-3}$ | ref |
|---|---|---|---|---|
| $C_3H_7NO$ (N,N-dimethylformamide) | 298 | 45 | 4.4 | 25 |
| | | 1000 | 3.5 | |
| | 333 | 45 | 4.8 | |
| | | 1000 | 4.9 | |
| $C_4H_6O_3$ (propylene carbonate) | 298 | 20 | 2.1 | 25 |
| | | 1000 | 3.1 | |
| | 333 | 20 | 3.1 | |
| | | 1000 | 3.1 | |

*System $LiClO_4$ - Acetamide - $H_2O$*

The isothermal section 298 K has been determined by Tarakanov (45). Four stoichiometric phases are observed $LiClO_4 \cdot 3H_2O$, $LiClO_4 \cdot 2CH_3CONH_2$, $LiClO_4 \cdot 4CH_3CONH_2$, $CH_3CONH_2$. The two intermediate compounds are very hygroscopic.

*System $LiClO_4$ - Dimethylurea- $H_2O$*

The isothermal section determined by Bestuzheva (55) shows that three solid phases must exist at 298 K : $LiClO_4 \cdot 3H_2O$, dimethylurea $C_3H_8N_2O$ and presumably, according to the compiler, $LiClO_4 \cdot 3C_3H_8N_2O \cdot H_2O$. The double-saturation points are not observed and the intermediate compound has a non-congruent solubility.

*System $LiClO_4$ - Hexamethylenetetramine - $H_2O$*

Four solubility curves are observed, involving $C_6H_{12}N_4$, $LiClO_4 \cdot 2C_6H_{12}N_4 \cdot 2H_2O$ which is an incongruently soluble compound, $LiClO_4 \cdot C_6H_{12}N_4 \cdot 3H_2O$ and $LiClO_4 \cdot 3H_2O$ Several anomalies are observed on the data sheet :

• The solubility diagram presented on the compilation sheet is not correct since the ratio $LiClO_4/H_2O$ is not the same for the various compounds, so that their representative points cannot be located on the same straight line.

(continued)

CRITICAL EVALUATION (continued)

• The nature of the solid phases presented in the last row of the table is not supported by the data.

Table 12 Solubility in mixed solvents at 298 K

Composition of double saturation points

| Equilibrium | composition (1) mol % (2) | | ref |
|---|---|---|---|
| System $LiClO_4$-$C_2H_5NO$-$H_2O$ | | | |
| liq. ↔ $LiClO_4 \cdot 3\,H_2O$ + $LiClO_4 \cdot 4CH_3CONH_2$ | 18.97 | 44.74 | 45 |
| liq. ↔ $LiClO_4 \cdot 4CH_3CONH_2$ + $LiClO_4 \cdot 2CH_3CONH_2$ | 17.82 | 57.86 | |
| liq. ↔ $LiClO_4 \cdot 2CH_3CONH_2$ + $CH_3CONH_2$ | 10.97 | 72.17 | |
| System $LiClO_4$-$C_3H_8N_2O$-$H_2O$ | | | |
| liq. ↔ $LiClO_4 \cdot 3\,H_2O$ + ? | 12.17 | 9.157 | 55 |
| liq. ↔ ? + $C_3H_8N_2O$ | ? | ? | |
| System $LiClO_4$-$C_6H_{12}N_4$-$H_2O$ | | | |
| liq. ↔ $LiClO_4 \cdot 3\,H_2O$ + $LiClO_4 \cdot C_6H_{12}N_4 \cdot 3H_2O$ | 8.89 | 2.09 | 43 |
| liq. ↔ $LiClO_4 \cdot C_6H_{12}N_4 \cdot 3H_2O$ + $LiClO_4 \cdot 2C_6H_{12}N_4 \cdot 2H_2O$ | 5.83* | 7.0* | |
| liq. ↔ $LiClO_4 \cdot 2C_6H_{12}N_4 \cdot 2H_2O$ + $C_6H_{12}N_4$ | 1.62* | 11.1* | |

* assumed by evaluator

*System $LiClO_4$ - Methylacetate - Propylene carbonate*

The solubility of $LiClO_4$ has been measured by Il'in and Demakin (58) at 6 temperatures between 283.2 and 323.2 K. A fitting equation proposed by the authors is presented in data sheet; it allows a precision better than 10 %, the calculation of solubility at any composition of solvents and a temperature range between 10 and 50 °C.

Ternary salt systems involving the same anion

*$LiClO_4$ - $MClO_4$ - $H_2O$, (M = Na, K, NH4, Tl)*

The systems have been studied at temperatures between 298 and 323 K. All solid phases are stoichiometric: the solid phase involving lithium is always $LiClO_4 \cdot 3H_2O$, the other observed phases are anhydrous perchlorates except for $NaClO_4$ which crystallizes, according to the temperature, as monohydrate or anhydrous salt. In a general way the data are coherent, but as they are presented in a single paper it was not possible to assess the accuracy of the data. The composition of eutonic points of the systems are presented in table 13

(continued)

CRITICAL EVALUATION (continued)

Table 13 Ternary systems $LiClO_4$ - $MClO_4$ - $H_2O$

M = Na, K, $NH_4$, Tl

Composition of eutonic points

| T / K | Equilibrium | (1) mol % | (2) | ref |
|---|---|---|---|---|
| 298 | liq. ↔ $LiClO_4 . 3 H_2O$ + $NaClO_4 . H2O$ | 2.36* | 20.23* | 21 |
| | liq. ↔ $LiClO_4 . 3 H_2O$ + $KClO_4$ | 9.24* | 0.036* | 24 |
| | liq. ↔ $LiClO_4 . 3 H_2O$ + $NH_4ClO_4$ | 8.92* | 0.68* | 23 |
| | liq. ↔ $LiClO_4 . 3 H_2O$ + $TlClO_4$ | 9.26* | 0.14* | 38 |
| 303 | liq. ↔ $LiClO_4 . 3 H_2O$ + $NH_4ClO_4$ | 9.66* | 0.599* | 17 |
| 308 | liq. ↔ $LiClO_4 . 3 H_2O$ + $NH_4ClO_4$ | 10.49* | 0.519 | 22 |
| 323 | liq. ↔ $LiClO_4 . 3 H_2O$ + $NaClO_4$ | 5.66* | 21.40* | 40 |
| | liq. ↔ $NaClO_4$ + $NaClO_4 . H_2O$ | 1.47** | 27.4** | 40 |

*average ** assumed by evaluator

Taking in account the common ion effect and the variation of the ionic strength of the solution, the ionic product S of $LiClO_4 \cdot 3H_2O$, we have :

$$S = x_1 (x_1+x_2) (1- x_1-x_2)^3 / (1+x_1+x_2)^5 \quad [4]$$

where $x_1$ and $x_2$ are the mole fractions of components. The data can be fitted by equation [5]

$$S = A + B x_1 + C x_1^2 \quad [5]$$

The coefficients A, B, C are presented in table 14 :

Table 14 Ionic product of $LiClO_4 \cdot 3H_2O$

in ternary systems $LiClO_4$ - $MClO_4$ - $H_2O$

| M | T/K | A | B | C |
|---|---|---|---|---|
| Na | 298 | - 0.5629 | 64.78 | -151 |
| | 323 | - 2.9187 | 94.57 | -198 |
| K | 298 | - 0.429 | 49.09 | 0 |
| NH4* | 298 | 1.8782 | 23.75 | 0 |
| Tl | 298 | 1.3383 | 30.16 | 0 |

*at 303 and 308 K the calculation of coefficients was not possible due to the scattering of experimental data

(continued)

CRITICAL EVALUATION (continued)

*Ternary systems $LiClO_4$ - $M(ClO_4)_2$ - $H_2O$, (M = Ca, Ba, Mn, Co, Ni, Cu )*

All systems have been studied at a single temperature 298 K. The isothermal sections have the same shape with two saturation curves and an eutonic point. The solid phases in equilibrium with liquid are $LiClO_4 \cdot 3\ H_2O$ and, according to the system $Ca(ClO_4)_2 \cdot 4\ H_2O$, $Ba(ClO_4)_2 \cdot 3\ H_2O$ or an hexahydrate $M(ClO_4)_2 \cdot 6\ H_2O$ for M = Mn, Co, Ni, Cu). The eutonic points of the systems are given in table 15.

Table 15 Ternary systems $LiClO_4$ - $M(ClO_4)_2$ - $H_2O$

Composition of eutonic points at 298 K

| Equilibrium | (1) mol % | (2) | ref |
|---|---|---|---|
| liq. ↔ $LiClO_4 \cdot 3\ H_2O$ + $Ca(ClO_4)_2 \cdot 4\ H_2O$ | 3.14 | 7.608 | 36 |
| liq. ↔ $LiClO_4 \cdot 3\ H_2O$ + $Ba(ClO_4)_2 \cdot 3\ H_2O$ | 2.62 | 7.85 | 48 |
| liq. ↔ $LiClO_4 \cdot 3\ H_2O$ + $Mn(ClO_4)_2 \cdot 6\ H_2O$ | 1.13 | 6.98 | 51 |
| liq. ↔ $LiClO_4 \cdot 3\ H_2O$ + $Co(ClO_4)_2 \cdot 6\ H_2O$ | 1.73* | 6.23* | 29 |
| liq. ↔ $LiClO_4 \cdot 3\ H_2O$ + $Ni(ClO_4)_2 \cdot 6\ H_2O$ | 2.157* | 6.00* | 29 |
| liq. ↔ $LiClO_4 \cdot 3\ H_2O$ + $Cu(ClO_4)_2 \cdot 6\ H_2O$ | 2.78* | 7.10 | 43 |

* average

If the solvation of ions is not taken in account, the ionic product of trihydrate $LiClO_4 \cdot 3\ H_2O$ is represented by equation [6] :

$$S = x_1\,(x_1+2x_2)\,(1-x_1-x_2)^3 / (1+x_1+2x_2)^5 \qquad [6]$$

and can be fitted by relation [5]. The coefficients of fitting equations are given in table 16.

Table 16 Ionic product of $LiClO_4 \cdot 3H_2O$

Systems $LiClO_4$ - $M(ClO_4)_2$ - $H_2O$ at 298 K

| M | A | B | C |
|---|---|---|---|
| Ca | - 0.0833 | 64.61 | -208 |
| Ba | - 0.1628 | 68.62 | -218 |
| Mn,Co, Ni | 0.1017 | 53.18 | -107 |
| Cu | 0.8245 | 18.97 | 186 |

The ionic product of $LiClO_4 \cdot 3\ H_2O$ is given by the same equation when M = Mn, C0, Ni showing that the structure of the saturated solution is the same and consequently, in spite of

[continued)

CRITICAL EVALUATION (continued)

the fact that each system has been determined by a a single author, the solubility data for the isothermal section 298 K of the systems involving Mn, Co and Ni can be recommended.
For the other sytems the data are coherent but no assessment can be done concerning the accuracy.

*Ternary systems $LiClO_4$ - $M(ClO_4)_3$ - $H_2O$ (M = Ce, Ga, Tb)*

All systems have been studied at a single temperature, 298 K. The isothermal sections have two saturation curves and an eutonic point. The solid phases in equilibrium with liquid are $LiClO_4 \cdot 3\ H_2O$ and, according to the system, $Ce(ClO_4)_3 \cdot 9\ H_2O$, $Ga(ClO_4)_3 \cdot 9\ H_2O$ or $Tb(ClO_4)_3 \cdot 9\ H_2O$. The eutonic points of the systems are presented in table 17

Table 17 Ternary systems $LiClO_4$ - $M(ClO_4)_3$ - $H_2O$

Composition of eutonic points at 298 K

| Equilibrium | (1) mol % | (2) | ref |
|---|---|---|---|
| liq. ↔ $LiClO_4 \cdot 3\ H_2O$ + $Ce(ClO_4)_3 \cdot 9\ H_2O$ | 1.54* | 7.35* | 47 |
| liq. ↔ $LiClO_4 \cdot 3\ H_2O$ + $Ga(ClO_4)_3 \cdot 9\ H_2O$ | 0.38* | 5.97* | 54 |
| liq. ↔ $LiClO_4 \cdot 3\ H_2O$ + $Tb(ClO_4)_3 \cdot 9\ H_2O$ | 1.47* | 6.64 | 42 |

* average

If the solvation of ions is not taken in account, the ionic product of trihydrate $LiClO_4 \cdot 3\ H_2O$ is represented by equation [7] :

$$S = x1\ (x_1+3x_2)\ (1-x_1-x_2)^3 / (1+x_1+3x_2)^5 \qquad [7]$$

and can be fitted by relation [5]. The coefficients of the fitting equations are given in table 18.

Table 18 Ionic product of $LiClO_4 \cdot 3H_2O$

Systems $LiClO_4$ - $M(ClO_4)_3$ - $H_2O$ at 298 K

| M | A | B | C |
|---|---|---|---|
| Ce | 0.329 | 45.56 | - 60 |
| Ga | 0.0678 | 53.33 | -111 |
| Tb | 0.0696 | 60.19 | -179 |

Ternary salt systems involving the same cation

*Ternary system $LiClO_4$ - $LiNO_3$ - $H_2O$*

Isothermal sections at 298 and 323 K are reported in three publications. Two of them (28, 30) concern the same set of data and have been condensed in the same data sheet. Two solid

(continued)

CRITICAL EVALUATION (continued)

phases, $LiClO_4 \cdot 3H_2O$ and $LiNO_3$, are observed. The existence of $LiNO_3 \cdot 5H_2O$ reported by Donnan and Burt (33) was not confirmed by the work of other researchers (8). The coordinates of eutonic points are given in table 19:

Table 19 System $LiClO_4$ - $LiNO_3$ - $H_2O$

Coordinates of eutonic points

| $T$ / K | $LiClO_4$ mol % | LiNO3 | ref |
|---|---|---|---|
| 298 | 2.09* | 17.79* | 28,30 |
| 323 | 4.30* | 26.86* | 40 |

* average

*Ternary system $LiClO_4$ - $Li_2CrO_4$ - $H_2O$*

Isothermal sections 298 and 308 K are reported (22, 26). Two solid phases, $LiClO_4 \cdot 3H_2O$ and $Li_2CrO_4 \cdot 2H_2O$, are observed. The coordinates of eutonic points are given in table 20.

Table 20 System $LiClO_4$ - $Li_2CrO_4$ - $H_2O$

Coordinates of eutonic points

| $T$ / K | $LiClO_4$ mol % | $Li_2CrO_4$ | ref |
|---|---|---|---|
| 298 | 0.455 | 11.27 | 26 |
| 323 | 0.344* | 2.09* | 22 |

*average

If the solvation of ions is not taken in account, the ionic product of $LiClO_4 \cdot 3\ H_2O$ is represented by equation [6] and can be fitted by relation [5]. The coefficients of fitting equations are given in table 21.

Table 21 Ionic product of $LiClO_4 \cdot 3H_2O$

Systems $LiClO_4$ - $Li_2CrO_4$ - $H_2O$

| $T$ / K | A | B | C |
|---|---|---|---|
| 298 | - 0.0411 | 64.9 | - 207 |
| 308 | - 0.0359 | 63.75 | - 164 |

III QUATERNARY SYSTEMS

In general a single set of data has been determined so that a critical evaluation cannot be performed.

(continued)

CRITICAL EVALUATION (continued)

Quaternary simple systems

*$LiClO_4$ - Ethanol - Ethyl acetate - $H_2O$*

The solubility of lithium perchlorate trihydrate in mixed. solvent ethanol-ethyl acetate is determined at 25 °C (6). The composition of solvent is measured in volume % and the solubility is expressed in mass % of $LiClO_4 \cdot 3H_2O$. The solid phase is assumed to be the trihydrate.

*$LiClO_4$ -$Mg(ClO_4)_2$ - Hexamethylenetetramine - $H_2O$*

The solubilities have been measured at 298 K by Kosheleva (52) The isobaric-isothermal monovariant lines and invariant points are determined. Six solid phases are observed $LiClO_4 \cdot 3H_2O$, $Mg(ClO_4)_2 \cdot 2C_6H_{12}N_4 \cdot 8H_2O$, $C_6H_{12}N_4$, $LiClO_4 \cdot 2C_6H_{12}N_4 \cdot 5H_2O$, $LiClO_4 \cdot C_6H_{12} \cdot N_4 \cdot 3H_2O$, $Mg(ClO_4)_2 \cdot 6H_2O$. A diagram has been drawn in Jänecke coordinates (see compilation sheet). Furthermore the quality of the graph shows evidently that all data must be considered as tentative

*$LiClO_4$ - $Cu(ClO_4)_2$ - N,N-dimethyl carbamide - $H_2O$*

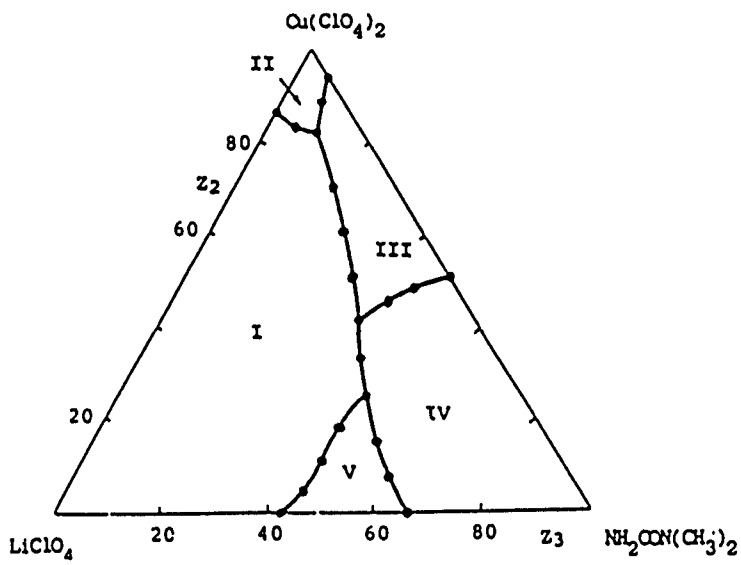
The quaternary system $LiClO_4$ - $Cu(ClO_4)_2$ - N,N-dimethyl carbamide - $H_2O$ has been investigated at 298 K.by Bestuzhzva et al. (50) The isobaric-isothermal monovariant lines and invariant points are determined. Five solid phases are observed :

I=$LiClO_4 \cdot 3H_2O$, II=$Cu(ClO_4)_2 \cdot 6H_2O$, III=$Cu(ClO_4)_2 \cdot 2NH_2CON(CH_3)_2 \cdot 4H_2O$

IV=$NH_2CON(CH_3)_2$, V=$LiClO_4 \cdot 3NH_2CON(CH_3)_2 \cdot H_2O$

A diagram has been drawn in Jänecke coordinates figure (3) where $Z_i = 100x_i/(x_1+x_2+x_3)$ and i= compounds (1), (2) or (3).

Figure 3 System $LiClO_4$ - $Cu(ClO_4)_2$ - N,N-dimethylcarbamide - Water




(continued)

CRITICAL EVALUATION (continued)

Quaternary reciprocal systems

Three quaternary reciprocal systems involving $LiCrO_4$ have been investigated. All data must be considered as tentative.

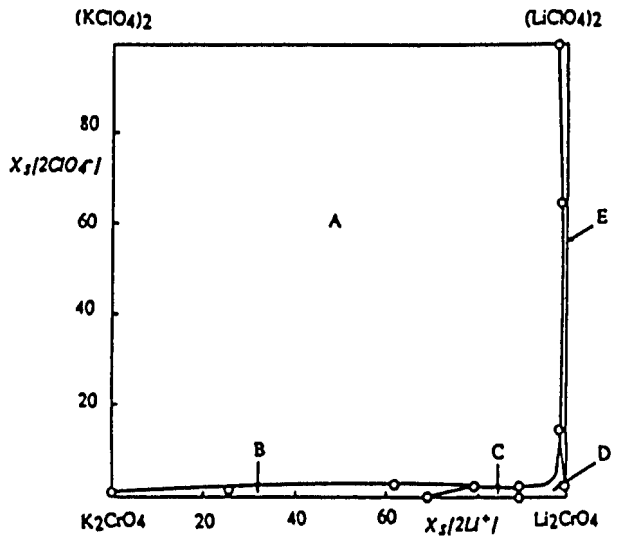
*$Li^+, K^+ // ClO_4^-, CrO_4^{2-} - H_2O$*

The quaternary system $Li^+$, $K^+$ // $ClO_4^-$, $CrO_4^{2-}$ - $H_2O$ has been investigated at 298 K by Voronina et al. (37). The isobaric-isothermal monovariant lines and invariant points are determined.

Five solid phases are observed A=$KClO_4$, B=$K_2CrO_4$, C=$Li_2CrO_4 \cdot K_2CrO_4 \cdot H_2O$, D=$Li_2CrO_4 \cdot 2H_2O$, E= $LiClO_4 \cdot 3H_2O$. A diagram (figure 4) has been drawn in Jänecke coordinates . The crystallization field of K $ClO_4$ is very large due to its small solubility .

Figure 4 Qaternary system $Li^+, K^+ / ClO_4^-, CrO_4^{2-} // H_2O$

Isothermal section at 298 K




*$Li^+, NH_4^+ // ClO_4^-, CrO_4^{2-} - H_2O$*

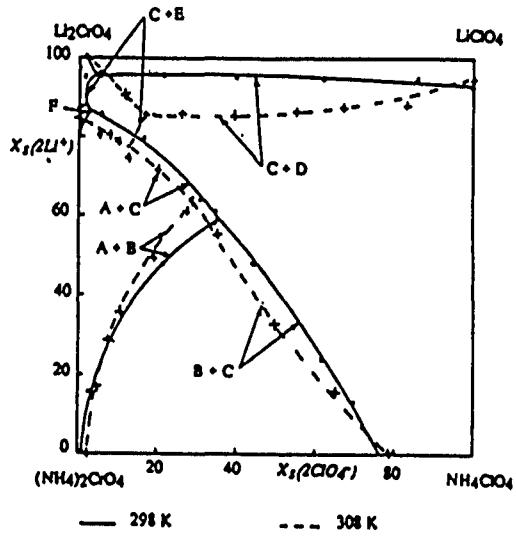
Two isothermal sections of the system $Li^+$, $NH_4^+$ // $ClO_4^-$, $CrO_4^{2-}$ - $H_2O$ have been determined. At 298 K. (31) six solid phases are observed : A=$(NH_4)_2CrO_4$, B=$NH_4ClO_4 \cdot (NH_4)_2CrO_4$, C=$NH_4ClO_4$, D=$LiClO_4 \cdot 3H_2O$, E=$Li_2CrO_4 \cdot 2H_2O$, F=$(NH_4)_2CrO_4 \cdot Li_2CrO_4 \cdot 2H_2O$. The compound F has a non congruent solubility. A diagram (figure 5) has been drawn in Jänecke coordinates. At 308 K (22) the phase diagram is very similar but the hydrated double salt F is no more observed. The isothermal section of the diagram is represented in figure 5 in dotted lines.

(continued)

CRITICAL EVALUATION (continuation)

Figure 5 Quaternary system $Li^+,NH_4^+/ClO_4^-,CrO_4^{2-}//H_2O$

Isothermal sections 298 and 308 K




*$Li^+$, $Mg^{2+}$ //$ClO_4^-$, $CrO_4^{2-}$ - $H_2O$*

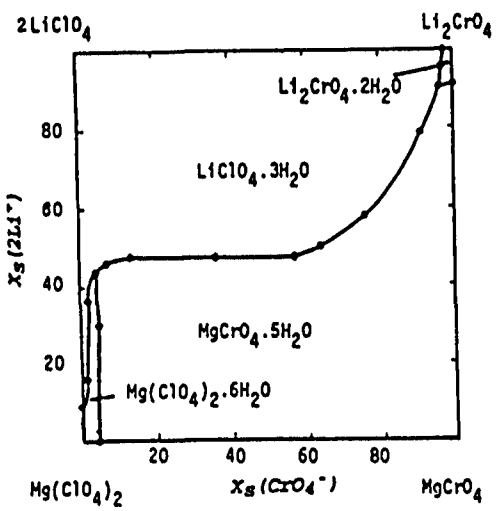
A section of the quaternary system $Li^+$, $Mg^{2+}$ // $ClO_4^-$, $CrO_4^{2-}$ - $H_2O$ has been determined at 298 K by Voronina (34). Four solid phases are observed :

A=$LiClO_4 \cdot 3H_2O$, $(NH_4)_2CrO_4$, B=$Mg(ClO_4)_2 \cdot 6H_2O$, C= $MgCrO_4 \cdot 5H_2O$, D=$Li_2CrO_4 \cdot 2H_2O$.

A graph in Janecke coordinates has been drawn (figure 6)

Figure 6 Quaternary system $Li^+,Mg^{2+}/ClO_4^-,CrO_4^{2-}//H_2O$

Isothermal section 298 K




(continued))

CRITICAL EVALUATION (continued)

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(continued)

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| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9] | Willard, H.H.; Smith, G.F. |
| (2) Water; $H_2O$; [7732-18-5] | *J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES: | PREPARED BY: |
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of lithium perchlorate in water at 25.00°C :

| mass % | g/100 $cm^{-3}$ sln | mol % | mol $dm^{-3}$ | mol $kg^{-1}$ | satd sln density/g $cm^{-3}$ |
|---|---|---|---|---|---|
| 37.385 | 47.42 | 9.182[b] | 4.457 | 5.612 [b] | 1.2683 |

[a] The solid phase is a mixture of anhydrous $LiClO_4$ and $LiClO_4.3H_2O$ .
[b] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-250°C in a current of air dried with $P_2O_5$. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:
Anhydrous $LiClO_4$ was prepared as described in ref. 1.

ESTIMATED ERROR:
Precision in temp. was ±0.01 °C .

REFERENCES:
1. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Simmons, J.P.; Ropp, C.D.L. |
| (2) Water; $H_2O$; [7732-18-5] | *J. Am. Chem. Soc.* 1928, *50*, 1650-3. |
| VARIABLES: | PREPARED BY: |
| Temperature: 273 - 445 K. | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of $LiClO_4$ in water at various temperatures:

| t / °C | T[a] / K | mass % | mol %[a] | molality[a]/mol kg$^{-1}$ | Solid phase |
|---|---|---|---|---|---|
| 0.0 | 273.2 | 29.90 | 6.736 | 4.009 | $LiClO_4.3H_2O$ |
| 10.0 | 283.2 | 32.88 | 7.660 | 4.604 | " |
| 20.0 | 293.2 | 35.95 | 8.679 | 5.276 | " |
| 25.0 | 298.2 | 37.48 | 9.216 | 5.635 | " |
| 30.0 | 303.2 | 38.87 | 9.721 | 5.977 | " |
| 40.0 | 313.2 | 41.97 | 10.911 | 6.798 | " |
| 64.6 | 337.8 | 50.0 | 14.48 | 9.40 | " |
| 77.9 | 351.1 | 55.0 | 17.15 | 11.49 | " |
| 89.2 | 362.4 | 60.0 | 20.26 | 14.10 | " |
| 92.3 | 365.5 | 62.5 | 22.01 | 15.67 | " |
| 94.3 | 367.5 | 65.0 | 23.92 | 17.46 | " |
| 95.1 | 368.3 | 66.32 | 25.01 | 18.51 | " |
| 94.8 | 368.0 | 66.67 | 25.30 | 18.80 | " |
| 93.2 | 366.4 | 70.0 | 28.3 | 21.9 | " |
| 92.7 | 365.9 | 70.3 | 28.6 | 22.2 | " |
| 92.5 | 365.7 | 70.33 | 28.64 | 22.28 | " |
| 93.2 | 366.4 | 70.5 | 28.8 | 22.5 | $LiClO_4.H_2O$ |
| 97.3 | 370.5 | 71.0 | 29.3 | 23.0 | " |
| 108.9 | 382.1 | 72.8 | 31.2 | 25.2 | " |
| 120.7 | 393.9 | 75.0 | 33.7 | 28.2 | " |
| 136.9 | 410.1 | 80.0 | 40.4 | 37.6 | " |
| 144.0 | 417.2 | 82.5 | 44.4 | 44.3 | " |
| 148.5 | 421.7 | 85.0 | 49.0 | 53.3 | " |
| 149.3 | 422.5 | 87.5 | 54.2 | 65.8 | " |
| 144.2 | 417.4 | 90.0 | 60.4 | 84.6 | " |
| 167.5 | 440.7 | 91.04 | 63.24 | 95.50 | $LiClO_4$ |
| 172.0 | 445.2 | 91.11 | 63.44 | 96.33 | " |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Soly measurements at the lower temperatures were carried out by agitating an excess of anhydrous salt with water in a soly tube in a thermostat until constancy in concentration was reached at the set temperature. These determinations were supplemented by results obtained by sealing known amounts of the anhydrous salt and water

SOURCE AND PURITY OF MATERIALS:

Anhydrous $LiClO_4$ was made using Richards and Willard's method (ref. 1). Two analyses using a modification of Lamb's method (ref. 2) gave values of 100.10 % and 100.15 % $LiClO_4$ purity.

ESTIMATED ERROR:

Temp. ± 0.1 °C; soly precision better than ± 0.1% (compiler).

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) *Alcohols*:<br>(A) Methanol (*methyl alcohol*); $CH_4O$; [67-56-1]<br>(B) Ethanol (*ethyl alcohol*); $C_2H_6O$; [64-17-5]<br>(C) 1-Propanol (*n-propyl alcohol*); $C_3H_8O$; [71-23-8]<br>(D) 1-Butanol (*n-butyl alcohol*); $C_4H_{10}O$; [71-36-3]<br>(E) 2-Methyl-1-propanol (*iso-butyl alcohol*); $C_4H_{10}O$; [78-83-1] | Willard, H.H.; Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4$ in various *alcohols* at 25.00°C :

| soly in : | methanol | ethanol | 1-propanol | 1-butanol | 2-methyl-1-propanol |
|---|---|---|---|---|---|
| g/100 g sln : | 64.57 | 60.28 | 51.22 | 44.23 | 36.73 |
| g/100 $cm^{-3}$ sln : | 89.44 | 79.41 | 61.49 | 49.25 | 38.94 |
| g/100 g solvent: | 182.25 | 151.76 | 105.00 | 79.31 | 58.05 |
| mol % [b] : | 35.44 | 39.66 | 37.23 | 35.59 | 28.80 |
| mol $dm^{-3}$ : | 8.406 | 7.463 | 5.779 | 6.646 | 3.660 |
| mol $kg^{-1}$ [b] : | 17.130 | 14.265 | 9.870 | 7.454 | 5.457 |

[a] In terms of the anhydrous salt which was the solid phase.
[b] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the | Lithium perchlorate trihydrate was prepared as described in ref.1 . Alcohols were purified by refluxing with calcium and fractional distillation.<br><br>(continued next page) |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) *Alcohols*:<br>(A) Methanol (*methyl alcohol*); $CH_4O$; [67-56-1]<br>(B) Ethanol (*ethyl alcohol*); $C_2H_6O$; [64-17-5]<br>(C) 1-Propanol (*n-propyl alcohol*); $C_3H_8O$; [71-23-8]<br>(D) 1-Butanol (*n-butyl alcohol*); $C_4H_{10}O$; [71-36-3]<br>(E) 2-Methyl-1-propanol (*iso-butyl alcohol*); $C_4H_{10}O$; [78-83-1] | Willard, H.H.; Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1923, *45*, 286-96. |

EXPERIMENTAL VALUES: (continued)

| | methanol | ethanol | 1-propanol | 1-butanol | 2-methyl-1-propanol |
|---|---|---|---|---|---|
| satd sln density/g $cm^{-3}$ : | 1.3849 | 1.3173 | 1.2006 | 1.1326 | 1.0602 |
| pure solvent density/g $cm^{-3}$ : | 0.78705 | 0.78515 | 0.8026 | 0.8059 | 0.7981 |

AUXILIARY INFORMATION (continued)

| METHOD/APPARATUS/PROCEDURE: | ESTIMATED ERROR: |
|---|---|
| thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles, making sure that organic solvent was completely removed before the salt was dried to constant weight at 250°C in a current of air dried with $P_2O_5$. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected. | Precision in temp. was ±0.01°C. |
| | REFERENCES:<br>1. Willard, H.H.; Smith, G.F. *J. Am. Chem.Soc.* 1922, *44*, 2816. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Simmons, J.P.; Ropp, C.D.L. |
| (2) Water; $H_2O$; [7732-18-5] | *J. Am. Chem. Soc.* 1928, *50*, 1650-3. |

EXPERIMENTAL VALUES: (continued)

COMMENTS AND/OR ADDITIONAL DATA

Densities of the saturated solutions at the lower temperatures were made by weighing a known volume of each.

Densities of saturated solutions of $LiClO_4$

| t / °C : | 0 | 10 | 20 | 25 | 30 | 40 |
|---|---|---|---|---|---|---|
| density / g $cm^{-3}$ : | 1.215 | 1.236 | 1.258 | 1.269 | 1.277 | 1.300 |

Saturated solutions of $LiClO_4$ cooled from temperatures between 0 °C to 90 °C, approximately, yielded hydrates which, after careful drying between filter papers, showed upon analysis a water content of 33.90 %, ( theoretical value for the trihydrate is 33.68 %). A powdered quantity of the trihydrate placed in a desicator over the anhydrous salt for six weeks yielded a lower hydrate. Determinations of the water content of this hydrate were made at frequent intervals until a constant value of 14.52 % was obtained. The theoretical value of the water content of the monohydrate is 14.48 %.

The transition temperature for $LiClO_4.3H_2O \equiv LiClO_4.H_2O$ was 92.53 °C.

The transition temperature for $LiClO_4.H_2O \equiv LiClO_4$ was 145.75 °C.

The melting point of the trihydrate was 95.1 °C while that for the monohydrate was 149 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: (continued)

in a tube immersed in water and gradually heating until the solid phase disappeared. The tube was cooled until hydrate crystallized out. The temperature was then carefully raised 0.1 °C at a time, with constant agitation, and the temperature noted at which the last trace of solid phase disappeared. This latter method was used for the measurements at the higher temperatures.

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2. Lamb; Marden *J. Am. Chem. Soc.* 1912, *34*, 812.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) *Alcohols* :<br>(A) 1,2-ethanediol (*ethylene glycol*); $C_2H_6O_2$; [107-21-1]<br>(B) 2-propanol (*isopropyl alcohol*); $C_3H_8O$; [67-63-0]<br>(C) 2-methyl-2-propanol (*t-butyl alcohol*); $C_4H_{10}O$; [75-65-0]<br>(D) 2-butanol (*sec-butyl alcohol*); $C_4H_{10}O$; [78-92-2]<br>(E) Cyclohexanol; $C_6H_{12}O$; [108-93-0]<br>(F) Benzenemethanol (*benzyl alcohol*); $C_7H_8O$; [100-51-6]<br>(G) 1-octanol; $C_8H_{18}O$; [111-87-5]<br>(H) 2-octanol; $C_8H_{18}O$; [123-96-6] | Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F.<br><br>*J. Chem. Eng. Data* 1961, *6*, 325-7. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4$ in various *alcohols* at 25.00°C :

| solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] | Analysis |
|---|---|---|---|---|
| (A) *ethylene glycol* | 96.7 ± 0.4 | 36.1 | 9.09 | Method I |
| (B) *isopropyl alcohol* | 112.1 ± 0.1 | 38.8 | 10.54 | " I |
| (C) *t-butyl alcohol* | 0.6 | 0.4 | 0.06 | " II |
| (D) *sec-butyl alcohol* | 77.1 ± 0.1 | 34.9 | 7.25 | " I |

[a] The solid phase was the anhydrous salt.

[b] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Method I: Solute content was determined by pptn as nitron perchlorate.<br>Method II: Solute content was determined as AgCl after conversion of perchlorate by fusion with anhydrous $Na_2CO_3$ in a Pt crucible. | Anhydrous lithium perchlorate was prepared (ref. 1) and analysed by precipitation as nitron perchlorate. Analysis showed 93.7 % in $ClO_4$ (theoretical 93.5 %).<br>Solvents were reagent grade.<br>(continued next page) |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) *Alcohols* :<br>(A) 1,2-ethanediol (*ethylene glycol*); $C_2H_6O_2$; [107-21-1]<br>(B) 2-propanol (*isopropyl alcohol*); $C_3H_8O$; [67-63-0]<br>(C) 2-methyl-2-propanol (*t-butyl alcohol*); $C_4H_{10}O$; [75-65-0]<br>(D) 2-butanol (*sec-butyl alcohol*); $C_4H_{10}O$; [78-92-2]<br>(E) Cyclohexanol; $C_6H_{12}O$; [108-93-0]<br>(F) Benzenemethanol (*benzyl alcohol*); $C_7H_8O$; [100-51-6]<br>(G) 1-octanol; $C_8H_{18}O$; [111-87-5]<br>(H) 2-octanol; $C_8H_{18}O$; [123-96-6] | Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F.<br><br>*J. Chem. Eng. Data* 1961, *6*, 325-7. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES: (continued)

Solubility[a] of $LiClO_4$ in various *alcohols* at 25.00°C :

| solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] | Analysis |
|---|---|---|---|---|
| (E) *cyclohexanol* | 5.9 ± 0.1 | 5.3 | 0.55 | Method II |
| (F) *benzyl alcohol* | 49.8 ± 0.3 | 33.6 | 4.68 | " II |
| (G) *1-octanol* | 43.8 ± 0.1 | 34.9 | 4.12 | " II |
| (H) *2-octanol* | 44.7 ± 0.2 | 35.4 | 4.20 | " II |

[a] The solid phase was the anhydrous salt. [b] Compiler's calculations.

AUXILIARY INFORMATION (continued)

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Three determinations were made from two separate samples. Constancy of density or of refractive index was taken as the criterion for saturation equilibrium. No details of saturation method were given. | Solvents E,F and G were further purified by fractional distillation after drying over anhydrous calcium sulfate. |
| | ESTIMATED ERROR:<br>± 0.02 °C in temperature. |

REFERENCE:

1. Markowitz, M.M. *J. Phys. Chem.* 1958, *62*, 827.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F. |
| (2) *Amines* : | *J. Chem. Eng. Data* 1961, *6*, 325-7. |
| (A) 1,2-ethanediamine (*ethylenediamine*); $C_2H_8N_2$; [107-15-3] | |
| (B) 1-propanamine (*n-propylamine*); $C_3H_9N$; [107-10-8] | |
| (C) 1-butanamine (*n-butylamine*); $C_4H_{11}N$; [109-73-9] | |
| (D) 2-butanamine (*sec-butylamine*); $C_4H_{11}N$; [13952-84-6] | |
| (E) 2-methyl-2-propanamine; $C_4H_{11}N$; [75-64-9] | |
| (F) Pyridine; $C_5H_5N$; [110-86-1] | |
| (G) Benzenamine (*aniline*); $C_6H_7N$; [62-53-3] | |
| (H) Cyclohexanamine (*cyclohexylamine*); $C_6H_{12}N$; [108-91-8] | |
| (I) N-methylbenzenamine (*N-methylaniline*); $C_7H_9N$; [100-61-8] | |
| (J) N-butyl-1-butanamine (*dibutylamine*); $C_8H_{19}N$; [111-92-2] | |

| VARIABLES: | PREPARED BY: |
|---|---|
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4$ at 25.00°C :

| solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] | Analysis |
|---|---|---|---|---|
| (A) *ethylenediamine* | 48.0 ± 0.3 | 21.3 | 4.51 | Method I |
| (B) *n-propylamine* | 59.1 ± 0.3 | 24.7 | 5.56 | " II |
| (C) *n-butylamine* | 45.7 ± 0.3 | 23.9 | 4.30 | " I |
| (D) *sec-butylamine* | 45.7 ± 0.2 | 23.9 | 4.30 | " I |

[a] The solid phase was the anhydrous salt.
[b] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Method I: Solute content was determined by pptn as nitron perchlorate. Method II: Solute content was determined as AgCl after conversion of perchlorate by fusion with anhydrous $Na_2CO_3$ in a Pt crucible. Three determinations were made from | Anhydrous lithium perchlorate was prepared (ref. 1) and analysed by precipitation as nitron perchlorate. Analysis showed 93.7 % in $ClO_4$ (theoretical 93.5 %). Solvents were reagent grade. Solvents B,C,D,E,F,G and H were (continued next page) |

COMPONENTS:

(1) Lithium perchlorate; $LiClO_4$; [7791-03-9]

(2) *Amines* :

(A) 1,2-ethanediamine (*ethylenediamine*); $C_2H_8N_2$; [107-15-3]

(B) 1-propanamine (*n-propylamine*); $C_3H_9N$; [107-10-8]

(C) 1-butanamine (*n-butylamine*); $C_4H_{11}N$; [109-73-9]

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

(E) 2-methyl-2-propanamine; $C_4H_{11}N$; [75-64-9]

(F) Pyridine; $C_5H_5N$; [110-86-1]

(G) Benzenamine (*aniline*); $C_6H_7N$; [62-53-3]

(H) Cyclohexanamine (*cyclohexylamine*); $C_6H_{12}N$; [108-91-8]

(I) N-methylbenzenamine (*N-methylaniline*); $C_7H_9N$; [100-61-8]

(J) N-butyl-1-butanamine (*dibutylamine*); $C_8H_{19}N$; [111-92-2]

ORIGINAL MEASUREMENTS:

Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F.

*J. Chem. Eng. Data* 1961, *6*, 325-7.

EXPERIMENTAL VALUES: (continued)

| | Solubility[a] of $LiClO_4$ at 25.00°C : | | | |
|---|---|---|---|---|
| solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] | Analysis |
| (E) *t-butylamine* | 10.7 ± 0.3 | 6.8 | 1.01 | Method I |
| (F) *pyridine* | 8.7 ± 0.1 | 6.1 | 0.82 | " I |
| (G) *aniline* | 6.1 ± 0.2 | 5.1 | 5.7 | " II |
| (H) *cyclohexylamine* | 16.9 ± 0.1 | 13.6 | 1.59 | " II |
| (I) *N-methylaniline* | 1.4 ± 0.1 | | 0.13 | " II |
| (J) *dibutylamine* | 45.6 ± 0.4 | 35.6 | 4.29 | " II |

[a] The solid phase was the anhydrous salt.

[b] Compiler's calculations.

AUXILIARY INFORMATION (continued)

METHOD/APPARATUS/PROCEDURE:

two separate samples. Constancy of density or of refractive index was taken as the criterion for saturation equilibrium. No details of saturation method were given.

SOURCE AND PURITY OF MATERIALS:

further purified by fractional distillation after drying over anhydrous calcium sulfate.

ESTIMATED ERROR:

± 0.02 °C in temperature.

REFERENCE:

1. Markowitz, M.M. *J. Phys. Chem.* 1958, *62*, 827.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) *Organic acids* :<br>(A) Ethanoic (*acetic*) acid; $C_2H_4O_2$; [64-19-7]<br>(B) 2-trifluoroethanoic (*trifluoroacetic*) acid; $C_2HF_3O_2$; [76-05-1]<br>(C) Butanoic (*butyric*) acid; $C_4H_8O_2$; [107-92-6]<br>(D) 1-octanoic (*n-octanoic*); $C_8H_{16}O_2$; [124-07-2] | Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F.<br><br>*J. Chem. Eng. Data* 1961, *6*, 325-7. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4$ in various *organic acids* at 25.00°C :

| *acid* | g/100g solvent | mol % | molality[b] / mol $kg^{-1}$ |
|---|---|---|---|
| (A) acetic | 108.7 ± 0.1 | 38.0 | 10.217 |
| (B) trifluoroacetic | 11.7 | 11.1 | 1.10 |
| (C) butyric | 60.0 ± 0.3 | 33.2 | 5.64 |
| (D) n-octanoic | 32.1 ± 0.3 | 30.3 | 3.02 |

[a] The solid phase was the anhydrous salt.
[b] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Solute content was determined by difference after direct titration of the acid solvent with base. Constancy of density or of refractive index was taken as the criterion for saturation equilibrium. Three determinations were made from two separate samples. | Anhydrous lithium perchlorate was prepared (ref. 1) and analysed by precipitation as nitron perchlorate. Analysis showed 93.7 % in $ClO_4$ (theoretical 93.5 %). Solvents were reagent grade. |
| | ESTIMATED ERROR:<br>± 0.02 °C in temperature. |

REFERENCE:

1. Markowitz, M.M. *J. Phys. Chem.* 1958, *62*, 827.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F. |
| (2) (A) Acetonitrile; $C_2H_3N$; [75-05-8] | *J. Chem. Eng. Data* 1961, *6*, 325-7. |
| (B) Benzonitrile; $C_7H_5N$; [100-47-0] | |
| (C) Formamide; $CH_3NO$; [75-12-7] | |
| (D) 2-aminoethanol; $C_2H_7NO$; [141-43-5] | |
| (E) N,N-dimethylformamide; $C_3H_7NO$; [68-12-2] | |
| (F) Acetic anhydride; $C_4H_6O_3$; [108-24-7] | |
| (G) 2-ethoxyethanol; $C_4H_{10}O_2$; [110-80-5] | |
| (H) 3-methylphenol; $C_7H_8O$; [108-39-4] | |

| VARIABLES: | PREPARED BY: |
|---|---|
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4$ at 25.00°C :

| | solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] | Analysis |
|---|---|---|---|---|---|
| (A) | acetonitrile | 16.3 ± 0.1 | 5.9 | 1.53 | Method III |
| (B) | benzonitrile | 21.9 ± 0.2 | 17.5 | 2.06 | " II |
| (C) | formamide | 142.1 ± 0.2 | 37.6 | 13.36 | " I |
| (D) | 2-aminoethanol | 78.9 ± 0.9 | 31.2 | 7.42 | " I |
| (E) | N,N-dimethylformamide | 75.0 ± 0.2 | 34.0 | 7.05 | " I |

[a] The solid phase was the anhydrous salt.
[b] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Method I : Solute content was determined by pptn as nitron perchlorate. Method II: Solute content was determined as AgCl after conversion of perchlorate by fusion with anhydrous $Na_2CO_3$ in a Pt crucible. Method III: Solute content was determined by evaporation of solvent | Anhydrous lithium perchlorate was prepared (ref. 1) and analysed by precipitation as nitron perchlorate. Analysis showed 93.7 % in $ClO_4$ (theoretical 93.5 %). Solvents were reagent grade. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F. |
| (2) (A) Acetonitrile; $C_2H_3N$; [75-05-8] | *J. Chem. Eng. Data* 1961, *6*, 325-7. |
| (B) Benzonitrile; $C_7H_5N$; [100-47-0] | |
| (C) Formamide; $CH_3NO$; [75-12-7] | |
| (D) 2-aminoethanol; $C_2H_7NO$; [141-43-5] | |
| (E) N,N-dimethylformamide; $C_3H_7NO$; [68-12-2] | |
| (F) Acetic anhydride; $C_4H_6O_3$; [108-24-7] | |
| (G) 2-ethoxyethanol; $C_4H_{10}O_2$; [110-80-5] | |
| (H) 3-methylphenol; $C_7H_8O$; [108-39-4] | |

| VARIABLES: | PREPARED BY: |
|---|---|
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4$ at 25.00°C :

| | solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] | Analysis |
|---|---|---|---|---|---|
| (F) | acetic anhydride | 8.1 | 7.2 | 0.76 | Method III |
| (G) | 2-ethoxyethanol | 136.6 ± 0.4 | 53.6 | 12.84 | " II |
| (H) | 3-methylphenol | 142.1 ± 0.2 | 37.6 | 13.36 | " II |

[a] The solid phase was the anhydrous salt.
[b] Compiler's calculations.

AUXILIARY INFORMATION (continued)

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| and prolonged vacuum drying of the residue at 150 °C. Constancy of density or of refractive index was taken as the criterion for saturation equilibrium. Three determinations were made from two separate samples. No details of saturation method were given. | Solvents A,C,E,G,& H were further purified by fractional distillation after drying over anhydrous calcium sulfate. |
| | ESTIMATED ERROR: ± 0.02 °C in temperature. |

REFERENCE:

1. Markowitz, M.M. *J. Phys. Chem.* 1958, *62*, 827.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) *Aldehydes and ketones* :<br>(A) Propanal (*propionaldehyde*); $C_3H_6O$; [123-38-6]<br>(B) Cyclopentanone; $C_5H_8O$; [120-92-3]<br>(C) Cyclohexanone; $C_6H_{10}O$; [108-94-1]<br>(D) Benzaldehyde; $C_7H_6O$; [100-52-7] | Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F.<br><br>*J. Chem. Eng. Data* 1961, *6*, 325-7. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4$ at 25.00°C.

| solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] |
|---|---|---|---|
| (A) *propionaldehyde* | 110.5 ± 0.7 | 37.6 | 10.39 |
| (B) *cyclopentanone* | 63.8 ± 0.2 | 33.5 | 6.00 |
| (C) *cyclohexanone* | 54.0 ± 0.3 | 33.2 | 5.08 |
| (D) *benzaldehyde* | 51.5 ± 0.3 | 33.9 | 4.84 |

[a] The solid phase was the anhydrous salt.
[b] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Solute content was determined as AgCl after conversion of perchlorate by fusion with anhydrous $Na_2CO_3$ in a Pt crucible. Constancy of density or of refractive index was taken as the criterion for saturation equilibrium. No details of saturation method were given. Three determinations were from two separate samples. | Anhydrous lithium perchlorate was prepared (ref. 1) and analysed by precipitation as nitron perchlorate. Analysis showed 93.7 % in $ClO_4$ (theoretical 93.5 %). Solvents were reagent grade. Solvents A, B and C were further purified by fractional distillation after drying over anhydrous calcium sulfate. |
| | ESTIMATED ERROR:<br>± 0.02 °C in temperature. |

REFERENCE:

1. Markowitz, M.M. *J. Phys. Chem.* 1958, *62*, 827.

COMPONENTS:

(1) Lithium perchlorate; $LiClO_4$; [7791-03-9]

(2) *Esters* :

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

(B) 1-butyl nitrite; (*n-butyl nitrite*); $C_4H_9NO_2$; [544-16-1]

(C) Diethyl carbonate; $C_5H_{10}O_3$; [105-58-9]

(D) Ethyl 3-oxobutanoate (*ethyl acetoacetate*); $C_6H_{10}O_3$; [141-97-9]

(E) Ethyl benzoate; $C_9H_{10}O_2$; [93-89-0]

(F) Benzyl acetate; $C_9H_{10}O_2$; [140-11-4]

(G) Diethyl-1,2-benzenedicarboxylate (*diethyl phthalate*); $C_{12}H_{14}O_4$; [84-66-2]

(H) Diethyl decanedioate (*diethyl sebacate*); $C_{14}H_{26}O_4$; [110-40-7]

ORIGINAL MEASUREMENTS:

Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F.

*J. Chem. Eng. Data* 1961, *6*, 325-7.

VARIABLES:

One temperature: 298.15 K

PREPARED BY:

C.Y. Chan

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4$ at 25.00°C :

| solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] | Analysis |
|---|---|---|---|---|
| (A) *ethyl acetate* | 95.1 | 44.1 | 8.94 | Method II |
| (B) *n-butyl nitrite* | 3.4 | 3.2 | 0.32 | " III |
| (C) *diethyl carbonate* | 52.6 ± 0.1 | 36.9 | 4.94 | " I |
| (D) *ethyl acetoacetate* | 76.7 ± 0.1 | 48.4 | 7.21 | " II |

[a] The solid phase was the anhydrous salt.

[b] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method I : Solute content was determined by pptn as nitron perchlorate.

Method II: Solute content was determined as AgCl after conversion of perchlorate by fusion with anhydrous $Na_2CO_3$ in a Pt crucible.

Method III: Solute content was determined by evaporation of solvent

SOURCE AND PURITY OF MATERIALS:

Anhydrous lithium perchlorate was prepared (ref. 1) and analysed by precipitation as nitron perchlorate. Analysis showed 93.7 % in $ClO_4$ (theoretical 93.5 %).

Solvents were reagent grade.

(continued next page)

COMPONENTS:

(1) Lithium perchlorate; $LiClO_4$; [7791-03-9]

(2) *Esters* :

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

(B) 1-butyl nitrite; (*n-butyl nitrite*); $C_4H_9NO_2$; [544-16-1]

(C) Diethyl carbonate; $C_5H_{10}O_3$; [105-58-9]

(D) Ethyl 3-oxobutanoate (*ethyl acetoacetate*); $C_6H_{10}O_3$; [141-97-9]

(E) Ethyl benzoate; $C_9H_{10}O_2$; [93-89-0]

(F) Benzyl acetate; $C_9H_{10}O_2$; [140-11-4]

(G) Diethyl-1,2-benzenedicarboxylate (*diethyl phthalate*); $C_{12}H_{14}O_4$; [84-66-2]

(H) Diethyl decanedioate (*diethyl sebacate*); $C_{14}H_{26}O_4$; [110-40-7]

ORIGINAL MEASUREMENTS:

Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F.

*J. Chem. Eng. Data* 1961, *6*, 325-7.

EXPERIMENTAL VALUES: (continued)

Solubility[a] of $LiClO_4$ at 25.00°C :

| solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] | Analysis |
|---|---|---|---|---|
| (E) *ethyl benzoate* | 29.2 ± 0.2 | 29.2 | 2.75 | Method II |
| (F) *benzyl acetate* | 50.1 ± 0.4 | 41.4 | 4.71 | " II |
| (G) *diethyl phthalate* | 5.5 | 1.2 | 5.2 | " II |
| (H) *diethyl sebacate* | 21.3 ± 0.1 | 34.0 | 2.00 | " II |

[a] The solid phase was the anhydrous salt.

[b] Compiler's calculations.

AUXILIARY INFORMATION (continued)

METHOD/APPARATUS/PROCEDURE:

and prolonged vacuum drying of the residue at 150 °C. Constancy of density or of refractive index was taken as the criterion for saturation equilibrium. Three determinations were made from two separate samples. No details of saturation method were given.

SOURCE AND PURITY OF MATERIALS:

Solvents B and C were further purified by fractional distillation after drying over anhydrous calcium sulfate.

ESTIMATED ERROR:

± 0.02 °C in temperature.

REFERENCE:

1. Markowitz, M.M. *J. Phys. Chem.* 1958, *62*, 827.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) *Ethers* :<br>(A) Methyl-oxirane (*propylene oxide*); $C_3H_6O$; [75-56-9]<br>(B) Tetrahydrofuran; $C_4H_8O$; [109-99-9]<br>(C) 1,1'-oxybis-butane (*n-butyl ether*); $C_8H_{18}O$; [142-96-1] | Markowitz, M.M.; Hawley, W.N.; Boryta, D.A.; Harris, R.F.<br><br>*J. Chem. Eng. Data* 1961, *6*, 325-7. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4$ at 25.00°C.

| solvent | g/100g solvent | mol % | mol $kg^{-1}$ [b] | Analysis |
|---|---|---|---|---|
| (A) *propylene oxide* | 91.4 ± 0.6 | 33.3 | 8.59 | Method II |
| (B) *tetrahydrofuran* | 27.1 ± 0.2 | 15.5 | 2.55 | " I |
| (C) *n-butyl ether* | 36.0 ± 0.2 | 14.3 | 12.8 | " I |

[a] The solid phase was the anhydrous salt.
[b] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Method I : Solute content was determined by pptn as nitron perchlorate.<br>Method II: Solute content was determined as AgCl after conversion of perchlorate by fusion with anhydrous $Na_2CO_3$ in a Pt crucible.<br>Constancy of density or of refracindex was taken as the criterion for saturation equilibrium. No details of saturation method were given.<br>Three determinations were made from two separate samples. | Anhydrous lithium perchlorate was prepared (ref. 1) and analysed by precipitation as nitron perchlorate. Analysis showed 93.7 % in $ClO_4$ (theoretical 93.5 %).<br>Solvents were reagent grade.<br>Solvents B and C were further purified by fractional distillation after drying over anhydrous calcium sulfate. |
| | ESTIMATED ERROR:<br>± 0.02 °C in temperature. |

REFERENCE:

1. Markowitz, M.M. *J. Phys. Chem.* 1958, *62*, 827.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Willard, H.H.; Smith, G.F. |
| (2) Acetone; $C_3H_6O$; [67-64-1] | *J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES: | PREPARED BY: |
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of lithium perchlorate in acetone at 25.00 °C, the solid phase being the anhydrous salt :

| g/100g sln | g/100cm$^3$ sln | g/100g solvent | mol % | mol dm$^{-3}$ | mol kg$^{-1}$ | satd sln density / g cm$^{-3}$ |
|---|---|---|---|---|---|---|
| 57.72 | 76.38 | 136.52 | 42.70[a] | 7.1785 | 12.83[a] | 1.3233 |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 cm$^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles. The salt was dried to constant wt. at 250°C in a current of air dried with $P_2O_5$, after ensuring that organic solvent was removed completely enough to avoid any danger of explosion. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:

Lithium perchlorate was prepared as described in ref. 1. Acetone refluxed with powdered KOH and purified by the bisulfite process. Its density was 0.7852 g cm$^{-3}$ at 25 °C; b.p. 56.16-56.51 °C.

ESTIMATED ERROR:

Precision in temp. was ±0.01°C .

REFERENCES:

1. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9] | Markarenko, B.K.; Mendzheritskii, E.A.; Sobolev, R.P.; Povarov, Yu.M.; Sereda, P.A. |
| (2) Tetrahydrofuran; $C_4H_8O$; [109-99-9] | *Elektrokhimiya* 1974, *10*, 355-8; **Soviet Electrochem. ( Engl. Transl.)*, 1974, *10*, 337-40. |
| VARIABLES: One temperature: 298.2 K | PREPARED BY: C.Y. Chan |

EXPERIMENTAL VALUES:

The solubility of lithium perchlorate in tetrahydrofuran at 25.0 °C is 2.2 mol $dm^{-3}$ and the specific conductivity of the saturated solution is $6.95 \times 10^{-3}$ S $cm^{-1}$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details of saturation method were given. The solubility was determined by polarographic analysis of the ions in the saturated solution. An MM34-04 conductivity meter was used to determine the specific conductivity of the solution. | The perchlorate was recrystallized twice from aqueous solution and dried under vacuum at 150-160 °C for 20-25 hours. Tetrahydrofuran was dried for 5-7 days with metallic lithium. The saturated solution contained not more than 0.02 % water. Sources of starting materials not given. |
| | ESTIMATED ERROR: Precision in temp. was ±0.1 °C . |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) 1,1'-oxybis-ethane ( *diethyl ether* ); $C_4H_{10}O$; [60-29-7] | Willard, H.H.; Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of lithium perchlorate in *diethyl ether* at 25.00 °C , the solid phase being the anhydrous salt:

| g/100g sln | g/100 $cm^3$ sln | g/100 g solvent | mol % | mol $dm^{-3}$ | mol $kg^{-1}$ | satd sln density / g $cm^{-3}$ |
|---|---|---|---|---|---|---|
| 53.21 | 64.47 | 113.72 | 44.21 | 6.059[a] | 10.69 [a] | 1.2116 |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles. The salt was dried to constant wt. at 250°C in a current of air dried with $P_2O_5$, after ensuring that organic solvent was removed completely enough to avoid any danger of explosion. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:

Lithium perchlorate was prepared as described in ref. 1. Ether was purified by refluxing with $P_2O_5$ and fractional distillation. The density of the fraction used was 0.70817 g $cm^{-3}$ at 25 °C.

ESTIMATED ERROR:

Precision in temp. was ±0.01°C .

REFERENCES:

1. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Willard, H.H.; Smith, G.F. |
| (2) Ethyl acetate; $C_4H_8O_2$; [141-78-6] | *J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES: | PREPARED BY: |
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of lithium perchlorate in ethyl acetate at 25.00°C, the solid phase being the anhydrous salt :

| g/100g sln | g/100cm$^3$ sln | g/100g solvent | mol % | mol dm$^{-3}$ | mol kg$^{-1}$ | satd sln density / g cm$^{-3}$ |
|---|---|---|---|---|---|---|
| 48.75 | 63.40 | 95.12 | 44.06[a] | 5.958 | 8.941[a] | 1.3005 |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 cm$^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles.

The salt was dried to constant wt. at 250°C in a current of air dried with $P_2O_5$, after ensuring that organic solvent was removed completely enough to avoid any danger of explosion.. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:

Lithium perchlorate was prepared as described in ref. 1. Ethyl acetate was purified by refluxing $P_2O_5$ and fractional distillation. Its density at 25°C was 0.8945 g cm$^{-3}$; b.p. 77.14 - 77.16 °C.

ESTIMATED ERROR:

Precision in temp. was ±0.01°C .

REFERENCES:

1. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9] | Sakk, Zh.G.; Rosolovskii, V.Ya. |
| (2) Hydrazine; $N_2H_4$; [302-01-2] | *Zh. Neorg. Khim.* 1972, *17*, 1783-4; **Russ. J. Inorg. Chem. (Engl. Transl.)* 1972, *17*, 927-8. |
| VARIABLES: | PREPARED BY: |
| One temperature: 298.2 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of lithium perchlorate in hydrazine at 25.0 °C was reported as 54.4 g(1)/100 g(2). The corresponding mol % and molality values are 14.08 % and 5.11 mol $kg^{-1}$, respectively (calculated by compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

4-6 g of the salt and 8-11 $cm^3$ of hydrazine were thermostated at 25°C for 7-8h with continuous stirring in a vessel isolated from atmospheric moisture. Samples for analysis were removed by withdrawing solution and part of the solid phase into another vessel fitted with a porosity no.4 filter at reduced pressure. After separating the phases, the solution was analysed for hydrazine using the procedure given in ref. 1.
Replicate solubility determinations were carried out.

SOURCE AND PURITY OF MATERIALS:

The methods of purification of the perchlorate and of the preparation of anhydrous hydrazine were as described in ref. 1.
Salt purity was about 99.5-99.9 %.

ESTIMATED ERROR:

Error in soly value was 0.4 %.

Precision in temp. was ±0.1 °C .

REFERENCES:

1. Rosolovskii, V.Ya.; Sakk, Zh.G. *Zh. Neorg. Khim.* 1970, *15*, 2262.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9] | Rosolovskii, V.Ya.; Sakk, Zh.G. |
| (2) Hydrazine; $N_2H_4$; [302-01-2] | *Zh. Neorg. Khim.* 1970, *15*, 2262-4; **Russ. J. Inorg. Chem. (Engl. Transl.)* 1970, *15*, 1169-70. |
| VARIABLES: | PREPARED BY: |
| One temperature: 273.2 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of lithium perchlorate in hydrazine at 0.0 °C was reported as 47.1 g(1)/100 g(2). The corresponding mol % and molality values are 12.4 % and 4.43 mol $kg^{-1}$, respectively (calculated by compiler).
The solid phase was $LiClO_4.2N_2H_4$ .

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
A solution of the perchlorate in hydrazine in the presence of the solid phase was stirred continuously for 2h in a thermostat at 0.0 °C. The liquid and solid phases were separated and analysed for perchlorate by precipitation as nitron perchlorate. Hydrazine in both phases was analysed by titration with standard iodine solution in the presence of sodium bicarbonate in excess.

SOURCE AND PURITY OF MATERIALS:
$LiClO_4$ was obtained by reacting 70 % $HClO_4$ (aq.) with $Li_2CO_3$, recrystallized twice from water and dried in a vacuum at 200 - 250 °C to constant weight. The hydrazine was 99.5 - 99.8 % pure.

ESTIMATED ERROR:
Precision in temp. was ±0.1 °C .
Soly precision not stated.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9] | Rosolovskii, V.Ya.; Sakk, Zh.G. |
| (2) Perchloric acid; $HClO_4$; [7601-90-3] | *Zh. Neorg. Khim.* 1968, *13*, 1115-8; **Russ. J. Inorg. Chem. (Engl. Transl.)* 1968, *13*, 582-4. |
| VARIABLES: One temperature: 273.2 K | PREPARED BY: C.Y. Chan |

EXPERIMENTAL VALUES:

Composition of saturated solution of lithium perchlorate in perchloric acid at 0 °C : (the solid phase was presumably the anhydrous salt)

| | mass % | | |
|---|---|---|---|
| | $LiClO_4$ | $HClO_4$ | |
| | 0.100 | 99.36 | The mean value of the solubility |
| | 0.104 | 99.37 | of $LiClO_4$ in perchloric acid is |
| | 0.101 | 99.38 | 0.100 mol %, or 0.018 mol $kg^{-1}$ |
| | 0.118 | 99.34 | (compiler). |
| Mean[a] : | 0.106 | 99.36 | |
| Std. dev.[a]: | 0.008 | 0.02 | ( [a] compiler's calculations ). |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Mixtures of 5-6 g of the perchlorate with 10-12 g of the acid were kept in a thermostat at 0°C (melting ice) for 10-15h with continuous stirring in tubes isolated from atmospheric moisture. After equilibrium has been attained, solid and liquid phases were separated on a glass filter. $ClO_4$ was determined gravimetrically as nitron perchlorate. Acid concentrations were determined by acid-base titration. $LiClO_4$ concentration was determined by dissolving a weighed sample of the satd solution (5-8g) in water and then evaporating to dryness. The dry residue was then dissolved in conc. $H_2SO_4$, the sln evaporated to dryness, and the resulting sulfate heated to constant weight at 600 °C.

SOURCE AND PURITY OF MATERIALS:

$LiClO_4$ was obtained by reacting 70 % $HClO_4$ (aq.) with $LiCO_3$, recrystallized twice from water and dried in a vacuum at 200 - 250 °C to constant weight. Analysis: Li 6.62 %, $ClO_4$ 92.69 %.
Anhyd. $HClO_4$ was distilled from a mixture of oleum and perchloric acid dihydrate at 100 °C under vacuum (ref.1). Acid purity was 99.6-100.2 % w/w as analysed.

ESTIMATED ERROR:

Not stated.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Hydrogen peroxide; $H_2O_2$; [7722-84-1] | Titova, K.V.; Kolmakova, E.I.; Rosolovskii, V.Ya.<br><br>*Zh. Neorg. Khim.* 1986, *31*, 3213-5; **Russ. J. Inorg. Chem. ( Engl. Transl. )* 1986, *31*, 1846-7. |
| VARIABLES:<br>One temperature: 273 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

The solubility[a] of lithium perchlorate in hydrogen peroxide at 0 °C :

| g(1)/ 100 g(2) | mass % | mol % | molality/ mol $kg^{-1}$ |
|---|---|---|---|
| 62.3 | 38.4 | 16.61 | 5.86 |

[a] Mass%, mol% and molality values calculated by compiler.
The solid phase was an unstable solvate.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details of saturation method was given. Solubility equilibrium was established in 1-1.5 h. The concencentration of the solutions did not change noticeably during the next 3h but after that slow decomposition of peroxide began. The concentration of perchlorate in the satd solution was determined gravimetrically by precipitation as nitron perchlorate. The solvated lithium perchlorate solid phase was too unstable for its composition to be determined. | Sources not given.<br>The $H_2O_2$ was 99.8% ± 0.2% pure.<br>No information on purity of salt. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9]<br>(2) Acetonitrile; $C_2H_3N$; [75-05-8] | Keller, R.; Foster, J.N.; Hansen, F.F.; Muirhead, J.S.<br><br>*NASA Contract. Rep. CR-1425* 1969, Lewis Research Center, NASA, U.S.A. |
| VARIABLES:<br>One temperature: 298 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of lithium perchlorate in acetonitrile at 25 °C is 1.06 mol $dm^{-3}$ ( 1.40 mol $kg^{-1}$ ).
Density of the saturated solution is 0.868 g $cm^{-3}$ and its viscosity is $7.09 \times 10^{-4}$ N s $m^{-2}$ .

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Saturated solutions were prepared by adding an excess of solute to the solvent in flasks in a dry box, the flasks being then stoppered with glass stoppers. The neck of each flask was then enclosed in a polythene bag containing dry $N_2$. The flasks were then placed in a bath which was at a temperature well above the final sampling temperature for 1-2 days, the solutions being magnetically stirred. After that they were equilibrated to 25 °C for several days with occasional stirring. Samples of the supernatant liquid were analysed for Li by atomic absorption spectrophotometry. | Lithium perchlorate was 99.9 % pure ( Atomergic Chemicals Co. ) and dried at 90-120 °C under vacuum. Acetonitrile ( J.T. Baker anal. reagent ) was redistilled from its mixture with $P_2O_5$, and the final fraction contained 49 mg $dm^{-3}$ of water but no detectable organic impurities. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Tomkins, R.P.T.; Turner, P.J. |
| (2) Acetonitrile; $C_2H_3N$; [75-05-8] | *J. Chem. Eng. Data* 1975, *20*, 50-2. |
| VARIABLES: | PREPARED BY: |
| Temperature: 297.35 K - 323.32 K | C.Y. Chan |

EXPERIMENTAL VALUES:

| No. of determinations | t / °C | Solubility of $LiClO_4$ in acetonitrile: molality / mol $kg^{-1}$ | mol % |
|---|---|---|---|
| 3 | 24.20 ± 0.01 | 1.5419 | 5.953 |
| 3 | 29.16 ± 0.20 | 1.8632 | 7.106 |
| 3 | 32.80 ± 0.15 | 2.1402 | 8.076 |
| 3 | 36.33 ± 0.05 | 2.4615 | 9.178 |
| 3 | 40.12 ± 0.05 | 2.9548 | 10.818 |
| 4 | 41.94 ± 0.05 | 3.2253 | 11.693 |
| 4 | 45.67 ± 0.03 | 3.9402 | 13.923 |
| 3 | 48.28 ± 0.05 | 4.4614 | 15.480 |
| 5 | 49.50 ± 0.03 | 5.2365 | 17.694 |
| 4 | 49.41 ± 0.01 | 6.1452 | 20.146 |
| 2 | 50.17 ± 0.30 | 6.2239 | 20.35 |
| 2[b] | 48.85 ± 0.05 | 7.1865 | 22.78 |
| 1[b] | 41.21 | 7.5868 | 23.75 |
| 1[b] | 45.66 | 7.7932 | 24.24 |
| 1[b] | 45.43 | 8.4018 | 25.60 |

[b] Poor reproducibility and formation of addition compounds; apparent solubility values only.

METHOD/APPARATUS/PROCEDURE:

A cooling method was used in which the temperature at which crystallization occurred was recorded. Solid $LiClO_4$ and solvent were placed in a Pyrex test tube and stirred with a teflon-coated magnetic stirring bar. The temperature was measured with a calibrated mercury-in-glass thermometer graduated in 0.1 °C intervals. The whole unit was placed in a 250 mL Erlenmeyer flask which served as support and air-jacket. Supercooling of 2-5 °C was observed and corrected for by "back-extrapolation". To reduce supercooling to 1-2 °C, it was necessary to cool the solution until solid formed, then raise the temperature to 3-5 °C above the crystallization point and cycle the temperature 5 or 6 times over a short range to ensure the presence of nuclei in the solution. No details of analysis method was given.

| SOURCE AND PURITY OF MATERIALS: | ESTIMATED ERROR: |
|---|---|
| No details available. | Not stated, except for temp. as tabulated. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9] | Kenttamaa, J. |
| (2) Sulfinylbis-methane (*dimethyl sulphoxide, DMSO*); $C_2H_6OS$; [67-68-5] | *J. Suomen Chemist* 1960, *33B*, 179-82. |
| VARIABLES: Three temperatures: 298 K, 308 K, 318 K. | PREPARED BY: C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of lithium perchlorate in *DMSO* :

| t / °C | mol / 100g *DMSO* | molality / $kg^{-1}$ |
|---|---|---|
| 25 | 0.27 | 2.7 |
| 35 | 0.27 | 2.7 |
| 45 | 0.29 | 2.9 |

[a] The solid phase was the solvate $2LiClO_4.7(CH_3)_2SO$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
The solute and solvent were contained in glass-stoppered flasks immersed in a thermostat at 50 °C initially and the flasks were shaken from time to time. About two weeks were allowed for attainment of equilibrium, after which the temp. was lowered to the desired temp. and the flasks allowed to stand for another week. The solutions were analysed for lithium by flame photometry. All analyses were in duplicate or triplicate.

SOURCE AND PURITY OF MATERIALS:
DMSO of "practical quality" was purified by repeated crystallization. The final product used had a melting-point of 18.5 °C. The purity of the perchlorate was not specified. The salt was dried for a few days at a temperature "high enough" to remove any moisture.

ESTIMATED ERROR:
Precision in soly. : ± 5 %

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Tomkins, R.P.T.; Turner, P.J. |
| (2) Propionitrile; $C_3H_5N$; [107-12-0] | *J. Chem. Eng. Data* 1975, *20*, 50-2. |
| VARIABLES: | PREPARED BY: |
| Temperature: 230 K - 285 K | C.Y. Chan |

EXPERIMENTAL VALUES:

| No. of determinations | t / °C | Solubility[a] of $LiClO_4$ in propionitrile: molality / mol $kg^{-1}$ | mol % |
|---|---|---|---|
| 1 | -43 | 1.4148 | 7.23 |
| 4 | -21.3 ± 0.1 | 2.5088 | 12.141 |
| 4 | -12.1 ± 0.2 | 3.0093 | 14.218 |
| 4 | -0.5 ± 3.5 | 3.7315 | 17.049 |
| 3 | -3.9 ± 2.0 | 4.1544 | 18.62 |
| 2 | 2.1 ± 0.1 | 6.8443 | 27.378 |
| 2 | 12 ± 4 | 8.1469 | 30.97 |

[a] The solid phase was the anhydrous salt.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A cooling method was used in which the temperature at which crystallization occurred was recorded. 15-20 $cm^3$ samples were cooled with dry ice and methanol. The sample tube was vacuum-jacketted and contents stirred with a propeller through a silicone oil seal. Temperature was measured with a chromel-alumel thermocouple immersed in a silicone oil well and a Keithley 147 null detector used in conjunction with a Moseley X-Y recorder. The thermocouple emf was also read on a digital multimeter. Sample composition was changed over a small range by adding solvent from a weighed syringe through a septum cap on the end of a side arm of the sample tube.

SOURCE AND PURITY OF MATERIALS:

Not stated.

ESTIMATED ERROR:

Not stated, except for temperature precision as given above.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Water; $H_2O$; [7732-18-5]<br>(3) *Alcohols*:<br>(A) Methanol (*methyl alcohol*); $CH_4O$; [67-56-1]<br>(B) Ethanol (*ethyl alcohol*); $C_2H_6O$; [64-17-5]<br>(C) 1-Propanol (*n-propyl alcohol*); $C_3H_8O$; [71-23-8]<br>(D) 1-Butanol (*n-butyl alcohol*); $C_4H_{10}O$; [71-36-3]<br>(E) 2-Methyl-1-propanol (*iso-butyl alcohol*); $C_4H_{10}O$; [78-83-1] | Willard, H.H.; Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| **VARIABLES:**<br>One temperature: 298.15 K | **PREPARED BY:**<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of $LiClO_4.3H_2O$ in various *alcohols*[b] at 25.00°C :

| soly in : | methanol | ethanol | 1-propanol | 1-butanol | 2-methyl-1-propanol |
|---|---|---|---|---|---|
| g/100 g sln | 60.95 | 42.16 | 26.82 | 21.40 | 18.85 |
| g/100 $cm^{-3}$ sln | 69.61 | 43.18 | 25.07 | 19.435 | 16.75 |
| mol $dm^{-3}$ | 4.338 | 2.691 | 1.563 | 1.211 | 1.044 |
| mol $kg^{-1}$ [c] | 14.671 | 6.851 | 3.445 | 2.559 | 2.183 |
| satd sln density/g $cm^{-3}$ | 1.1420 | 1.0241 | 0.9349 | 0.9082 | 0.8887 |
| pure solvent density/g $cm^{-3}$ | 0.78705 | 0.78515 | 0.8026 | 0.8059 | 0.7981 |

[a] In terms of the anhydrous salt. The solid phase was not exactly specified; presumably, it was the trihydrate.

[b] More correctly, mixtures of alcohol and water from the trihydrate, (compiler). The alcohol-water solvent compositions were not determined, but could be calculated if it was assumed that for each mole of $LiClO_4$ dissolved 3 moles of water went into solution.

[c] Compiler's calculations.

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Water; $H_2O$; [7732-18-5]<br>(3) *Alcohols*:<br>(A) Methanol (*methyl alcohol*); $CH_4O$; [67-56-1]<br>(B) Ethanol (*ethyl alcohol*); $C_2H_6O$; [64-17-5]<br>(C) 1-Propanol (*n-propyl alcohol*); $C_3H_8O$; [71-23-8]<br>(D) 1-Butanol (*n-butyl alcohol*); $C_4H_{10}O$; [71-36-3]<br>(E) 2-Methyl-1-propanol (*iso-butyl alcohol*); $C_4H_{10}O$; [78-83-1] | Willard, H.H.; Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1923, *45*, 286-96. |

AUXILIARY INFORMATION (continued)

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln. were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles, making sure that organic solvent was completely removed before the salt was dried to constant weight at 250°C in a current of air dried with $P_2O_5$. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected. | Lithium perchlorate trihydrate was prepared as described in ref.1 . Alcohols were purified by refluxing with calcium and fractional distillation. |
| | ESTIMATED ERROR:<br>Precision in temp. was ±0.01°C. |
| | REFERENCES:<br>1. Willard, H.H.; Smith, G.F. *J. Am. Chem.Soc.* 1922, *44*, 2816. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Water; $H_2O$; [7732-18-5]<br>(3) Acetone; $C_3H_6O$; [67-64-1] | Willard, H.H.; Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of lithium perchlorate trihydrate in acetone at 25.00 °C , the solid phase being $LiClO_4 \cdot 3H_2O$ :

| g/100g sln | g/100 $cm^3$ sln | g/100 g solvent | mol $dm^{-3}$ | mol $kg^{-1}$ | satd sln density / g $cm^{-3}$ |
|---|---|---|---|---|---|
| 49.04 | 53.77 | 96.23 | 5.054[b] | 9.045[b] | 1.0965 |

[a] In terms of the anhydrous salt.
[b] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles. The salt was dried to constant wt. at 250°C in a current of air dried with $P_2O_5$, after ensuring that organic solvent was removed completely enough to avoid any danger of explosion. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:

Lithium perchlorate was prepared as described in ref. 1. Acetone refluxed with powdered KOH and purified by the bisulfite process. Its density was 0.7852 g $cm^{-3}$ at 25 °C; b.p. 56.16-56.51 °C.

ESTIMATED ERROR:

Precision in temp. was ±0.01°C .

REFERENCES:

1. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Water; $H_2O$; [7732-18-5]<br>(3) Ethyl acetate; $C_4H_8O_2$; [141-78-6] | Willard, H.H.; Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] lithium perchlorate trihydrate in ethyl acetate at 25.00°C, the solid phase being $LiClO_4.3H_2O$ :

| mass % | g/100 $cm^3$ sln | g/100 g solvent | mol $dm^{-3}$ | mol $kg^{-1}$ | satd sln density / g $cm^{-3}$ |
|---|---|---|---|---|---|
| 26.35 | 27.41 | 35.78 | 1.7085 | 3.363[b] | 1.0402 |

[a] In terms of the anhydrous salt.
[b] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles. The salt was dried to constant wt. at 250°C in a current of air dried with $P_2O_5$, after ensuring that organic solvent was removed completely enough to avoid any danger of explosion. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:
Lithium perchlorate was prepared as described in ref. 1. Ethyl acetate was purified by refluxing with $P_2O_5$ and fractional distillation. Its density at 25°C was 0.8945 g $cm^{-3}$ and its b.p. was 77.14 - 77.16 °C.

ESTIMATED ERROR:
Precision in temp. was ± 0.01°C

REFERENCES:
1. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Water; $H_2O$; [7732-18-5]<br>(3) 1,1'-oxybis-ethane ( *diethyl ether* ); $C_4H_{10}O$; [60-29-7] | Willard, H.H.; Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES:<br>One temperature: 298.15 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of lithium perchlorate trihydrate in *diethyl ether*[b] at 25.00 °C , the solid phase being $LiClO_4.3H_2O$ :

| g/100g sln | g/100 $cm^3$ sln | g/100 g solvent | mol $dm^{-3}$ | mol $kg^{-1}$ | satd sln density / g $cm^{-3}$ |
|---|---|---|---|---|---|
| 0.196 | 0.139 | 0.196 | 0.0131[b] | 0.0184[b] | 0.7091 |

[a] In terms of the anhydrous salt.
[b] Saturated with water from the trihydrate.
[c] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles.
The salt was dried to constant wt. at 250°C in a current of air dried with $P_2O_5$, after ensuring that organic solvent was removed completely enough to avoid any danger of explosion.
Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:
Lithium perchlorate was prepared as described in ref. 1. Ether was purified by refluxing with $P_2O_5$ and fractional distillation. The density of the fraction used was 0.70817 g $cm^{-3}$ at 25 °C.

ESTIMATED ERROR:
Precision in temp. was ±0.01°C .

REFERENCES:
1. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9]<br>(2) Water; $H_2O$; [7732-18-5]<br>(3) N,N-dimethylformamide; $C_3H_7NO$; [68-12-2] | Keller, R.; Foster, J.N.; Hansen, F.F.; Muirhead, J.S.<br><br>*NASA Contract. Rep. CR-1425* 1969, Lewis Research Center, NASA, U.S.A. |
| VARIABLES:<br>Two temperatures: 298 K and 333 K.<br>Water content. | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of lithium perchlorate in N,N-dimethylformamide containing trace concentrations of water at 25 °C and 60 °C :
( the solid phase was the anhydrous salt )

| $H_2O$ content / mg $dm^{-3}$ | $LiClO_4$ solubility / mol $dm^{-3}$ | |
|---|---|---|
| | 25 °C | 60 °C |
| 45 | 4.4 | 4.8 |
| 1000 | 3.5 | 4.9 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
Saturated solutions were prepared by adding an excess of solute to the solvent in flasks in a dry box, the flasks being then stoppered with glass stoppers. The neck of each flask was then enclosed in a polythene bag containing dry $N_2$. The flasks were then placed in a bath which was at a temperature well above the final sampling temperature for 1-2 days, the solutions being magnetically stirred. After that they were equilibrated to 25 °C for several days with occasional stirring. Samples of the supernatant liquid were analysed for Li by atomic absorption spectrophotometry.

SOURCE AND PURITY OF MATERIALS:
Lithium perchlorate was 99.9 % pure ( Atomergic Chemicals Co. ) and dried at 90-120 °C under vacuum. Dimethylformamide ( Matheson, Coleman and Bell, spectropure ), after pre-treatment with chromatographic grade molecular sieves for 6 hours, was distilled at 151 °C and at atmos. pressure. Final sample used contained 45 mg $dm^{-3}$ $H_2O$ and 35 mg $dm^{-3}$ impurities.

ESTIMATED ERROR:
Not stated.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9] | Keller, R.; Foster, J.N.; Hansen, F.F.; Muirhead, J.S. |
| (2) Water; $H_2O$; [7732-18-5] | *NASA Contract. Rep. CR-1425* 1969, Lewis Research Center, NASA, U.S.A. |
| (3) Propylene carbonate; $C_4H_6O_3$; [108-32-7] | |
| VARIABLES: | PREPARED BY: |
| Two temperatures: 298 K and 333 K. Water content. | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of lithium perchlorate in propylene carbonate containing trace concentrations of water at 25 °C and 60 °C :
( the solid phase was the anhydrous salt )

| $H_2O$ content / mg $dm^{-3}$ | $LiClO_4$ solubility / mol $dm^{-3}$ | |
|---|---|---|
| | 25 °C | 60 °C |
| 20 | 2.1 | 3.1 |
| 1000 | 3.1 | 3.1 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
Saturated solutions were prepared by adding an excess of solute to the solvent in flasks in a dry box, the flasks being then stoppered with glass stoppers. The neck of each flask was then enclosed in a polythene bag containing dry $N_2$. The flasks were then placed in a bath which was at a temperature well above the final sampling temperature for 1-2 days, the solutions being magnetically stirred. After that they were equilibrated to 25 °C for several days with occasional stirring. Samples of the supernatant liquid were analysed for Li by atomic absorption spectrophotometry.

SOURCE AND PURITY OF MATERIALS:
Lithium perchlorate was 99.9 % pure ( Atomergic Chemicals Co. ) and dried at 90-120 °C under vacuum. Propylene carbonate (Matheson, Coleman and Bell, spectro pure ) was fractionally distilled in the presence of $CaH_2$ and dry $N_2$. The sample used contained 20 mg $dm^{-3}$ $H_2O$ and less than 35 mg $dm^{-3}$ of organic impurities.

ESTIMATED ERROR:
Not stated.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Tarakanov, V.F. |
| (2) Acetamide; $C_2H_5NO$; [60-35-5] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1975, *144*, 92-4. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 298 K. | E.S. Gryzlova |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$CH_3CONH_2$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.50 | - | 9.223 | - | 5.640 | - | A |
| 3 | 36.55 | 14.60 | 10.40 | 7.48 | 7.033 | 5.060 | A |
| 6 | 37.98 | 49.72 | 18.97 | 44.74 | 29.02 | 68.43 | A + B |
| 8 | 35.43 | 53.99 | 18.15 | 49.83 | 31.48 | 86.39 | B |
| 11 | 32.96 | 59.42 | 17.82 | 57.86 | 40.66 | 132.0 | B + C |
| 15 | 24.43 | 66.59 | 12.38 | 60.76 | 25.57 | 125.5 | C |
| 17 | 20.35 | 74.35 | 10.97 | 72.17 | 36.09 | 237.5 | C + D |
| 20 | 10.35 | 81.53 | 5.045 | 71.58 | 11.98 | 170.0 | D |
| 22 | - | 84.24 | - | 61.98 | - | 90.49 | D |

[a] Values calculated by C.C. Ho;
[b] A = $LiClO_4.3H_2O$; B = $LiClO_4.2CH_3CONH_2$; C = $LiClO_4.4CH_3CONH_2$; D = $CH_3CONH_2$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details were given. | No details were given. |
| | ESTIMATED ERROR: |
| | Nothing specified. |
| | REFERENCES: |

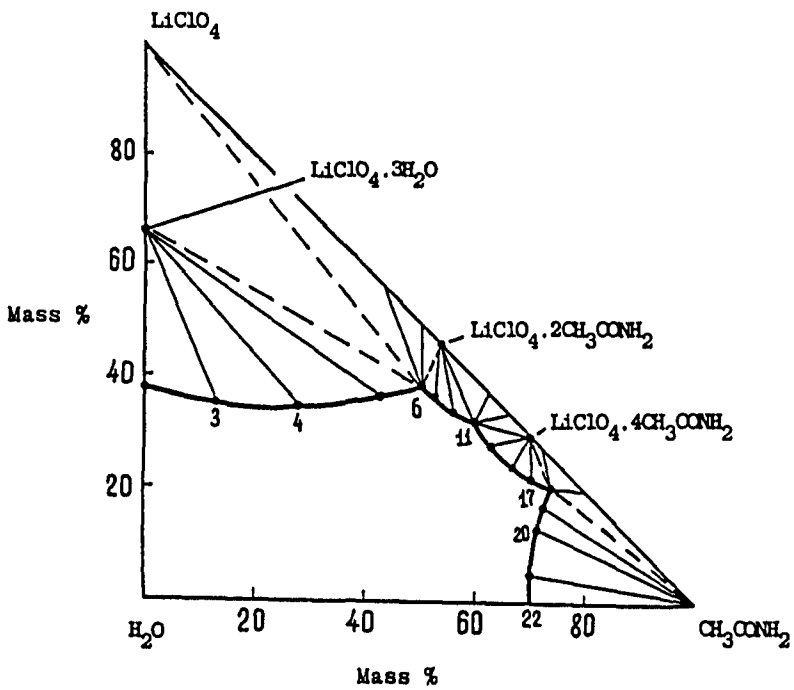
(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Tarakanov, V.F. |
| (2) Acetamide; $C_2H_5NO$; [60-35-5] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* |
| (3) Water; $H_2O$; [7732-18-5] | 1975, *144*, 92-4. |

EXPERIMENTAL VALUES: (continued)

COMMENTS/ADDITIONAL DATA:

The solubility isotherm (see Figure below) shows four branches of crystallization: lithium perchlorate crystallizes as trihydrate; lithium perchlorate diacetamide; lithium perchlorate tetraacetamide; and acetamide. $LiClO_4.2CH_3CONH_2$ and $LiClO_4.4CH_3CONH_2$ are very hygroscopic. The compounds are formed through the interaction between $Li^+$ cations and acetamide molecules.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) N(1'), N(1)-dimethylcarbamide (*dimethylurea*); $C_3H_8N_2O$; [1320-50-9]<br>(3) Water; $H_2O$; [7732-18-5] | Bestuzheva, M.M.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1979, *178*, 58-63. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>I.S. Bodnya |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$C_3H_8N_2O$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.46 | - | 9.209 | - | 5.630 | - | $LiClO_4.3H_2O$ |
| 2 | 37.01 | 1.63 | 9.222 | 0.490 | 5.669 | 0.301 | " |
| 3 | 34.78 | 12.49 | 9.627 | 4.175 | 6.200 | 2.688 | " |
| 4 | 35.61 | 17.09 | 10.61 | 6.149 | 7.076 | 4.101 | " |
| 5 | 36.79 | 22.93 | 12.17 | 9.157 | 8.585 | 6.461 | " |
| 6 | 25.40 | 47.19 | 10.40 | 23.33 | 8.710 | 19.54 | $C_3H_8N_2O$ |
| 7 | 22.09 | 41.33 | 7.670 | 17.33 | 5.676 | 12.82 | " |
| 8 | 17.91 | 33.06 | 5.156 | 11.49 | 3.433 | 7.653 | " |
| 9 | 3.53 | 25.28 | 0.777 | 6.717 | 0.466 | 4.030 | " |
| 10 | - | 23.55 | - | 5.925 | - | 3.496 | " |

[a] Values calculated by C.C. Ho.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Equilibrium was reached in 2-3 days. $ClO_4^-$ was determined gravimetrically by precipitating with nitron. (2) was determined by Kjeldahl's method. The densities and viscosities of the saturated solutions were determined. | Nothing specified<br><br>ESTIMATED ERROR:<br>Nothing specified.<br><br>REFERENCES: |

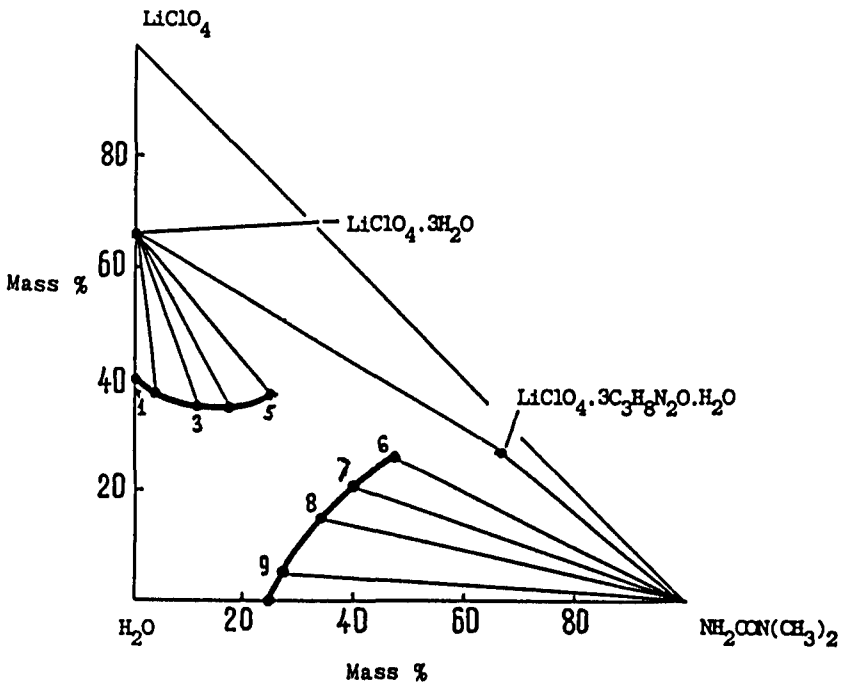
(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Bestuzheva, M.M. |
| (2) N(1), N(1)-dimethylcarbamide (*dimethylurea*); $C_3H_8N_2O$; [1320-50-9] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1979, *178*, 58-63. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

COMMENTS/ADDITIONAL DATA:

Addition of dimethylurea to lithium perchlorate causes some irregularity in the solubility: from 37.46% (point 1) to 34.78% (point 3). Further addition of dimethylurea increases the solubility of lithium perchlorate and this results in the complete dissolution of the solid phase. A new compound is presumably formed, i.e. $LiClO_4.3C_3H_8ON_2.H_2O$. The diagram below shows a probable region of formation of this compound.
The compound contains 29.9% $LiClO_4$; 64.58% $C_3H_8N_2O$ and 5.52% $H_2O$.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Hexamethylenetetramine; $C_6H_{12}N_4$; [100-97-0]<br>(3) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Kosheleva, N.I.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1975, *144*, 107-11. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>I.S. Bodnya |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$C_6H_{12}N_4$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | - | 46.50 | - | 10.05 | - | 6.200 | A |
| 2 | 4.60 | 46.70 | 1.40 | 10.82 | 0.888 | 6.840 | A |
| 3 | 6.24 | 48.10 | 2.00 | 11.69 | 1.285 | 7.514 | A |
| 4 | 6.48 | 45.10 | 1.98 | 10.48 | 1.258 | 6.644 | C |
| 5 | 9.45 | 41.35 | 2.85 | 9.470 | 1.805 | 5.995 | C |
| 6 | 12.23 | 37.26 | 3.610 | 8.346 | 2.276 | 5.262 | C |
| 7 | 15.96 | 33.85 | 4.721 | 7.599 | 2.989 | 4.811 | C |
| 8 | 18.64 | 35.92 | 5.932 | 8.675 | 3.856 | 5.639 | D |
| 9 | 18.60 | 34.54 | 5.785 | 8.152 | 3.731 | 5.258 | D |
| 10 | 20.53 | 27.82 | 5.922 | 6.090 | 3.736 | 3.842 | D |
| 11 | 21.75 | 24.62 | 6.090 | 5.232 | 3.812 | 3.275 | D |
| 12 | 25.25 | 20.06 | 6.947 | 4.189 | 4.340 | 2.616 | D |
| 13 | 26.45 | 17.06 | 7.091 | 3.471 | 4.401 | 2.154 | D |
| 14 | 30.22 | 13.26 | 8.079 | 2.690 | 5.026 | 1.674 | D |
| 15 | 32.82 | 10.38 | 8.726 | 2.094 | 5.431 | 1.304 | D |

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Equilibrium was reached in 1 day. $ClO_4^-$ was determined gravimetrically by nitron precipitation. (2) was determined by Kjeldahl's method. The compositions of the solid phases were determined by chemical analyses and by Schreinemakers' method of residues. The densities and viscosities of the saturated solutions were measured. | No details were given.<br><br>ESTIMATED ERROR:<br>Nothing specified.<br><br>REFERENCES: |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Karnaukhov, A.S.; Kosheleva, N.I. |
| (2) Hexamethylenetetramine; $C_6H_{12}N_4$; [100-97-0] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1975, *144*, 107-11. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

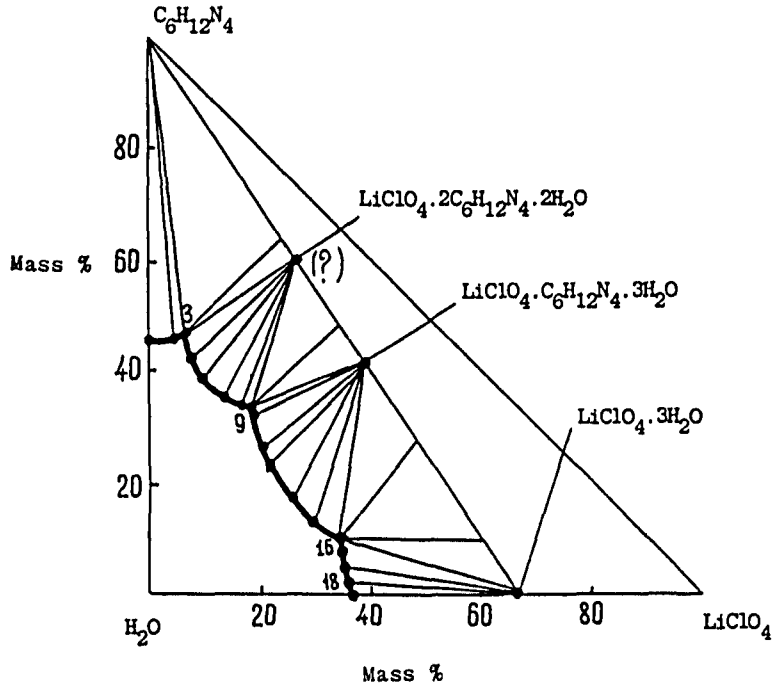
| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 16 | 33.26 | 10.32 | 8.886 | 2.093 | 5.541 | 1.305 | B + D |
| 17 | 33.75 | 6.95 | 8.671 | 1.355 | 5.350 | 0.836 | " B |
| 18 | 36.55 | 3.11 | 9.247 | 0.597 | 5.694 | 0.368 | B |
| 19 | 37.55 | - | 9.241 | - | 5.652 | - | B |

[a] Values calculated by C.C. Ho;

[b] A = $C_6H_{12}N_4$; B = $LiClO_4.3H_2O$; C = $LiClO_4.2C_6H_{12}N_4.2H_2O$; D = $LiClO_4.C_6H_{12}N_4.3H_2O$.

COMMENTS/ADDITIONAL DATA:

The solubility isotherm (see Figure below) shows four branches of crystallization: the crystallization of hexamethylenetetramine (point 1-2); the crystallization of the compound $LiClO_4.2C_6H_{12}N_4.2H_2O$ which is congruently soluble (point 4 through 7); the crystallization of the double complex compound $LiClO_4.C_6H_{12}N_4.3H_2O$ (points 9 through 15); the branch from point 17 to point 19 indicates the crystallization of lithium perchlorate trihydrate. Hexamethylenetetramine is salted out.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1). Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Methyl acetate; $C_3H_6O_2$; [79-20-9]<br>(3) Propylene carbonate; $C_4H_6O_3$ [108-32-7] | Il'in, K.K.; Demakhin, A.G.<br><br>*Zh. Neorg. Khim.* 1989, *34*, 780-2; **Russ. J. Inorg. Chem. (Engl. Transl.)* 1989, *34*, 436-8. |
| VARIABLES:<br>Temperature: 283.2 K - 323.2 K.<br>Composition. | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-methyl acetate-propylene carbonate at various temperatures ( the solid phase was the anhydrous salt over the whole temperature range studied for both the pure and mixed solvents ):

| | Liquid phase composition | | | | | |
|---|---|---|---|---|---|---|
| $t$ / °C | mass % | | | mol %[a] | | |
| | (1) | (2) | (3) | (1) | (2) | (3) |
| 10.0 | 47.4 | 0.0 | 52.6 | 46.4 | 0.0 | 53.6 |
| | 48.0 | 10.4 | 41.6 | 45.2 | 14.1 | 40.8 |
| | 49.0 | 20.4 | 30.6 | 44.5 | 26.6 | 28.9 |
| | 49.9 | 30.1 | 20.0 | 43.8 | 37.9 | 18.3 |
| | 50.9 | 39.3 | 9.8 | 43.3 | 48.0 | 8.7 |
| | 52.5 | 47.5 | 0.0 | 43.5 | 56.5 | 0.0 |
| 20.0 | 47.8 | 0.0 | 52.2 | 46.8 | 0.0 | 53.2 |
| | 48.5 | 10.3 | 41.2 | 45.7 | 13.9 | 40.4 |
| | 49.4 | 20.2 | 30.4 | 44.9 | 26.4 | 28.8 |
| | 50.5 | 29.7 | 19.8 | 44.4 | 37.5 | 18.1 |
| | 51.5 | 38.8 | 9.7 | 43.9 | 47.5 | 8.6 |
| | 53.5 | 46.5 | 0.0 | 44.5 | 55.5 | 0.0 |

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Special vessels, reported in ref.(1) were used in the preparation of the satd solutions. The salt and the solvents were placed in the vessels under conditions which ensured that atmospheric moisture was excluded. The vessels were equilibrated in an U-10 ultrathermostat which was maintained to within ±0.1 K of the required temperature. Equilibrium was reached after continuous stirring for 7-8 h and was reached both from | " Chemically pure " $LiClO_4$ was was dried in a vacuum at 160 °C for 3 h. "Chem. pure" propylene carbonate was vacuum-distilled and "pure" grade methyl acetate was purified as recommended in ref. (3). The solvents were dried using zeolites of type NaA. Water content in propylene carbonate was 0.015 mass % and in methyl acetate 0.014 mass %. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Il'in, K.K.; Demakhin, A.G. |
| (2) Methyl acetate; $C_3H_6O_2$; [79-20-9] | *Zh. Neorg. Khim.* 1989, *34*, 780-2; **Russ. J. Inorg. Chem. (Engl. Transl.)* 1989, *34*, 436-8. |
| (3) Propylene carbonate; $C_4H_6O_3$ [108-32-7] | |

EXPERIMENTAL VALUES: (continued)

| | Liquid phase composition | | | | | |
|---|---|---|---|---|---|---|
| t / °C | mass % | | | mol %[a] | | |
| | (1) | (2) | (3) | (1) | (2) | (3) |
| 25.0 | 48.2 | 0.0 | 51.8 | 47.2 | 0.0 | 52.8 |
| | 48.4 | 5.2 | 46.4 | 46.4 | 7.2 | 46.4 |
| | 48.7 | 10.3 | 41.0 | 45.8 | 13.9 | 40.2 |
| | 49.1 | 15.3 | 35.6 | 45.4 | 20.3 | 34.3 |
| | 49.6 | 20.2 | 30.2 | 45.1 | 26.4 | 28.6 |
| | 50.7 | 29.6 | 19.7 | 44.6 | 37.4 | 18.0 |
| | 51.7 | 38.6 | 9.7 | 44.1 | 47.3 | 8.6 |
| | 53.5 | 46.5 | 0.0 | 44.5 | 55.5 | 0.0 |
| 30.0 | 48.4 | 0.0 | 51.6 | 47.4 | 0.0 | 52.6 |
| | 49.0 | 10.2 | 40.8 | 46.2 | 13.8 | 40.0 |
| | 50.0 | 20.0 | 30.0 | 45.5 | 26.1 | 28.4 |
| | 51.1 | 29.3 | 19.6 | 45.0 | 37.0. | 18.0 |
| | 52.1 | 38.3 | 9.6 | 44.5 | 47.0 | 8.5 |
| | 53.9 | 46.1 | 0.0 | 44.9 | 55.1 | 0.0 |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: (continued)

supersaturated and unsaturated sln. $LiClO_4$ concentration was determined by gravimetric precipitation with nitron (2). Each reported soly value was the mean of 5 determinations. The compositions of solvent mixtures were checked by measuring their refractive indices and comparison with those previously measured for known mixtures of the solvents.

ESTIMATED ERROR:

"Relative error" in soly values did not exceed ± 0.5 %.

Temperature precision ± 0.1 K.

REFERENCES:

(1) Frontas'ev, V.P.; Sakharova, Yu G.; Sakharova, N.N. *Zh. Neorg. Khim.* 1965, *10*, 1816.

(2) Schumacher, J.C. *Perchlorates* (Russ. Transl.), Goskhimizdat, Moscow, 1963, 274.

(3) Keil, B. *Laboratorni Technika Organicke Chemie* (Russ. Transl.), Izd. Mir., Moscow, 1966.

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Il'in, K.K.; Demakhin, A.G. |
| (2) Methyl acetate; $C_3H_6O_2$; [79-20-9] | *Zh. Neorg. Khim.* 1989, *34*, 780-2; **Russ. J. Inorg. Chem. (Engl. Transl.)* 1989, *34*, 436-8. |
| (3) Propylene carbonate; $C_4H_6O_3$ [108-32-7] | |

EXPERIMENTAL VALUES: (continued)

| | Liquid phase composition | | | | | |
|---|---|---|---|---|---|---|
| $t$ / °C | mass % | | | mol %[a] | | |
| | (1) | (2) | (3) | (1) | (2) | (3) |
| 40.0 | 49.0 | 0.0 | 51.0 | 48.0 | 0.0 | 52.0 |
| | 49.5 | 10.1 | 40.4 | 46.7 | 13.7 | 39.7 |
| | 50.6 | 19.8 | 29.6 | 46.0 | 25.9 | 28.1 |
| | 51.7 | 29.0 | 19.3 | 45.6 | 36.7 | 17.7 |
| | 52.6 | 37.9 | 9.5 | 45.0 | 46.5 | 8.5 |
| | 54.3 | 45.7 | 0.0 | 45.3 | 54.7 | 0.0 |
| 50.0 | 49.5 | 0.0 | 50.5 | 48.5 | 0.0 | 51.5 |
| | 50.1 | 10.0 | 39.9 | 47.2 | 13.5 | 39.2 |
| | 51.3 | 19.5 | 29.2 | 46.7 | 25.5 | 27.7 |
| | 52.3 | 28.6 | 19.1 | 46.2 | 36.3 | 17.6 |
| | 53.2 | 37.4 | 9.4 | 45.6 | 46.0 | 8.4 |
| | 54.9 | 45.1 | 0.0 | 45.9 | 54.1 | 0.0 |

[a] Compiler's calculations.

COMMENTS AND/OR ADDITIONAL DATA

At all temperatures, the solubility of lithium perchlorate in methyl acetate is higher than that in propylene carbonate which is the more polar solvent. The solubility of the salt increases linearly with temperature and may be approximated by an equation of the form,

$\log s = A + B/T$, where $s$ is the solubility of the salt in mass %, $T$ the absolute temperature, and A and B are empirical constants, "least squares best-fit" values of which are given below for pure methyl acetate, pure propylene carbonate and mixtures of the two solvents.

| Solvent mass % of methyl acetate : | 0.00 | 20.00 | 40.00 | 60.00 | 80.00 | 100.00 |
|---|---|---|---|---|---|---|
| A : | 1.8308 | 1.8329 | 1.8490 | 1.8606 | 1.8579 | 1.8752 |
| -B / K : | 44.113 | 43.152 | 45.357 | 46.165 | 42.840 | 43.765 |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Andronova, N.P.; Bogomolova, V.V.; Gulyakova, N.I. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1969, *66*, 57-61. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298 K. | E.S. Gryzlova |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$NaClO_4$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.62 | - | 9.266 | - | 5.669 | - | $LiClO_4.3H_2O$ |
| 2 | 36.82 | 0.95 | 9.088 | 0.204 | 5.561 | 0.125 | " |
| 3 | 36.31 | 2.00 | 9.024 | 0.432 | 5.532 | 0.265 | " |
| 4 | 35.91 | 4.00 | 9.108 | 0.882 | 5.617 | 0.544 | " |
| 5 | 31.26 | 11.92 | 8.288 | 2.746 | 5.171 | 1.713 | " |
| 6 | 28.72 | 16.74 | 7.861 | 3.981 | 4.950 | 2.507 | " |
| 7 | 21.84 | 29.09 | 6.483 | 7.503 | 4.183 | 4.842 | " |
| 8 | 13.14 | 43.90 | 4.308 | 12.51 | 2.875 | 8.346 | " |
| 9 | 7.23 | 57.59 | 2.728 | 18.88 | 1.932 | 13.37 | " |
| 10 | 6.23 | 60.19 | 2.426 | 20.36 | 1.744 | 14.64 | $LiClO_4.3H_2O$ + $NaClO_4.H_2O$ |
| 11 | 6.10 | 59.86 | 2.35 | 20.07 | 1.684 | 14.36 | " |
| 12 | 5.96 | 60.19 | 2.31 | 20.26 | 1.655 | 14.52 | " |
| 13 | 5.76 | 60.21 | 2.22 | 20.20 | 1.591 | 14.45 | $NaClO_4.H_2O$ |
| 14 | 0.38 | 66.99 | 0.15 | 23.16 | 0.109 | 16.77 | " |
| 15 | - | 67.89 | - | 23.73 | - | 17.27 | " |

[a] Values calculated by C.C. Ho.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Equilibrium was reached after 6-8 days. $Li^+$ was determined by the periodate method; $ClO_4^-$ by precipitating with nitron. The compositions of the solid phases were determined by Schreinemakers' method of residues. The densities, viscosities and refractive indexes of the saturated solutions were measured. | No details were given. |
| | **ESTIMATED ERROR:** Nothing specified. |
| | **REFERENCES:** |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Andronova, N.P. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. zap. Yarosl. gos. ped. Inst.* 1973, *120*, 43-6. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 323 K | R.A. Vasina |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$NaClO_4$-$H_2O$ at 323 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 45.28 | - | 12.29 | - | 7.778 | - | $LiClO_4.3H_2O$ |
| 40.63 | 7.37 | 11.47 | 1.81 | 7.344 | 1.158 | " |
| 39.05 | 10.60 | 11.30 | 2.67 | 7.290 | 1.719 | " |
| 31.58 | 22.06 | 9.73 | 5.91 | 6.403 | 3.886 | " |
| 25.78 | 31.62 | 8.46 | 9.01 | 5.688 | 6.062 | " |
| 21.43 | 40.55 | 7.62 | 12.53 | 5.298 | 8.711 | " |
| 17.01 | 49.98 | 6.66 | 17.01 | 4.844 | 12.366 | " |
| 13.45 | 56.91 | 5.65 | 20.78 | 4.265 | 15.681 | " |
| 13.29 | 57.72 | 5.66 | 21.37 | 4.309 | 16.261 | $LiClO_4.3H_2O$ + $NaClO_4$ |
| 13.28 | 57.75 | 5.66 | 21.39 | 4.309 | 16.281 | " |
| 13.28 | 57.74 | 5.66 | 21.39 | 4.307 | 16.272 | " |
| 13.28 | 57.83 | 5.67 | 21.46 | 4.321 | 16.349 | " |
| 10.42 | 60.62 | 4.45 | 22.50 | 3.382 | 17.096 | $NaClO_4$ |
| 9.75 | 61.83 | 4.22 | 23.23 | 3.225 | 17.768 | " |
| 5.44 | 66.87 | 2.40 | 25.59 | 1.847 | 19.723 | " |
| 3.26 | 70.07 | 1.47 | 27.47 | 1.149 | 21.458 | $NaClO_4.H_2O$ |
| - | 73.75 | - | 29.25 | - | 22.946 | " |

[a] Compiler's calculation

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Details of saturation were not given. The composition of solid phases was determined graphically by Schreinemaker's method of "residues". | Not stated. |
| | ESTIMATED ERROR: Not stated. |

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Potassium perchlorate; $KClO_4$; [7778-74-7]<br>(3) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Voronina, T.N.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1969, *66*, 132-7. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$KClO_4$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | - | 2.05 | - | 0.2714 | - | 0.1511 | $KClO_4$ |
| 2 | 2.29 | 1.27 | 0.400 | 0.170 | 0.223 | 0.0950 | " |
| 3 | 6.41 | 0.70 | 1.154 | 0.0968 | 0.649 | 0.0544 | " |
| 4 | 11.84 | 0.68 | 2.238 | 0.0987 | 1.272 | 0.0561 | " |
| 5 | 14.83 | 0.67 | 2.883 | 0.1000 | 1.650 | 0.0572 | " |
| 6 | 17.46 | 0.64 | 3.481 | 0.0980 | 2.004 | 0.0564 | " |
| 7 | 26.95 | 0.50 | 5.913 | 0.0842 | 3.492 | 0.0497 | " |
| 8 | 23.30 | 0.40 | 4.914 | 0.0648 | 2.870 | 0.0378 | " |
| 9 | 26.12 | 0.37 | 5.672 | 0.0617 | 3.340 | 0.0363 | " |
| 10 | 31.67 | 0.18 | 7.293 | 0.0318 | 4.368 | 0.0191 | " |
| 11 | 37.50 | 0.19 | 9.245 | 0.0360 | 5.657 | 0.0220 | $KClO_4$ + $LiClO_4.3H_2O$ |
| 12 | 37.48 | 0.197 | 9.239 | 0.0373 | 5.653 | 0.0228 | " |
| 13 | 37.49 | 0.19 | 9.242 | 0.0360 | 5.654 | 0.0220 | " |
| 14 | 37.52 | 0.19 | 9.252 | 0.0360 | 5.662 | 0.0220 | " |
| 15 | 37.70 | - | 9.295 | - | 5.688 | - | $LiClO_4.3H_2O$ |

[a] Values calculated by C.C. Ho.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Equilibrium was reached after 3-4 days. The salt concns were calculated using the data of chemical analysis. $K^+$ was determined gravimetrically by the tetraphenylborate method; $Li^+$ by the volumetric periodate method; $ClO_4^-$ by difference. The compositions of the solid phases were determined by Schreinemakers' method of residues and then confirmed under a microscope. The densities, viscosities of the saturated solutions were measured. | Lithium and potassium perchlorates were reagent grade. They were further purified by recrystallization. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]<br>(3) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Ganina, G.I.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 101-6 |
| VARIABLES:<br>Temperature: 298 K<br>Composition | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system : $NH_4ClO_4$-$LiClO_4$-$H_2O$ at 25°C

| Point | Liquid phase composition: mass % (1) | mass % (2) | mol %[a] (1) | mol %[a] (2) | molality[a]/mol kg$^{-1}$ (1) | molality[a]/mol kg$^{-1}$ (2) | Solid phase |
|---|---|---|---|---|---|---|---|
| 1 | - | 19.96 | - | 3.683 | - | 2.123 | $NH_4ClO_4$ |
| 2 | 1.39 | 18.82 | 0.284 | 3.481 | 0.164 | 2.008 | " |
| 3 | 2.59 | 18.22 | 0.532 | 3.390 | 0.307 | 1.958 | " |
| 4 | 5.72 | 15.23 | 1.176 | 2.836 | 0.680 | 1.640 | " |
| 5 | 8.54 | 12.64 | 1.759 | 2.358 | 1.018 | 1.365 | " |
| 6 | 12.24 | 9.36 | 2.530 | 1.752 | 1.467 | 1.016 | " |
| 7 | 16.41 | 7.34 | 3.467 | 1.404 | 2.023 | 0.819 | " |
| 8 | 20.82 | 5.89 | 4.536 | 1.162 | 2.670 | 0.684 | " |
| 9 | 25.98 | 4.49 | 5.896 | 0.923 | 3.512 | 0.550 | " |
| 10 | 32.68 | 3.61 | 7.928 | 0.793 | 4.821 | 0.482 | " |
| 11 | 35.95 | 3.24 | 9.033 | 0.737 | 5.557 | 0.453 | $NH_4ClO_4$ + $LiClO_4.3H_2O$ |
| 12 | 35.74 | 2.93 | 8.922 | 0.662 | 5.478 | 0.407 | " |
| 13 | 35.79 | 2.95 | 8.942 | 0.667 | 5.491 | 0.410 | " |
| 14 | 35.61 | 2.94 | 8.877 | 0.664 | 5.447 | 0.407 | " |
| 15 | 35.76 | 2.97 | 8.934 | 0.672 | 5.486 | 0.413 | " |
| 16 | 35.43 | 2.94 | 8.812 | 0.662 | 5.404 | 0.406 | " |
| 17 | 36.18 | 1.99 | 8.975 | 0.447 | 5.500 | 0.274 | $LiClO_4.3H_2O$ |
| 18 | 36.49 | 1.25 | 9.003 | 0.279 | 5.509 | 0.171 | " |
| 19 | 37.30 | - | 9.152 | - | 5.592 | - | " |

[a] Editors' calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal solubility. Details of saturation method were not given. $NH_4^+$ was determined by distilling off $NH_3$ into 4% $H_3BO_3$ solution and then titrating with 0.2 mol dm$^{-3}$ $H_2SO_4$ ; $ClO_4^-$ was determined by nitron precipitation ; $Li^+$ by difference. The compositions of the solid phases were determined by Schreinemakers' method of "residues". The densities, viscosities and electric conductivities of the saturated solutions were measured. | Not stated. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES:<br>None. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Aravamudan, G. |
| (2) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | *Ind. J. Chem.* 1964, *43*, 475-507. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 303.15 K. | C.Y. Chan |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$NH_4ClO_4$-$H_2O$ at 303.15 K :

| Solution composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 38.87 | - | 9.72 | - | 5.977 | - | $LiClO_4.3H_2O$ |
| 38.23 | 1.457 | 9.66 | 0.3334 | 5.958 | 0.2056 | " |
| 37.85 | 2.591 | 9.66 | 0.5986 | 5.973 | 0.3703 | $LiClO_4.3H_2O$ + $NH_4ClO_4$ |
| 36.22 | 2.880 | 9.09 | 0.6545 | 5.590 | 0.4025 | $NH_4ClO_4$ |
| 30.22 | 4.228 | 7.175 | 0.9090 | 4.333 | 0.5490 | " |
| 22.99 | 6.563 | 5.167 | 1.336 | 3.067 | 0.7929 | " |
| 17.24 | 9.046 | 3.742 | 1.778 | 2.198 | 1.045 | " |
| 15.42 | 10.20 | 3.324 | 1.991 | 1.949 | 1.117 | " |
| 10.50 | 13.31 | 2.222 | 2.551 | 1.295 | 1.487 | " |
| 4.96 | 17.55 | 1.037 | 3.321 | 0.602 | 1.928 | " |
| - | 22.00 | - | 4.146 | - | 2.401 | " |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions were variously prepared as follows:
(i) addition of $LiClO_4.3H_2O$ crystals to satd $NH_4ClO_4$ solutions at 30°C;
(ii) from admixture of water and crystals of both salts;
(iii) slow evaporation of solutions containing both salts at 30°C until slightly beyond the point of incipient crystallization.
They were then agitated continuously at 30.00°C until equilibrium was attained, generally after less than 24h. Saturated solutions were then separated from the solid phases, whose compositions were ascertained using Schreinemakers' method. Ammonium ion was analysed using the formalin method (ref.1) and lithium by weighing as sulphate after volatilization of ammonia and perchorate using sulphuric acid.

SOURCE AND PURITY OF MATERIALS:

$LiClO_4.3H_2O$ was prepared from dilute $HClO_4$ and $Li_2CO_3$ and $NH_4ClO_4$ from ammonia and $HClO_4$. No details of purity of starting materials were given.

ESTIMATED ERROR:

Temp. precision: ± 0.02 K.

REFERENCES:

1. Stockdale, D. *Analyst* 1959, *84*, 667.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Guseva, A.D.; Lepeshkov, I.N. |
| (2) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 3-22 |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 308 K | I.S. Bodnya ; N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system : $NH_4ClO_4$-$LiClO_4$-$H_2O$ at 35°C

| Point | Liquid phase composition: mass % (1) | mass % (2) | mol %[a] (1) | mol %[a] (2) | molality[a]/mol kg$^{-1}$ (1) | molality[a]/mol kg$^{-1}$ (2) | Solid phase |
|---|---|---|---|---|---|---|---|
| 1 | - | 22.43 | - | 4.246 | - | 2.461 | $NH_4ClO_4$ |
| 2 | 3.34 | 20.62 | 0.709 | 3.964 | 0.413 | 2.308 | " |
| 3 | 4.80 | 19.46 | 1.022 | 3.752 | 0.596 | 2.187 | " |
| 4 | 9.90 | 15.90 | 2.141 | 3.113 | 1.254 | 1.824 | " |
| 5 | 17.15 | 11.35 | 3.814 | 2.286 | 2.255 | 1.351 | " |
| 6 | 21.20 | 9.42 | 4.824 | 1.941 | 2.872 | 1.156 | " |
| 7 | 25.76 | 7.31 | 6.024 | 1.548 | 3.618 | 0.930 | " |
| 8 | 30.05 | 5.00 | 7.187 | 1.083 | 4.349 | 0.655 | " |
| 9 | 31.40 | 4.66 | 7.599 | 1.021 | 4.616 | 0.620 | " |
| 10 | 35.13 | 3.62 | 8.780 | 0.819 | 5.391 | 0.503 | " |
| 11 | 39.80 | 2.43 | 10.39 | 0.574 | 6.476 | 0.358 | $NH_4ClO_4$ + $LiClO_4.3H_2O$ |
| 12 | 40.10 | 2.19 | 10.47 | 0.518 | 6.531 | 0.323 | " |
| 13 | 40.35 | 1.99 | 10.54 | 0.471 | 6.578 | 0.294 | " |
| 14 | 40.38 | 2.17 | 10.58 | 0.515 | 6.607 | 0.321 | " |
| 15 | 40.70 | 1.88 | 10.67 | 0.446 | 6.662 | 0.279 | $LiClO_4.3H_2O$ |
| 16 | 40.80 | 0.97 | 10.58 | 0.228 | 6.586 | 0.142 | " |
| 17 | 41.09 | - | 10.56 | - | 6.556 | - | " |

[a] Editors' calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method. Details of saturation were not given. $Li^+$ was determined by precipitating as lithium zinc uranyl acetate; $NH_4^+$ by distillation of $NH_3$ ; $ClO_4^-$ by difference. | The chemically pure salts were further purified by recrystallization. The hydrate of lithium perchlorate was dehydrated. |
| | **ESTIMATED ERROR:** Not stated. |
| | **REFERENCES:** None. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Ivanov, S.A.; Smirnov, V.N. |
| (2) Thallium perchlorate; $TlClO_4$; [13453-40-2] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1972, *103*, 33-5. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298 K. | N.A. Vasina |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$TlClO_4$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | - | 14.09 | - | 0.9631 | - | 0.5398 | $TlClO_4$ |
| 2 | 9.35 | 5.26 | 1.814 | 0.357 | 1.029 | 0.203 | " |
| 4 | 18.67 | 3.33 | 3.886 | 0.243 | 2.250 | 0.141 | " |
| 5 | 28.17 | 1.99 | 6.383 | 0.158 | 3.791 | 0.0938 | " |
| 6 | 36.76 | 1.78 | 9.182 | 0.156 | 5.622 | 0.0953 | " |
| 7 | 37.14 | 1.70 | 9.310 | 0.149 | 5.708 | 0.0915 | $TlClO_4$ + $LiClO_4.3H_2O$ |
| 9 | 36.92 | 1.58 | 9.215 | 0.138 | 5.643 | 0.0846 | " |
| 10 | 37.12 | 1.83 | 9.320 | 0.161 | 5.715 | 0.0987 | $LiClO_4.3H_2O$ |
| 11 | 37.78 | - | 9.323 | - | 5.707 | - | " |

[a] Values calculated by C.C. Ho.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details were given. | Nothing specified. |
| | ESTIMATED ERROR: |
| | Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Calcium perchlorate; $Ca(ClO_4)_2$; [13477-36-6]<br>(3) Water; $H_2O$; [7732-18-5] | Ivanov, S.A.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1971, *95*, 11-13 |
| **VARIABLES:**<br>One temperature: 298 K<br>Composition. | **PREPARED BY:**<br>Kozyreva, N.A. |

**EXPERIMENTAL VALUES:**

Solubility of the system $LiClO_4$-$Ca(ClO_4)_2$-$H_2O$ at 25°C :

| Liquid phase composition | | | | | | Solid[b] phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 37.52 | 0.00 | 9.230 | 0.00 | 5.644 | 0.000 | A |
| 29.56 | 11.73 | 7.748 | 1.369 | 4.733 | 0.836 | A |
| 14.24 | 31.58 | 4.089 | 4.037 | 2.470 | 2.439 | A |
| 10.18 | 39.44 | 3.130 | 5.398 | 1,899 | 3.276 | A |
| 8.89 | 48.35 | 3.14 | 7.608 | 1.954 | 4.731 | A + B |
| 3.26 | 52.71 | 1.14 | 8.183 | 0.696 | 5.009 | B |
| 0.00 | 65.50 | 0.00 | 12.52 | 0.00 | 7.944 | B |

[a] Compiler's calculations. [b] A = $LiClO_4.3H_2O$
B = $Ca(ClO_4)_2.4H_2O$

The solid phases at the isothermal double saturation point are $Ca(ClO_4)_2.4H_2O$ and $LiClO_4.3H_2O$.

**AUXILIARY INFORMATION**

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method. Conditions of saturation were not given. The chemical, thermographic and X-ray powder analyses were employed. | Not stated. |
| | ESTIMATED ERROR: Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Ganina, G.I.; Pisarenko O.N. |
| (2) Barium perchlorate; $Ba(ClO_4)_2$; [13465-95-7] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1976, *154*, 41-4. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298 K | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system : $Ba(ClO_4)_2$-$LiClO_4$-$H_2O$ at 25° C

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 37.34 | - | 9.166 | - | 5.601 | - | $LiClO_4.3H_2O$ |
| 29.90 | 15.77 | 8.405 | 1.403 | 5.173 | 0.863 | " |
| 19.52 | 33.43 | 6.339 | 3.435 | 3.900 | 2.113 | " |
| 14.71 | 42.96 | 5.286 | 4.885 | 3.270 | 3.018 | " |
| 11.49 | 51.21 | 4.634 | 6.534 | 2.895 | 4.083 | " |
| 6.16 | 58.24 | 2.623 | 7.847 | 1.626 | 4.865 | $LiClO_4.3H_2O$ + $Ba(ClO_4)_2.3H_2O$ |
| 5.25 | 60.06 | 2.291 | 8.294 | 1.423 | 5.149 | $Ba(ClO_4)_2.3H_2O$ |
| 1.41 | 62.17 | 0.597 | 8.330 | 0.364 | 5.077 | " |
| - | 62.63 | - | 8.240 | - | 4.984 | " |

[a] Editors' calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE

The isothermal method was used. The densities and viscosities of the saturated solutions were measured. No description of the chemical analysis was given but literature references were cited [1,2].

SOURCE AND PURITY OF MATERIALS:

Not stated.

ESTIMATED ERROR:

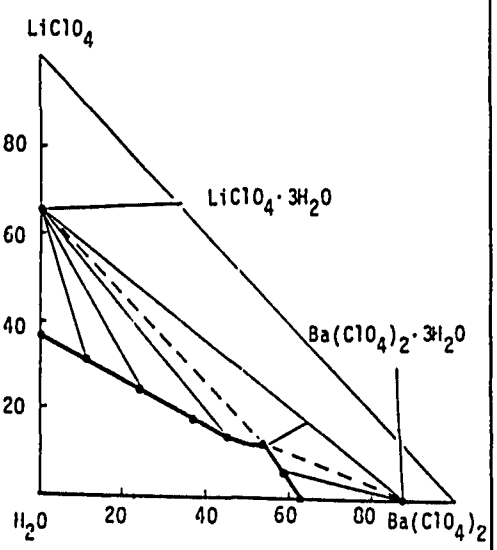
Not stated.

REFERENCES:


1. Hillebrand, W.F.; Lundell, G. G.E.F. *Appl. Inorg. Anal.*, New-York-London, Wiley, 1963.
2. Pribil,R. *Komplexone in der chemischen Analyse. Berlin, Deut. Verl. der Wissens.*, 1961.


COMMENTS AND/OR ADDITIONAL DATA:

The solubility isotherm (mass %) is shown below. The eutonic composition is 6.16 % $LiClO_4$, 58.24 % $Ba(ClO_4)_2$ and 35.60 % $H_2O$.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Manganese perchlorate; $Mn(ClO_4)_2$; [13770-16-6]<br>(3) Water; $H_2O$; [7732-18-5] | Ganina, G.I.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1977, *164*, 58-60. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$Mn(ClO_4)_2$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.34 | - | 9.166 | - | 5.601 | - | A |
| 2 | 34.81 | 4.23 | 8.777 | 0.447 | 5.367 | 0.273 | A |
| 3 | 30.87 | 9.07 | 7.928 | 0.976 | 4.831 | 0.595 | A |
| 4 | 26.43 | 13.00 | 6.784 | 1.399 | 4.101 | 0.8455 | A |
| 5 | 23.72 | 16.91 | 6.219 | 1.858 | 3.755 | 1.122 | A |
| 6 | 20.48 | 21.19 | 5.478 | 2.376 | 3.300 | 1.431 | A |
| 7 | 13.83 | 30.76 | 3.907 | 3.642 | 2.346 | 2.187 | A |
| 8 | 9.60 | 39.25 | 2.926 | 5.014 | 1.764 | 3.023 | A |
| 9 | 5.30 | 44.36 | 1.650 | 5.789 | 0.990 | 3.472 | A |
| 10 | 4.02 | 47.74 | 1.301 | 6.477 | 0.783 | 3.899 | A |
| 11 | 3.38 | 49.95 | 1.13 | 6.980 | 0.681 | 4.216 | A + B |
| 12 | 1.54 | 50.84 | 0.506 | 7.008 | 0.304 | 4.206 | B |
| 13 | - | 51.91 | - | 7.116 | - | 4.252 | B |

[a] Values calculated by C.C. Ho; [b] A = $LiClO_4.3H_2O$; B = $Mn(ClO_4)_2.6H_2O$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. The compositions of liquid phases and residues were analysed for perchlorate ion (ref. 1) and manganese ion (ref. 2). Densities and viscosities of the solutions were measured. | No details were given. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES:<br>1. Hillebrand, W.F.; Lundell, G. E.F. *Applied Inorganic Analysis*, 2nd edi, New York, Wiley, 1963.<br>2. Pribil, R. *Komplexone in der chemischen Analyse*, Berlin, 1961. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Cobalt perchlorate; $Co(ClO_4)_2$; [13455-31-7]<br>(3) Water; $H_2O$; [7732-18-5] | Druzhinina, G.V.; Guseva, A.D.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1970, *79*, 32-5. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$Co(ClO_4)_2$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.53 | - | 9.23 | - | 5.647 | - | A |
| 2 | 30.73 | 8.02 | 7.765 | 0.836 | 4.716 | 0.508 | A |
| 3 | 26.11 | 13.60 | 6.733 | 1.447 | 4.071 | 0.875 | A |
| 4 | 17.75 | 23.74 | 4.758 | 2.626 | 2.851 | 1.574 | A |
| 5 | 13.24 | 32.71 | 3.827 | 3.902 | 2.302 | 2.347 | A |
| 6 | 6.67 | 43.72 | 2.10 | 5.679 | 1.264 | 3.418 | A |
| 7 | 5.29 | 46.64 | 1.715 | 6.240 | 1.034 | 3.763 | A + B |
| 8 | 5.41 | 46.48 | 1.752 | 6.213 | 1.057 | 3.747 | A + B |
| 9 | 4.54 | 47.90 | 1.488 | 6.477 | 0.897 | 3.906 | B |
| 10 | - | 51.93 | - | 7.018 | - | 4.190 | B |

[a] Values calculated by C.C. Ho; [b] A = $LiClO_4.3H_2O$; B = $Co(ClO_4)_2.6H_2O$

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| $Co^{2+}$ was determined by complexometric titration; $ClO_4^-$ by precipitating with nitron. The compositions of the true solid phases were determined by Schreinemakers' method of residues. | Nothing stated. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Nickel perchlorate; $Ni(ClO_4)_2$; [13637-71-3]<br>(3) Water; $H_2O$; [7732-18-5] | Druzhinina, G.V.; Guseva, A.D.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 32-5. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$Ni(ClO_4)_2$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.58 | - | 9.252 | - | 5.659 | - | A |
| 2 | 32.96 | 4.97 | 8.208 | 0.511 | 4.991 | 0.311 | A |
| 3 | 28.41 | 11.02 | 7.272 | 1.165 | 4.409 | 0.706 | A |
| 4 | 21.11 | 20.22 | 5.615 | 2.221 | 3.382 | 1.338 | A |
| 5 | 14.40 | 29.12 | 4.000 | 3.341 | 2.396 | 2.001 | A |
| 6 | 10.33 | 36.90 | 3.063 | 4.519 | 1.840 | 2.714 | A |
| 7 | 7.86 | 41.11 | 2.410 | 5.205 | 1.448 | 3.127 | A |
| 8 | 6.62 | 45.15 | 2.135 | 6.013 | 1.290 | 3.634 | A + B |
| 9 | 6.76 | 45.03 | 2.180 | 5.998 | 1.318 | 3.626 | A + B |
| 10 | 3.19 | 47.98 | 1.025 | 6.364 | 0.614 | 3.814 | B |
| 11 | - | 52.05 | - | 7.056 | - | 4.214 | B |

[a] Values calculated by C.C. Ho; [b] A = $LiClO_4.3H_2O$; B = $Ni(ClO_4)_2.6H_2O$

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. $Ni^{2+}$ was determined by complexometric titration. $ClO_4^-$ by precipitating with nitron. The compositions of the true solid phases were determined by Schreinemakers' method of residues. | Nickel perchlorate was synthesized from nickel carbonate by treating the latter with perchloric acid followed by recrystallization. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Copper perchlorate; $Cu(ClO_4)_2$; [13770-18-8]<br>(3) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Kosheleva, N.I.; Pisarenko, O.N.<br><br>*Sb. Nauch. Tr. Yarosl. Gos. Ped. Inst.*, 1975, *144*, 3-7. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$Cu(ClO_4)_2$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.58 | - | 9.252 | - | 5.659 | - | A |
| 2 | 34.37 | 4.18 | 8.615 | 0.425 | 5.257 | 0.259 | A |
| 3 | 29.60 | 6.60 | 7.236 | 0.654 | 4.361 | 0.394 | A |
| 4 | 23.10 | 12.10 | 5.625 | 1.194 | 3.351 | 0.7115 | A |
| 5 | 17.33 | 19.50 | 4.351 | 1.985 | 2.579 | 1.176 | A |
| 6 | 14.26 | 22.80 | 3.608 | 2.339 | 2.130 | 1.380 | A |
| 7 | 12.29 | 28.10 | 3.271 | 3.032 | 1.938 | 1.796 | A |
| 8 | 10.00 | 32.50 | 2.757 | 3.632 | 1.635 | 2.154 | A |
| 9 | 8.94 | 40.00 | 2.737 | 4.963 | 1.646 | 2.985 | A |
| 10 | 7.62 | 49.30 | 2.702 | 7.087 | 1.663 | 4.360 | A + B |
| 11 | 8.00 | 49.25 | 2.85 | 7.119 | 1.759 | 4.390 | A + B |
| 12 | 2.94 | 51.40 | 1.00 | 7.101 | 0.605 | 4.289 | B |
| 13 | - | 63.20 | - | 10.55 | - | 6.544 | B |

[a] Values calculated by C.C. Ho; [b] A = $LiClO_4.3H_2O$; B = $Cu(ClO_4)_2.6H_2O$

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Equilibrium of the saturated solutions was reached in 2h. $Cu^{2+}$ was determined spectrophotometrically; $ClO_4^-$ gravimetrically by precipitating with nitron; $Li^+$ was determined by difference. | No details were given.<br><br>ESTIMATED ERROR:<br>Nothing specified.<br><br>REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Cerium perchlorate; $Ce(ClO_4)_3$; [14017-47-1]<br>(3) Water; $H_2O$; [7732-18-5] | Druzhinina, G.V.; Rybina, T.V.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1976, *154*, 52-5. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>N.A. Vasina |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$Ce(ClO_4)_3$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.34 | - | 9.17 | - | 5.601 | - | A |
| 2 | 30.69 | 5.04 | 7.459 | 0.297 | 4.488 | 0.179 | A |
| 3 | 22.57 | 17.80 | 5.955 | 1.139 | 3.558 | 0.681 | A |
| 4 | 17.24 | 24.94 | 4.727 | 1.659 | 2.803 | 0.984 | A |
| 5 | 9.44 | 39.50 | 2.945 | 2.990 | 1.738 | 1.764 | A |
| 6 | 6.63 | 47.42 | 2.290 | 3.974 | 1.356 | 2.354 | A |
| 7 | 4.06 | 59.38 | 1.732 | 6.147 | 1.044 | 3.704 | A |
| 8 | 3.35 | 64.05 | 1.585 | 7.351 | 0.966 | 4.481 | A + B |
| 9 | 3.16 | 64.14 | 1.492 | 7.347 | 0.908 | 4.473 | A + B |
| 10 | - | 64.78 | - | 7.026 | - | 4.195 | B |

[a] Values calculated by C.C. Ho; [b] A = $LiClO_4.3H_2O$; B = $Ce(ClO_4)_3.9H_2O$

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Equilibrium was reached after 1-2 days. $ClO_4^-$ was determined gravimetrically by precipitating with nitron, $Ce^{3+}$ by titrating with Trilon B using the indicator xylenol orange. | No details given. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Gadolinium perchlorate; $Gd(ClO_4)_3$; [14017-52-8]<br>(3) Water; $H_2O$; [7732-18-5] | Rybina, T.V.; Druzhinina, G.V.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1978, *169*, 26-8. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$Gd(ClO_4)_3$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.34 | - | 9.166 | - | 5.601 | - | $LiClO_4.3H_2O$ |
| 2 | 26.16 | 11.73 | 6.611 | 0.6922 | 3.959 | 0.4145 | " |
| 3 | 15.10 | 27.36 | 4.179 | 1.768 | 2.467 | 1.044 | " |
| 4 | 10.74 | 35.03 | 3.167 | 2.412 | 1.862 | 1.418 | " |
| 5 | 6.07 | 44.45 | 1.967 | 3.363 | 1.153 | 1.972 | " |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
The isothermal method was used. Samples of liquid phases and residues were taken one or two days after a second salt was added and then analysed. $ClO_4^-$ was determined gravimetrically as nitron perchlorate (ref. 1). $Gd^{3+}$ by complexometric titration with Trilon B in the presence of xylenol orange with urotropin buffer (ref. 2).

SOURCE AND PURITY OF MATERIALS:
Gadolinium perchlorate was prepared in the laboratory (ref. 3). Analysis revealed the gadolinium perchlorate and water contents to correspond to $Gd(ClO_4)_3.9H_2O$.

ESTIMATED ERROR:
Temperature: ± 0.1°C.
Solubility : Nothing specified.

REFERENCES:
1. Muller, G. *Praktikum der Quantitativen Chemischen Analyse*, Leipzig, 1951, 328.
2. Nikolayev (ed) *A Short Course of Radiochemistry*, Vysshaya shkola, Moscow 1969 (in Russian).
3. Paraguzova, T.V.; Druzhinina, G.V. *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1977, *164*, 62.
(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Rybina, T.V.; Druzhinina, G.V. |
| (2) Gadolinium perchlorate; $Gd(ClO_4)_3$; [14017-52-8] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1978, *169*, 26-8. |
| (3) Water; $H_2O$; [7732-18-5] | |

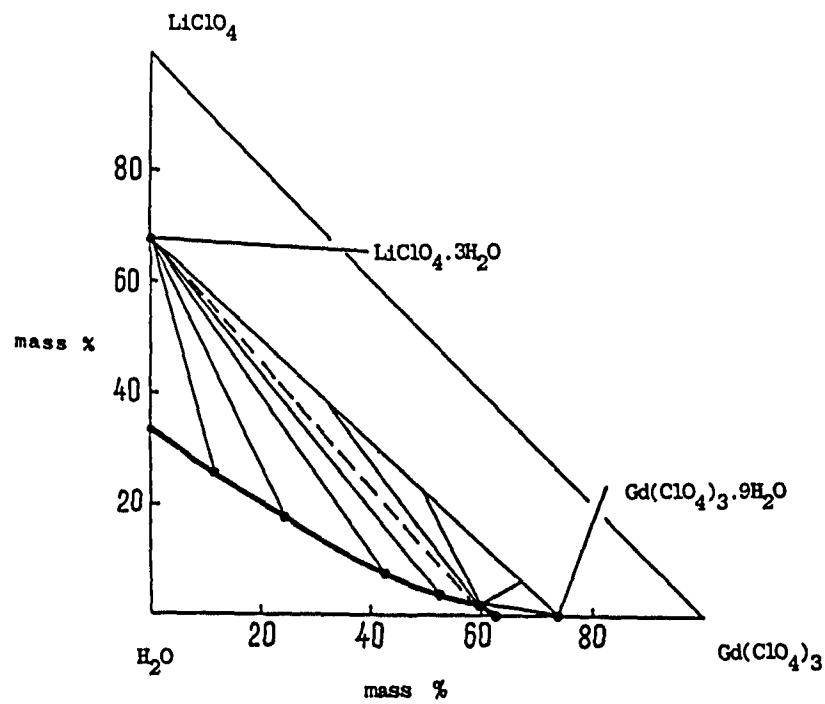
EXPERIMENTAL VALUES: (continued)

| | Liquid phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 6 | 2.07 | 52.28 | 0.729 | 4.301 | 0.426 | 2.514 | $LiClO_4.3H_2O$ |
| 7 | 2.09 | 55.27 | 0.783 | 4.837 | 0.461 | 2.845 | " |
| 8 | 0.89 | 61.18 | 0.372 | 5.973 | 0.221 | 3.540 | $LiClO_4.3H_2O$ + $Gd(ClO_4)_3.9H_2O$ |
| 9 | 0.93 | 61.28 | 0.390 | 6.002 | 0.231 | 3.559 | " |
| 10 | 0.90 | 61.04 | 0.375 | 5.941 | 0.222 | 3.520 | " |
| 11 | - | 64.62 | - | 6.736 | - | 4.009 | $Gd(ClO_4)_3.9H_2O$ |

[a] Values calculated by C.C. Ho.

COMMENTS/ADDITIONAL DATA:

The solubility isotherm (see Figure below) shows a strong salting-out action of gadolinium perchlorate.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Terbium perchlorate; $Tb(ClO_4)_3$; [14014-09-6]<br>(3) Water; $H_2O$; [7732-18-5] | Andronova, N.P.<br><br>*Sb. Nauch. Tr. Yarosl. Gos. Ped. Inst.* 1975, *144*, 24-6. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>I.S. Bodnya |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$Tb(ClO_4)_3$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.66 | - | 9.280 | - | 5.678 | - | A |
| 2 | 24.17 | 18.87 | 6.623 | 1.203 | 3.988 | 0.7245 | A |
| 3 | 19.87 | 24.26 | 5.590 | 1.588 | 3.343 | 0.9496 | A |
| 4 | 18.16 | 28.62 | 5.355 | 1.964 | 3.207 | 1.176 | A |
| 5 | 11.82 | 38.20 | 3.742 | 2.814 | 2.223 | 1.671 | A |
| 6 | 4.61 | 50.70 | 1.645 | 4.208 | 0.970 | 2.481 | A |
| 7 | 3.09 | 56.48 | 1.212 | 5.153 | 0.718 | 3.055 | A |
| 8 | 2.10 | 60.25 | 0.881 | 5.878 | 0.524 | 3.500 | A |
| 9 | 3.19 | 62.87 | 1.462 | 6.702 | 0.883 | 4.051 | A + B |
| 10 | 3.42 | 62.53 | 1.561 | 6.641 | 0.944 | 4.016 | A + B |
| 11 | 3.05 | 62.54 | 1.38 | 6.590 | 0.833 | 3.975 | A + B |
| 12 | 1.63 | 63.73 | 0.737 | 6.709 | 0.442 | 4.023 | B |
| 13 | - | 64.21 | - | 6.602 | - | 3.923 | B |

[a] Values calculated by C.C. Ho; [b] A = $LiClO_4.3H_2O$; B = $Tb(ClO_4)_3.9H_2O$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. $ClO_4^-$ was determined gravimetrically as nitron perchlorate. $Tb^{3+}$ was determined by complexometric titration with Trilon B in the presence of indicator xylenol orange with urotropin buffer. | Terbium perchlorate was prepared by heating terbium nitrate and then dissolving the terbium oxide in 56% perchloric acid. The salt was recrystallized from distilled water and washed with chloroform. After desiccating over $P_2O_5$, the salt contained 72.52 mass % $Tb(ClO_4)_3$ |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Ganina, G.I.; Karnaukhov, A.S.; Lepeshkov, I.N. |
| (2) Lithium nitrate; $LiNO_3$; [7790-69-4] | *Zh. Neorg. Khim.* 1970, *15*, 2825-7; **Russ. J. Inorg. Chem., ( Engl.Transl. )* 1970, *15*, 1469-70. |
| (3) Water; $H_2O$; [7732-18-5] | Andronova, N.P.; Ganina, G.I. *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1970, *78*, 55-8. |
| VARIABLES: One temperature: 298 K. Composition. | PREPARED BY: C.Y. Chan, I.S. Bodnya |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$LiNO_3$-$H_2O$ at 25 °C :

| Solution composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/ mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 37.34 | - | 9.17 | - | 5.601 | - | $LiClO_4.3H_2O$ |
| 35.52 | 2.40 | 8.75 | 0.91 | 5.378 | 0.561 | " |
| 32.88 | 5.68 | 8.13 | 2.17 | 5.030 | 1.341 | " |
| 30.19 | 8.41 | 7.44 | 3.20 | 4.622 | 1.987 | " |
| 26.79 | 11.98 | 6.58 | 4.54 | 4.113 | 2.838 | " |
| 25.04 | 14.29 | 6.18 | 5.44 | 3.879 | 3.416 | " |
| 21.32 | 18.23 | 5.25 | 6.92 | 3.315 | 4.374 | " |
| 17.91 | 22.28 | 4.417 | 8.48 | 2.815 | 5.403 | " |
| 15.47 | 26.78 | 3.889 | 10.39 | 2.518 | 6.726 | " |
| 13.41 | 30.98 | 3.441 | 12.27 | 2.267 | 8.08 | " |
| 10.29 | 36.82 | 2.712 | 14.97 | 1.829 | 10.10 | " |
| 7.86 | 39.69 | 2.075 | 16.17 | 1.409 | 10.98 | " |
| 7.68 | 42.43 | 2.086 | 17.80 | 1.447 | 12.34 | " |

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Details of saturation method were not given. The composition and nature of the solid phases were determined using Schreinemakers' method, chemical analysis and X-ray and optical crystallography methods. Nitrate was determined using Devarda's method (no ref. given), lithium by lithium zinc uranyl acetate precipitation (ref.1), and perchlorate by difference. | No information given. |
| | ESTIMATED ERROR: Not stated. |
| | REFERENCES: 1. Grüttner, B. *Z. Analyt. Chem.* 1951, *133*, 40. |

(continued next page)

COMPONENTS:

(1) Lithium perchlorate; $LiClO_4$; [7791-03-9]

(2) Lithium nitrate; $LiNO_3$; [7790-69-4]

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

ORIGINAL MEASUREMENTS:

Ganina, G.I.; Karnaukhov, A.S.; Lepeshkov, I.N.

*Zh. Neorg. Khim.* 1970, *15*, 2825-7; **Russ. J. Inorg. Chem., ( Engl.Transl. )* 1970, *15*, 1469-70.

Andronova, N.P.; Ganina, G.I.

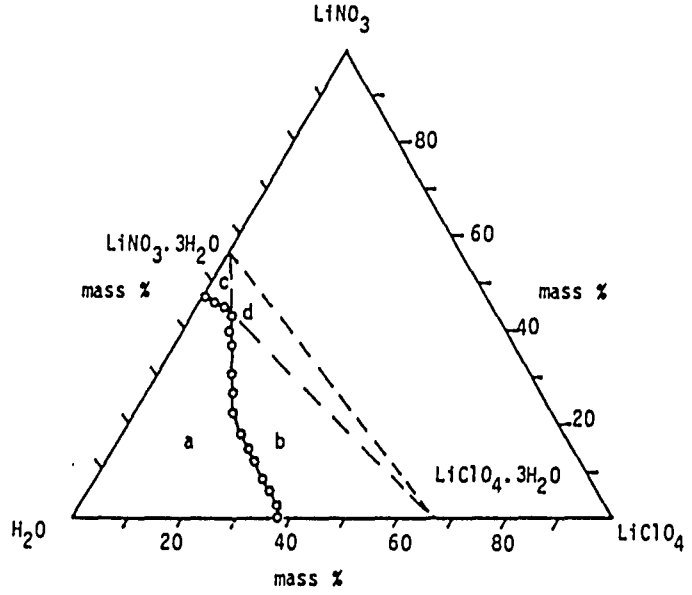
*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1970, *78*, 55-8.

EXPERIMENTAL VALUES: (continued)

Solubility system $LiClO_4$-$LiNO_3$-$H_2O$ at 25 °C :

| Solution composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/ mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 7.66 | 42.35 | 2.080 | 17.75 | 1.440 | 12.29 | $LiClO_4.3H_2O$ + $LiNO_3.3H_2O$ |
| 7.83 | 42.11 | 2.125 | 17.64 | 1.470 | 12.20 | " " |
| 7.55 | 42.78 | 2.058 | 17.99 | 1.429 | 12.49 | " " |
| 7.71 | 42.53 | 2.100 | 17.87 | 1.456 | 12.40 | $LiNO_3.3H_2O$ |
| 4.51 | 44.61 | 1.206 | 18.42 | 0.833 | 12.72 | " |
| 2.76 | 45.52 | 0.729 | 18.56 | 0.502 | 12.77 | " |
| - | 46.99 | - | 18.81 | - | 12.86 | " |

COMMENTS AND/OR ADDITIONAL DATA




Isothermal phase diagram for the system $LiClO_4$-$LiNO_3$-$H_2O$ at 25 °C :

region a —— unsaturated sln mixture.

" b —— mixed sln ($LiClO_4$ satd ) + solid $LiClO_4.3H_2O$.

" c —— mixed sln ($LiNO_3$ satd ) + solid $LiNO_3.3H_2O$.

" d —— mixed sln (both salts satd ) + solid $LiClO_4.3H_2O$ + solid $LiNO_3.3H_2O$.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Lithium nitrate; $LiNO_3$; [7790-69-4]<br>(3) Water; $H_2O$; [7732-18-5] | Andronova, N.P.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1973, *120*, 44-6. |
| VARIABLES:<br>One temperature: 323 K.<br>Composition. | PREPARED BY:<br>N.A. Vasina |

EXPERIMENTAL VALUES:

Solubility system $LiNO_3$-$LiClO_4$-$H_2O$ at 50°C :

| | Liquid phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 45.27 | - | 12.29 | - | 7.775 | - | $LiClO_4.3H_2O$ |
| 2 | 38.08 | 8.98 | 10.45 | 3.801 | 6.761 | 2.460 | " |
| 3 | 35.75 | 12.74 | 9.942 | 5.467 | 6.524 | 3.587 | " |
| 4 | 27.80 | 20.84 | 7.653 | 8.853 | 5.088 | 5.885 | " |
| 5 | 22.90 | 27.73 | 6.410 | 11.98 | 4.360 | 8.147 | " |
| 6 | 12.80 | 51.21 | 4.206 | 25.96 | 3.343 | 20.64 | " |
| 7 | 12.92 | 52.07 | 4.306 | 26.78 | 3.469 | 21.57 | $LiClO_4.3H_2O$ + $LiNO_3$ |
| 8 | 12.70 | 52.30 | 4.232 | 26.89 | 3.411 | 21.67 | " |
| 9 | 13.03 | 52.17 | 4.357 | 26.92 | 3.519 | 21.74 | " |
| 10 | 11.33 | 53.32 | 3.747 | 27.21 | 3.013 | 21.88 | $LiNO_3$ |
| 11 | 8.30 | 55.98 | 2.716 | 28.26 | 2.184 | 22.73 | " |
| 12 | 4.18 | 60.03 | 1.356 | 30.06 | 1.098 | 24.33 | " |
| 13 | 1.20 | 61.98 | 0.382 | 30.43 | 0.306 | 24.42 | " |
| 14 | - | 62.77 | - | 30.58 | - | 24.46 | " |

[a] Values calculated by C.C. Ho.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Equilibrium was reached in 6-8 days, and in the $LiNO_3$ crystallization field in 10 days. The solubility isotherm was constructed using data from chemical analysis. Analytical methods were not stated. | Nothing specified. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Lithium chromate; $Li_2CrO_4$; [14307-35-8]<br>(3) Water; $H_2O$; [7732-18-5] | Lepeshkov, I.N.; Ganina, G.I.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1969, *66*, 96-100. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>I.S. Bodnya |

EXPERIMENTAL VALUES:

Solubility system $Li_2CrO_4$-$LiClO_4$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 37.34 | - | 9.166 | - | 5.601 | - | A |
| 2 | 36.24 | 1.81 | 8.980 | 0.367 | 5.499 | 0.225 | A |
| 3 | 35.42 | 3.34 | 8.859 | 0.684 | 5.436 | 0.420 | A |
| 4 | 32.72 | 6.74 | 8.268 | 1.395 | 5.080 | 0.857 | A |
| 5 | 29.62 | 2.38 | 6.838 | 0.460 | 4.094 | 0.269 | A |
| 6 | 24.82 | 14.71 | 6.300 | 3.059 | 3.858 | 1.873 | A |
| 7 | 16.99 | 22.01 | 4.298 | 4.562 | 2.618 | 2.778 | A |
| 8 | 12.50 | 27.00 | 3.190 | 5.644 | 1.942 | 3.436 | A |
| 9 | 7.54 | 32.79 | 1.949 | 6.945 | 1.188 | 4.231 | A |
| 10 | 4.81 | 37.32 | 1.275 | 8.106 | 0.781 | 4.966 | A |
| 11 | 2.75 | 40.97 | 0.746 | 9.104 | 0.459 | 5.605 | A |
| 12 | 1.99 | 42.32 | 0.544 | 9.484 | 0.336 | 5.851 | A |
| 13 | 1.45 | 45.90 | 0.414 | 10.74 | 0.259 | 6.713 | A |
| 14 | 1.56 | 47.17 | 0.455 | 11.27 | 0.286 | 7.084 | A + B |
| 15 | 1.56 | 47.17 | 0.455 | 11.27 | 0.286 | 7.084 | A + B |
| 16 | 1.56 | 47.17 | 0.455 | 11.27 | 0.286 | 7.084 | A + B |
| 17 | 1.56 | 47.17 | 0.455 | 11.27 | 0.286 | 7.084 | A + B |
| 18 | 0.91 | 48.15 | 0.27 | 11.56 | 0.168 | 7.278 | B |
| 19 | - | 48.88 | - | 11.71 | - | 7.363 | B |

[a] Values calculated by C.C. Ho; [b] A = $LiClO_4.3H_2O$; B = $Li_2CrO_4.2H_2O$

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Equilibrium was reached after 5-6 days. The solutions and solid residues were analysed for the chromate ions and lithium ions. The compositions of the solid phases were determined by Schreinemakers' method of residues. The densities, viscosities and electric conductivities of the saturated solutions were measured. | No details were given. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Lithium chromate; $Li_2CrO_4$; [14307-35-8]<br>(3) Water; $H_2O$; [7732-18-5] | Guseva, A.D.;Lepeshkov, I.N.<br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 3-22. |
| VARIABLES:<br>One temperature: 308 K.<br>Composition. | PREPARED BY:<br>N.A. Vasina |

EXPERIMENTAL VALUES:

Solubility system $Li_2CrO_4$-$LiClO_4$-$H_2O$ at 35°C :

| | Liquid phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | 41.09 | - | 10.56 | - | 6.556 | - | $LiClO_4.3H_2O$ |
| 2 | 38.35 | 3.34 | 9.950 | 0.710 | 6.182 | 0.441 | " |
| 3 | 36.23 | 4.89 | 9.339 | 1.033 | 5.784 | 0.639 | " |
| 4 | 32.32 | 9.41 | 8.414 | 2.007 | 5.214 | 1.243 | " |
| 5 | 25.51 | 17.67 | 6.793 | 3.855 | 4.220 | 2.395 | " |
| 6 | 17.48 | 25.16 | 4.639 | 5.470 | 2.864 | 3.377 | " |
| 7 | 7.97 | 36.09 | 2.166 | 8.036 | 1.339 | 4.968 | " |
| 8 | 4.01 | 42.48 | 1.130 | 9.808 | 0.704 | 6.113 | " |
| 9 | 1.18 | 49.36 | 0.354 | 12.12 | 0.224 | 7.684 | $LiClO_4.3H_2O$ + $Li_2CrO_4.2H_2O$ |
| 10 | 1.20 | 49.19 | 0.359 | 12.05 | 0.227 | 7.635 | " |
| 11 | 1.22 | 49.18 | 0.365 | 12.05 | 0.231 | 7.635 | " |
| 12 | 1.06 | 49.34 | 0.317 | 12.09 | 0.201 | 7.660 | " |
| 13 | 1.09 | 49.48 | 0.327 | 12.15 | 0.207 | 7.708 | " |
| 14 | 1.02 | 49.54 | 0.306 | 12.17 | 0.194 | 7.715 | $Li_2CrO_4.2H_2O$ |
| 15 | - | 50.56 | - | 12.42 | - | 7.874 | " |

[a] Values calculated by C.C. Ho.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Details of saturation method were not given. $Li^+$ was determined by precipitating with lithium zinc uranyl acetate; $CrO_4^{2-}$ by iodine titration; $ClO_4^-$ by difference. | Chemically pure salts were further purified by recrystallization. Hydrates of lithium salts were dehydrated. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$ [7791-03-9]<br>(2) Water; $H_2O$; [7732-18-5]<br>(3) Ethanol (*ethyl alcohol*); $C_2H_6O$; [64-17-5]<br>(4) Ethyl acetate; $C_4H_8O_2$; [141-78-6] | Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1925, *47*, 762-9. |
| VARIABLES:<br>One temperature: 298.2 K | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of lithium perchlorate trihydrate in ethanol-ethyl acetate mixtures at 25.0 °C :

| | Volume % | | | | | | |
|---|---|---|---|---|---|---|---|
| Ethanol (abs) : | 0 | 5 | 10 | 20 | 30 | 40 | 50 |
| Ethyl acetate : | 100 | 95 | 90 | 80 | 70 | 60 | 50 |
| | mass %($LiClO_4.3H_2O$)[a] | | | | | | |
| | 26.35 | - | 31.05 | 33.59 | 35.10 | 36.51 | 37.96 |

| | Volume % | | | | | |
|---|---|---|---|---|---|---|
| Ethanol (abs) : | 60 | 70 | 80 | 90 | 95 | 100 |
| Ethyl acetate : | 40 | 30 | 20 | 10 | 5 | 0 |
| | mass %($LiClO_4.3H_2O$)[a] | | | | | |
| | 38.58 | 39.79 | 40.84 | 41.77 | - | 42.16 |

[a] Solute and solid phase were the trihydrate.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The experimental technique used was essentially the same as that reported in ref. 1 ( see compilation ). Duplicate measurements were made. | Lithium perchlorate trihydrate was prepared in the same manner as that reported in ref. 1. |

ESTIMATED ERROR:
Temperature ± 0.1 °C . Precision in soly determination not stated.

REFERENCES:
1. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1923, *45*, 286.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Kosheleva, N.I |
| (2) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1977, *164*, 5-7. |
| (3) Hexamethylenetetramine; $C_6H_{12}N_4$; [100-97-0] | |
| (4) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | | mol %[a] | | | phase |
| Point | (1) | (2) | (3) | (1) | (2) | (3) | |
| 7 | 6.03 | 0.22 | 44.80 | 1.832 | 0.0319 | 10.33 | A + B + C |
| 8 | 10.21 | 0.35 | 15.84 | 2.234 | 0.0365 | 2.630 | A + C + D |
| 9 | 35.58 | 0.41 | 4.23 | 9.076 | 0.0499 | 0.819 | A + D + E |
| 10 | 3.63 | 46.76 | 1.56 | 1.168 | 7.170 | 0.381 | F + A + E |

| | molality[a]/mol $kg^{-1}$ | | | Solid[b] |
|---|---|---|---|---|
| Point | (1) | (2) | (3) | phase |
| 1 | - | 0.091 | 6.338 | A + B |
| 2 | 1.285 | - | 7.514 | B + C |
| 3 | 3.667 | - | 5.458 | C + D |
| 4 | 5.541 | - | 1.305 | D + E |
| 5 | 0.461 | 3.924 | - | E + F |
| 6 | - | 3.046 | 0.154 | F + B |
| 7 | 1.158 | 0.0201 | 6.528 | A + B + C |
| 8 | 1.304 | 0.0213 | 1.535 | A + C + D |
| 9 | 5.594 | 0.0307 | 0.505 | A + D + E |
| 10 | 0.710 | 4.360 | 0.232 | F + A + E |

[a] Values calculated by C.C. Ho;

[b] A = $Mg(ClO_4)_2.2C_6H_{12}N_4.8H_2O$; B = $C_6H_{12}N_4$; C = $LiClO_4.2C_6H_{12}N_4.5H_2O$; D = $LiClO_4.C_6H_{12}N_4.3H_2O$; E = $LiClO_4.3H_2O$; F = $Mg(ClO_4)_2.6H_2O$.

COMMENTS/ADDITIONAL DATA:

The nodal points and monovariant lines of the solubility isotherm (see Figure) shows the crystallization fields of six solid phases: (1) $C_6H_{12}N_4$; (2) $LiClO_4.2C_6H_{12}N_4.5H_2O$; (3) $LiClO_4.C_6H_{12}N_4.3H_2O$; (4) $Mg(ClO_4)_2.2C_6H_{12}N_4.8H_2O$; (5) $Mg(ClO_4)_2.6H_2O$ and (6) $LiClO_4.3H_2O$.

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8]<br>(3) Hexamethylenetetramine; $C_6H_{12}N_4$; [100-97-0]<br>(4) Water; $H_2O$; [7732-18-5] | Kosheleva, N.I<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1977, *164*, 5-7. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$Mg(ClO_4)_2$-$C_6H_{12}N_4$-$H_2O$ at 25°C :

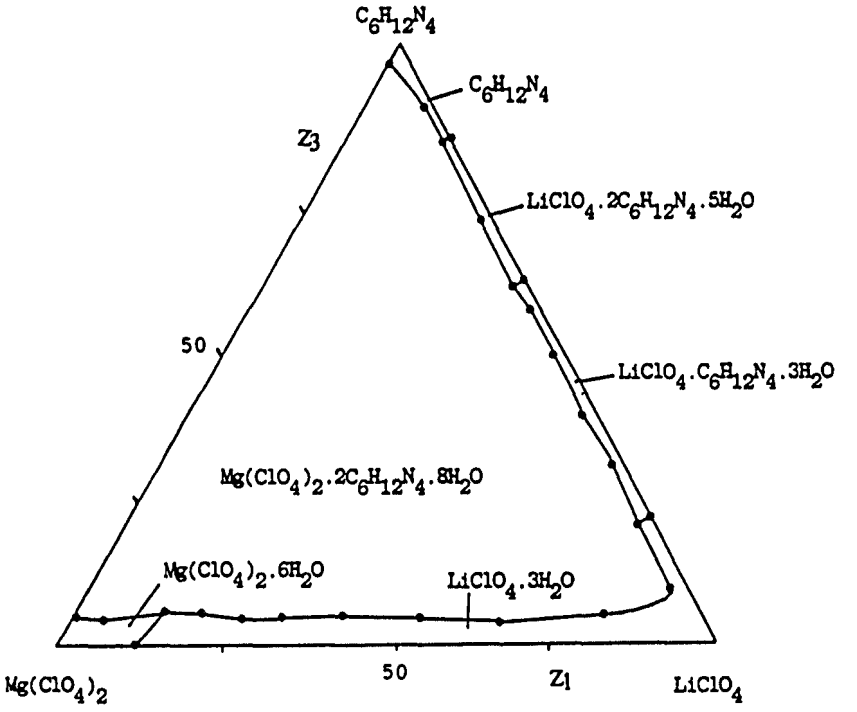
| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | | mol %[a] | | | phase |
| Point | (1) | (2) | (3) | (1) | (2) | (3) | |
| 1 | - | 1.06 | 46.55 | - | 0.146 | 10.23 | A + B |
| 2 | 6.24 | - | 48.10 | 1.998 | - | 11.69 | B + C |
| 3 | 18.10 | - | 35.50 | 5.673 | - | 8.444 | C + D |
| 4 | 33.26 | - | 10.32 | 8.886 | - | 2.093 | D + E |
| 5 | 2.55 | 45.50 | - | 0.770 | 6.551 | - | E + F |
| 6 | - | 39.96 | 1.27 | - | 5.189 | 0.263 | F + B |

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used.<br>Details not given. | No details were given. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES:<br>1. Karnaukhov, A.S.; Kosheleva, N.I. *Sb. Fiziko-khim. issled. ravnov. v rastvorakh.* 1975, *144*, 107.<br>2. Voronina, T.N.; Karnaukhov, A.S. *ibid*, 1970, *78*, 27-31<br>3. Karnaukhov, A.S.; Kosheleva, N.I. *ibid*, 1976, *154*, 62<br>(continued next page) |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8]<br>(3) Hexamethylenetetramine; $C_6H_{12}N_4$; [100-97-0]<br>(4) Water; $H_2O$; [7732-18-5] | Kosheleva, N.I<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1977, *164*, 5-7. |

COMMENTS/ADDITIONAL DATA: (continued)




$Z_i = 100\ x_i/(x_1+x_2+x_3)$

Editors' note: Only 10 original data points were tabulated for the solubility system in this compilation but the above diagram appears to have been constructed from more data, the original reference source of which was not stated.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Copper perchlorate; $Cu(ClO_4)_2$; [13770-18-8]<br>(3) N(1), N(1)-dimethylcarbamide; $C_3H_8N_2O$; [1320-50-9]<br>(4) Water; $H_2O$; [7732-18-5] | Bestuzheva, I.M.; Kinderov, A.P.; Bestuzheva, I.L.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1977, *164*, 5-7. |
| VARIABLES:<br>One temperature: 298 K.<br>Composition. | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $LiClO_4$-$Cu(ClO_4)_2$-$NH_2CON(CH_3)_2$-$H_2O$ at 25°C :

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | | mol %[a] | | | phase |
| Point | (1) | (2) | (3) | (1) | (2) | (3) | |
| 1 | 7.81 | 49.27 | - | 2.777 | 7.102 | - | A + B |
| 2 | - | 58.65 | 3.88 | - | 9.520 | 1.876 | A + C |
| 3 | - | 19.17 | 19.69 | - | 1.979 | 6.056 | C + D |
| 4 | 25.40 | - | 47.19 | 10.40 | - | 23.33 | D + E |
| 5 | 36.79 | - | 22.93 | 12.17 | - | 9.157 | E + B |
| 6 | 7.80 | 48.16 | 2.21 | 2.816 | 7.047 | 0.963 | A + B |
| 7 | 5.91 | 45.47 | 6.05 | 2.088 | 6.512 | 2.581 | A + B + C |
| 8 | 5.90 | 41.60 | 11.28 | 2.109 | 6.027 | 4.868 | C + B |
| 9 | 8.15 | 36.42 | 14.41 | 2.884 | 5.225 | 6.158 | C + B |
| 10 | 8.67 | 32.53 | 18.37 | 3.066 | 4.663 | 7.843 | C + B |
| 11 | 10.18 | 27.22 | 22.73 | 3.583 | 3.884 | 9.660 | C + B |
| 12 | 12.21 | 22.68 | 23.26 | 4.116 | 3.099 | 9.468 | B + C + D |
| 13 | 13.12 | 18.52 | 26.52 | 4.377 | 2.505 | 10.68 | B + D |
| 14 | 15.58 | 13.97 | 28.61 | 5.144 | 1.870 | 11.41 | B + D + E |

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. The compositions of the liquid phases were determined by the content of the ions in solution. (3) was determined by Kjeldahl's method; $Cu^{2+}$ by iodine titration; $ClO_4^-$ gravimetrically with nitron; $Li^+$ by difference. | No details were given.<br><br>ESTIMATED ERROR:<br>Nothing specified.<br><br>REFERENCES:<br>(continued next page) |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Bestuzheva, I.M.; Kinderov, A.P.; Bestuzheva, I.L. |
| (2) Copper perchlorate; $Cu(ClO_4)_2$; [13770-18-8] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1977, *164*, 5-7. |
| (3) N(1), N(1)-dimethylcarbamide; $C_3H_8N_2O$; [1320-50-9] | |
| (4) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

| | Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|---|
| | mass % | | | mol %[a] | | | phase |
| Point | (1) | (2) | (3) | (1) | (2) | (3) | |
| 15 | 17.21 | 9.20 | 31.71 | 5.614 | 1.217 | 12.49 | E + D |
| 16 | 18.03 | 5.08 | 34.19 | 5.751 | 0.657 | 13.17 | E + D |
| 17 | 5.18 | 29.05 | 28.93 | 1.922 | 4.370 | 12.96 | C + D |
| 18 | 8.26 | 24.75 | 21.15 | 2.626 | 3.190 | 8.119 | C + D |
| 19 | 18.22 | 3.950 | 26.52 | 5.135 | 0.4512 | 9.024 | B + E |
| 20 | 20.82 | 7.290 | 27.16 | 6.492 | 0.9214 | 10.23 | B + E |
| 21 | 17.59 | 12.03 | 26.51 | 5.610 | 1.555 | 10.21 | B + E |
| 22 | 1.78 | 51.76 | 4.44 | 0.644 | 7.595 | 1.941 | A + C |
| 23 | 4.11 | 48.33 | 3.73 | 1.432 | 6.825 | 1.569 | A + C |

| | molality[a]/mol $kg^{-1}$ | | | Solid[b] |
|---|---|---|---|---|
| Point | (1) | (2) | (3) | phase |
| 1 | 1.710 | 4.374 | - | A + B |
| 2 | - | 5.964 | 1.175 | A + C |
| 3 | - | 1.195 | 3.655 | C + D |
| 4 | 8.710 | - | 19.54 | D + E |
| 5 | 8.585 | - | 6.461 | E + B |
| 6 | 1.753 | 4.387 | 0.600 | A + B |
| 7 | 1.305 | 4.070 | 1.613 | A + B + C |
| 8 | 1.345 | 3.845 | 3.106 | C + B |
| 9 | 1.868 | 3.383 | 3.987 | C + B |
| 10 | 2.016 | 3.066 | 5.157 | C + B |
| 11 | 2.400 | 2.601 | 6.470 | C + B |
| 12 | 2.742 | 2.065 | 6.308 | B + C + D |
| 13 | 2.947 | 1.687 | 7.194 | B + D |
| 14 | 3.500 | 1.272 | 7.761 | B + D + E |
| 15 | 3.863 | 0.837 | 8.593 | E + D |
| 16 | 3.969 | 0.453 | 9.088 | E + D |
| 17 | 1.322 | 3.005 | 8.913 | C + D |
| 18 | 1.694 | 2.057 | 5.236 | C + D |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Bestuzheva, I.M.; Kinderov, A.P.; Bestuzheva, I.L. |
| (2) Copper perchlorate; $Cu(ClO_4)_2$; [13770-18-8] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1977, *164*, 5-7. |
| (3) N(1), N(1)-dimethylcarbamide; $C_3H_8N_2O$; [1320-50-9] | |
| (4) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

| Point | molality[a]/mol kg$^{-1}$ (1) | (2) | (3) | Solid[b] phase |
|---|---|---|---|---|
| 19 | 3.338 | 0.2933 | 5.866 | B + E |
| 20 | 4.375 | 0.6210 | 6.891 | B + E |
| 21 | 3.769 | 1.045 | 6.858 | B + E |
| 22 | 0.398 | 4.694 | 1.199 | A + C |
| 23 | 0.881 | 4.202 | 0.966 | A + C |

[a] Values calculated by C.C. Ho;

[b] A = $Cu(ClO_4)_2.6H_2O$; B = $LiClO_4.3H_2O$; C = $Cu(ClO_4)_2.2NH_2CON(CH_3)_2.4H_2O$; D = $NH_2CON(CH_3)_2$; E = $LiClO_4.3NH_2CON(CH_3)_2.H_2O$;

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Lithium chromate; $Li_2CrO_4$; [14307-35-8]<br>(3) Potassium perchlorate; $KClO_4$; [7778-74-7]<br>(4) Potassium chromate; $K_2CrO_4$; [7789-00-6]<br>(5) Water; $H_2O$; [7732-18-5] | Voronina, T.N.; Karnaukhov, A.S.; Lepeshkov, I.N.<br><br>*Zh. Neorg. Khim.* 1971, *16*, 871-3;<br>**Russ. J. Inorg. Chem., ( Engl. Transl. )* 1971, *16*, 466-7. |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298 K.<br>Composition. | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility system $2K^+,2Li^+ \;||\; 2ClO_4^-,CrO_4^{2-} - H_2O$ at 25 °C :

| Solution composition mass % | | | | solute ion mol % [a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| | | | | $100x_s$(cation) | | $100x_s$(anion) | | |
| (1) | (2) | (3) | (4) | ($2Li^+$) | ($2K^+$) | ($2ClO_4^-$) | ($CrO_4^{2-}$) | |
| - | - | 0.15 | 39.68 | - | 100.00 | 0.26 | 99.74 | A + B |
| - | 7.02 | 0.74 | 29.82 | 25.71 | 74.29 | 1.27 | 98.73 | " " |
| - | 22.46 | 2.10 | 19.02 | 62.08 | 37.89 | 2.72 | 97.28 | " " |
| - | 32.87 | - | 19.43 | 68.95 | 31.05 | - | 100.00 | B + C |
| - | 34.15 | 2.01 | 11.47 | 79.82 | 20.08 | 2.20 | 97.80 | A + B + C |
| - | 44.28 | - | 7.91 | 89.43 | 11.57 | - | 100.00 | C + D |
| - | 45.29 | 1.98 | 3.12 | 89.94 | 10.06 | 1.84 | 98.16 | A + D + C |
| 28.90 | 9.59 | 1.93 | - | 96.78 | 3.22 | 65.91 | 34.09 | A + D |
| 1.56 | 47.17 | - | - | 100.00 | - | 1.98 | 98.02 | D + E |
| 12.40 | 46.71 | 0.34 | - | 99.71 | 0.29 | 14.30 | 85.70 | A + D + E |
| 28.64 | 9.64 | 0.17 | - | 99.71 | 0.29 | 64.56 | 35.44 | A + E |
| 31.66 | - | 0.19 | - | 99.54 | 0.46 | 100.00 | - | " " |

[a] $x_s[2Li^+] = [n(Li^+)/\{n(Li^+) + n(K^+)\}] = \{1-x_s[2K^+]\}$
$x_s[2ClO_4^-] = [0.5n(ClO_4^-)/\{0.5n(ClO_4^-) + n(CrO_4^{2-})\}] = \{1-x_s[CrO_4^{2-}]\}$
n( ) = amount

[b] A = $KClO_4$ ; B = $K_2CrO_4$ ; C = $Li_2CrO_4.K_2CrO_4.H_2O$
D = $Li_2CrO_4.2H_2O$ ; E = $LiClO_4.3H_2O$

[c] Values in parentheses () original values appear to be in error appear to be in error.

(continued next page)

COMPONENTS:

(1) Lithium perchlorate; $LiClO_4$; [7791-03-9]

(2) Lithium chromate; $Li_2CrO_4$; [14307-35-8]

(3) Potassium perchlorate; $KClO_4$; [7778-74-7]

(4) Potassium chromate; $K_2CrO_4$; [7789-00-6]

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

ORIGINAL MEASUREMENTS:

Voronina, T.N.; Karnaukhov, A.S.; Lepeshkov, I.N.

*Zh. Neorg. Khim.* 1971, *16*, 871-3; **Russ. J. Inorg. Chem., ( Engl. Transl. )* 1971, *16*, 466-7.

EXPERIMENTAL VALUES: (continued)

Solubility system $2K^+,2Li^+ \,||\, 2ClO_4^-,CrO_4^{2-} - H_2O$ at 25 °C :

| Solution composition molality / mol $kg^{-1}$ | | | | Solid phase[b] |
|---|---|---|---|---|
| (1) | (2) | (3) | (4) | |
| - | - | 0.018 | 3.396 | A + B |
| - | 0.866 | 0.086 | 2.460 | " " |
| - | 3.065 | 0.269 | 1.736 | " " |
| - | 5.306 | - | 2.098 | B + C |
| - | 5.021 | 0.277 | 1.128 | A + B + C |
| - | 7.131 | - | 0.852 | C + D |
| - | 7.029 | 0.288 | 0.324 | A + D + C |
| 4.559 | 1.239 | 0.234 | - | A + D |
| 0.286 | 7.084 | - | - | D + E |
| 2.874 | 8.870 | 0.061 | - | A + D + E |
| 4.374 | 1.206 | 0.020 | - | A + E |
| 4.367 | - | 0.020 | - | A + E |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Details of method of saturation not given. $K^+$ was determined gravimetrically using sodium tetraphenylborate, $Li^+$ by the periodate method, $CrO_4^{2-}$ iodometrically, and $ClO_4^-$ by difference. For certain data, $Li^+$ was determined gravimetrically as lithium zinc uranyl acetate. Saturated solutions with compositions corresponding to the 'nodal' points of the quaternary reciprocal aqueous system were gradually treated with a third salt until a new solid phase appeared. With constant stirring, periods of equilibrium varied from 4 - 12 days. Solid phases were examined under a microscope.

SOURCE AND PURITY OF MATERIALS:

Not stated.

ESTIMATED ERROR:

Not stated.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Ganina, G.I.; Karnaukhov, A.S.; Lepeshkov, I.N. |
| (2) Lithium chromate; $Li_2CrO_4$; [14307-35-8] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 151-6 |
| (3) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (4) Ammonium chromate; $(NH_4)_2CrO_4$; [7788-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 298 K | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system : $2NH_4^+$, $2Li^+//2ClO_4^-$, $CrO_4^{2-}$-$H_2O$ at 25°C

| Point | Liquid phase composition, mass % | | | | Liquid phase composition, mol %[a] | | | | Solid phase |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 1 | 35.77 | - | 3.01 | - | 8.942 | - | 0.681 | - | $NH_4ClO_4$ + $LiClO_4.3H_2O$ |
| 2 | 33.09 | 3.72 | 2.54 | - | 8.343 | 0.768 | 0.580 | - | " |
| 3 | 26.83 | 11.14 | 2.67 | - | 6.899 | 2.326 | 0.622 | - | " |
| 4 | 18.55 | 19.78 | 2.86 | - | 4.823 | 4.213 | 0.673 | - | " |
| 5 | 10.12 | 28.42 | 3.06 | - | 2.656 | 6.110 | 0.727 | - | " |
| 6 | 8.58 | 28.27 | 2.80 | - | 2.196 | 5.928 | 0.649 | - | " |
| 7 | 0.61 | 43.94 | 3.45 | - | 0.176 | 10.38 | 0.901 | - | $NH_4ClO_4$ + $LiClO_4.3H_2O$ + $Li_2CrO_4.2H_2O$ |
| 8 | 1.56 | 47.17 | - | - | 0.455 | 11.27 | - | - | $Li_2CrO_4.2H_2O$ + $LiClO_4.3H_2O$ |
| 9 | - | 47.81 | 1.37 | 2.27 | - | 11.92 | 0.377 | 0.483 | $Li_2CrO_4.2H_2O$ + $NH_4ClO_4$ |
| 10 | - | 43.51 | 1.36 | 4.43 | - | 10.50 | 0.363 | 0.913 | " |
| 11 | - | 31.44 | 3.12 | 3.38 | - | 6.480 | 0.711 | 0.595 | $Li_2CrO_4.2H_2O$ + $NH_4ClO_4$ + $(NH_4)_2CrO_4$ |
| 12 | - | 46.60 | 2.99 | 6.93 | - | 12.62 | 0.895 | 1.603 | $Li_2CrO_4.2H_2O$ + $(NH_4)_2CrO_4$. $Li_2CrO_4.2H_2O$ + $(NH_4)_2CrO_4$ |
| 13 | - | 43.79 | 0.85 | 9.52 | - | 11.42 | 0.245 | 2.121 | $(NH_4)_2CrO_4$ + $(NH_4)_2CrO_4$. $Li_2CrO_4.2H_2O$ |
| 14 | - | 43.14 | 0.52 | 10.51 | - | 11.26 | 0.150 | 2.343 | " |
| 15 | - | 42.08 | - | 11.27 | - | 10.85 | - | 2.481 | " |

(continued next page)

COMPONENTS:

(1) Lithium perchlorate; $LiClO_4$; [7791-03-9]

(2) Lithium chromate; $Li_2CrO_4$; [14307-35-8]

(3) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]

(4) Ammonium chromate; $(NH_4)_2CrO_4$; [7788-98-9]

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

ORIGINAL MEASUREMENTS:

Ganina, G.I.; Karnaukhov, A.S.; Lepeshkov, I.N.

*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 151-6

EXPERIMENTAL VALUES: (continued)

Solubility system : $2NH_4^+$, $2Li^+//2ClO_4^-$, $CrO_4^{2-}$-$H_2O$ at 25°C (cont.)

| Point | Liquid phase composition, mass % (1) | (2) | (3) | (4) | mol %[a] (1) | (2) | (3) | (4) | Solid phase |
|---|---|---|---|---|---|---|---|---|---|
| 16 | - | 46.86 | - | 7.95 | - | 12.35 | 1.789 | - | $Li_2CrO_4.2H_2O$ + $(NH_4)_2CrO_4$. $Li_2CrO_4.2H_2O$ |
| 17 | - | 45.76 | 0.97 | 7.34 | - | 11.91 | 1.632 | 0.279 | " |
| 18 | - | 46.97 | 1.63 | 7.29 | - | 12.59 | 1.669 | 0.483 | " |
| 19 | - | 44.54 | 5.79 | 4.45 | - | 11.70 | 0.998 | 1.681 | $(NH_4)_2CrO_4$ + $NH_4ClO_4$ |
| 21 | - | 25.64 | 9.70 | 1.50 | - | 5.201 | 0.260 | 2.175 | " |
| 23 | - | 14.32 | 12.05 | 0.90 | - | 2.591 | 0.139 | 2.410 | " |
| 24 | - | 11.98 | 12.27 | 1.02 | - | 2.120 | 0.154 | 2.400 | $(NH_4)_2CrO_4$ + $NH_4ClO_4$ + $NH_4ClO_4$. $(NH_4)_2CrO_4$ |
| 25 | - | 7.48 | 12.70 | 1.54 | - | 1.274 | 0.224 | 2.391 | $NH_4ClO_4$ + $NH_4ClO_4$. $(NH_4)_2CrO_4$ |
| 27 | - | 3.65 | 16.87 | 2.61 | - | 0.631 | 0.385 | 3.223 | " |
| 29 | - | 1.78 | 17.32 | 2.82 | - | 0.304 | 0.411 | 3.266 | " |
| 31 | - | - | 0.72 | 26.18 | - | - | 4.064 | 0.145 | $(NH_4)_2CrO_4$ + $NH_4ClO_4$. $(NH_4)_2CrO_4$ |
| 33 | - | 2.99 | 0.78 | 19.24 | - | 0.520 | 2.856 | 0.150 | " |
| 35 | - | 8.61 | 6.90 | 6.16 | - | 1.469 | 0.897 | 1.301 | " |
| 37 | - | 11.04 | 11.64 | 1.66 | - | 1.934 | 0.248 | 2.254 | " |

[a] Editors' calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. To the starting solutions corresponding to the non-variant points of the ternary systems a third salt was added until a new solid phase appeared. Details of the saturation method were not given. $NH_4^+$ was determined by distilling off ammonia unto 4 % boric acid solution and then titrating with 0.2 mol $L^{-1}$ $H_2SO_4$; $CrO_4^{2-}$ iodimetrically; $Li^+$ gravimetrically as lithium zinc uranyl acetate; $ClO_4^-$ by difference. The crystals were examined under a microscope.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Guseva, A.D.; Lepeshkov, I.N. |
| (2) Lithium chromate; $Li_2CrO_4$; [14307-35-8] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 3-22 |
| (3) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (4) Ammonium chromate; $(NH_4)_2CrO_4$; [7788-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| Temperature: 308 K | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system: $2NH_4^+$, $2Li^+//2ClO_4^-$, $CrO_4^{2-}$-$H_2O$ at 35°C

| Point | Liquid phase composition, mass % | | | | Liquid phase composition, mol %[a] | | | | Solid phase |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 1 | - | - | 1.19 | 28.00 | - | - | 0.246 | 4.464 | $NH_4ClO_4$. $(NH_4)_2CrO_4$ |
| 2 | - | 2.99 | 1.56 | 15.70 | - | 0.504 | 0.291 | 2.261 | $NH_4ClO_4$. $(NH_4)_2CrO_4$ + $(NH_4)_2CrO_4$ |
| 3 | - | 5.24 | 2.41 | 12.87 | - | 0.885 | 0.450 | 1.857 | " |
| 4 | - | 6.08 | 3.25 | 10.58 | - | 1.020 | 0.603 | 1.516 | " |
| 5 | - | 8.89 | 6.38 | 6.49 | - | 1.518 | 1.204 | 0.947 | " |
| 6 | - | 11.70 | 9.64 | 2.40 | - | 2.038 | 1.856 | 0.357 | " |
| 7 | - | 13.38 | 10.94 | 1.73 | - | 2.389 | 2.159 | 0.264 | $NH_4ClO_4$. $(NH_4)_2CrO_4$ + $NH_4ClO_4$ + $(NH_4)_2CrO_4$ |
| 8 | - | 10.48 | 12.11 | 1.85 | - | 1.838 | 2.348 | 0.277 | $NH_4ClO_4$. $(NH_4)_2CrO_4$ + $NH_4ClO_4$ |
| 9 | - | 5.43 | 14.91 | 3.32 | - | 0.944 | 2.866 | 0.493 | " |
| 10 | - | 2.71 | 19.54 | 3.88 | - | 0.484 | 3.856 | 0.592 | $NH_4ClO_4$. $(NH_4)_2CrO_4$ + $NH_4ClO_4$ |
| 11 | - | - | 20.45 | 3.62 | - | - | 3.945 | 0.539 | $NH_4ClO_4$. $(NH_4)_2CrO_4$ |
| 12 | - | 16.66 | 8.73 | 2.04 | - | 3.022 | 1.751 | 0.316 | $(NH_4)_2CrO_4$ + $NH_4ClO_4$ |
| 13 | - | 17.78 | 5.60 | 3.58 | - | 3.212 | 1.118 | 0.552 | " |
| 14 | - | 19.93 | 4.75 | 3.41 | - | 3.647 | 0.961 | 0.533 | " |

(continued next page)

COMPONENTS:

(1) Lithium perchlorate; $LiClO_4$; [7791-03-9]

(2) Lithium chromate; $Li_2CrO_4$; [14307-35-8]

(3) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]

(4) Ammonium chromate; $(NH_4)_2CrO_4$; [7788-98-9]

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

ORIGINAL MEASUREMENTS:

Guseva, A.D.; Lepeshkov, I.N.

*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 3-22

EXPERIMENTAL VALUES: (continued)

Solubility system : $2NH_4^+$, $2Li^+//2ClO_4^-$, $CrO_4^{2-}$-$H_2O$ at 35°C (cont.)

| Point | Liquid phase composition, mass % (1) | (2) | (3) | (4) | mol % (1) | (2) | (3) | (4) | Solid phase |
|---|---|---|---|---|---|---|---|---|---|
| 15 | - | 20.96 | 4.04 | 3.91 | - | 3.873 | 0.825 | 0.617 | $(NH_4)_2CrO_4$ + $NH_4ClO_4$ |
| 16 | - | 22.27 | 2.67 | 5.20 | - | 4.176 | 0.553 | 0.833 | $(NH_4)_2CrO_4$ + $NH_4ClO_4$ + $Li_2CrO_4.2H_2O$ |
| 17 | - | 27.52 | 0.46 | 5.74 | - | 5.388 | 0.099 | 0.960 | $(NH_4)_2CrO_4$ + $Li_2CrO_4.2H_2O$ |
| 18 | - | 46.63 | - | 8.73 | - | 12.41 | - | 1.983 | " |
| 19 | - | 49.37 | - | 1.12 | - | 12.11 | - | 0.335 | $Li_2CrO_4.2H_2O$ + $LiClO_4.3H_2O$ |
| 20 | 1.53 | 48.91 | 3.65 | - | 0.484 | 12.68 | 1.046 | - | " |
| 21 | 1.38 | 32.07 | 5.73 | - | 0.352 | 6.702 | 1.324 | - | " |
| 22 | 0.77 | 21.28 | 6.58 | - | 0.173 | 3.912 | 1.337 | - | $Li_2CrO_4.2H_2O$ + $NH_4ClO_4$ + $LiClO_4.3H_2O$ |
| 23 | 4.45 | 18.17 | 6.32 | - | 1.001 | 3.347 | 1.287 | - | $LiClO_4.3H_2O$ + $NH_4ClO_4$ |
| 24 | 9.51 | 14.07 | 5.86 | - | 2.147 | 2.602 | 1.198 | - | " |
| 25 | 15.64 | 9.39 | 5.41 | - | 3.562 | 1.752 | 1.116 | - | " |
| 26 | 17.78 | 6.76 | 4.49 | - | 3.982 | 1.240 | 0.911 | - | " |
| 27 | 21.93 | 3.21 | 3.97 | - | 4.908 | 0.589 | 0.805 | - | " |
| 28 | 40.26 | - | 2.13 | - | 10.53 | - | 0.504 | - | " |

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Details of saturation method were not given. $Li^+$ was determined by precipitation as lithium uranyl acetate; $NH_4^+$ by distillation; $CrO_4^{2-}$ iodimetrically; $ClO_4^-$ by difference.

SOURCE AND PURITY OF MATERIALS:

The chemically pure salts were further purified by recrystallization. The hydrates of lithium salts were dehydrated.

ESTIMATED ERROR:

Not stated.

REFERENCES:

None.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9]<br>(2) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8]<br>(3) Lithium chromate: $Li_2CrO_4$; [14307-35-8]<br>(4) Magnesium chromate; $MgCrO_4$; [13423-61-5]<br>(5) Water; $H_2O$; [7732-18-5] | Voronina. T.N.<br><br>*Uch. Zap. Yasosl. Gos. Ped. Inst.* 1970, *79*, 3-8. |
| VARIABLES:<br>One temperature: 298.2 K<br>Composition | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system : $Mg(ClO_4)_2$-$MgCrO_4$-$LiClO_4$-$Li_2CrO_4$-$H_2O$ at 25.0°C

| Liquid phase composition | | | | | | | | Solid Phase[b] |
|---|---|---|---|---|---|---|---|---|
| mass % | | | | mol %[a] | | | | |
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 4.03 | - | 46.45 | - | 1.269 | - | 7.003 | - | A + B |
| 8.16 | - | 46.24 | 0.28 | 2.738 | - | 7.395 | 0.071 | A + B |
| 33.24 | - | 46.34 | 0.32 | 19.07 | - | 12.67 | 0.139 | A + B |
| 34.19 | - | 46.35 | 0.73 | 20.42 | - | 13.19 | 0.331 | A + B + C |
| - | - | 49.71 | - | - | - | 7.389 | - | B + C |
| 22.84 | - | 42.10 | 2.94 | 9.727 | - | 8.546 | 0.949 | B + C |
| 36.95 | - | 41.50 | 0.99 | 20.65 | - | 11.06 | 0.420 | A + C |
| 37.11 | - | 26.98 | 4.36 | 15.49 | - | 5.367 | 1.380 | A + C |
| 37.19 | - | 17.61 | 5.04 | 12.98 | - | 2.929 | 1.334 | A + C |
| 37.38 | - | 5.68 | 12.47 | 11.97 | - | 0.867 | 3.029 | A + C |
| 37.42 | 17.78 | - | 31.38 | 24.14 | 9.394 | - | 15.35 | A + C |
| 31.28 | 21.99 | - | 25.79 | 16.25 | 9.357 | - | 10.16 | A + C |
| 12.12 | 31.11 | - | 12.31 | 3.916 | 8.234 | - | 3.016 | A + C |

[a] Editors' calculations.

[b] A = $LiClO_4.3H_2O$ ; B = $Mg(ClO_4)_2.6H_2O$ ; C = $MgCrO_4.5H_2O$.

AUXILIARY INFORMATION

| METHOD/PROCEDURE/APPARATUS: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Periods of equilibration were 4-6 days. $CrO_4^{2-}$ was determined iodimetrically, $Mg^{2+}$ by titration with EDTA, $Li^+$ as lithium zinc uranyl acetate, and $ClO_4^-$ by difference. | The salts were recrystallized. |
| | ESTIMATED ERROR:<br>Temperature: ±0.1°C. |
| | REFERENCES:<br>None. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Lithium perchlorate; $LiClO_4$; [7791-03-9] | Voronina. T.N. |
| (2) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8] | *Uch. Zap. Yasosl. Gos. Ped. Inst.* 1970, *79*, 3-8. |
| (3) Lithium chromate: $Li_2CrO_4$; [14307-35-8] | |
| (4) Magnesium chromate; $MgCrO_4$; [13423-61-5] | |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system : $Mg(ClO_4)_2$-$MgCrO_4$-$LiClO_4$-$Li_2CrO_4$-$H_2O$ at 25.0°C (cont.)

| Liquid phase composition | | | | | | | | Solid Phase[b] |
|---|---|---|---|---|---|---|---|---|
| mass % | | | | mol %[a] | | | | |
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 2.45 | 34.40 | - | 0.88 | 0.614 | 7.062 | - | 0.167 | A + C |
| 1.83 | 44.27 | - | 4.79 | 0.552 | 10.93 | - | 1.095 | A + C + D |
| - | 44.52 | - | 4.83 | - | 10.75 | - | 1.080 | C + D |
| 2.75 | 46.80 | - | 1.76 | 0.833 | 11.62 | - | 0.404 | A + D |
| 1.56 | 47.17 | - | - | 0.455 | 11.27 | - | - | A + D |

[a] Editors' calculations.

[b] A = $LiClO_4.3H_2O$ ; C = $MgCrO_4.5H_2O$ ; D = $Li_2CrO_4.2H_2O$.

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | C.Y. Chan |
| (2) Water; $H_2O$; [7732-18-5] | Department of Chemistry |
| | University of Malaya |
| | Kuala Lumpur, Malaysia |

CRITICAL EVALUATION

I BINARY SYSTEMS

System $NaClO_4$-$H_2O$

The original values in mass % of the solubility of sodium perchlorate in water at various temperatures reported by different groups as well as the corresponding calculated mean values are given in Table 1. Original data listed which differed by more than twice the standard deviation of the initial mean at the temperature concerned were omitted in the computation of the final mean values. Twenty-six references to the solubility of $NaClO_4$ in water at 298 K were found. However, it should be noted that, as seen in Table 1, certain values have been obtained from more than one compilation reference source and in such cases, it is of the evaluator's opinion that the solubility determination had been carried out only once and that these reports had referred to the same result. Only Willard and Smith (6) indicated the precision of their solubility determination (± 0.05 %). None of the other groups gave any indication of the precision of their experimental results and insufficient information was available for obtaining fair estimates of the error limits of their data in the compilation. The solid phase in equilibrium with the saturated solution at temperatures below 328 K is the monohydrate, $NaClO_4.H_2O$, but at this temperature and above, the solid phase is the anhydrous salt.

Table 1. Solubility of sodium perchlorate in water at various temperatures

| *T/K* | mass % | Solid Phase | Source |
|---|---|---|---|
| 273 | (60.14)[a] | $NaClO_4.H_2O$ | Mikheeva and Titova (79) |
| " | 62.54 | " | Freeth (7) |
| " | 62.87 | " | Cornec and Dickely (10) |
| " | 62.89 | " | Karnaukhov and Makin (20) |
| Mean: | 62.77 | | |

[a] rejected in computation of the mean value

(continued)

CRITICAL EVALUATION (continued)

Table 1 (continued)

| T/K | mass % | Solid Phase | Source |
|---|---|---|---|
| 288 | 65.51 | $NaClO_4 \cdot H_2O$ | Freeth (7) |
| " | 65.63 | " | Cornec and Dickely (10) |
| " | 64.63 | " | Carlson (2) |
| Mean: | 65.26 | | |
| 293 | 66.84 | $NaClO_4 \cdot H_2O$ | Karnaukhov (21) |
| " | (67.48)[a] | " | Zaitseva and Lepeshkov (50) |
| " | 67.58 | " | Karnaukhov (17) |
| " | 67.60 | " | Freeth (7) |
| " | 67.63 | " | Cornec and Dickely (10) |
| " | 67.64 | " | Kudryakova and Karnaukhov (57) |
| " | 67.65 | " | Leboshchina and Kudryakova (85) |
| " | 67.70 | " | Molchanov (28) |
| 298.15 | 67.70 | | Willard and Smith (6) |
| 298 | 67.70 | " | Chernykh, Ivanov and Alekseeva (51) |
| " | 67.79 | " | Lilich and Ovtrakht (27) |
| " | 67.80 | " | Lepeshkov and Druzhinina (34) Druzhinina and Paraguzova (82) Sal'nikova, Karnaukhov and Lepeshkov (65); Karnaukhov and Sal'nilova (55) |
| " | 67.82 | " | Karnaukhov and Makin (20) |
| " | 67.84 | " | Bestuzheva, Kinderov and Karnaukhov (87); Bestuzheva (88) |
| 298.2 | 67.86 | " | Karnaukhov and Kudryakova (32) |
| 298 | 67.89 | " | Karnaukhov and Tarakanov (56,64) Lepeshkov and Tarakanov (70) |
| " | 67.89 | " | Andronova (62); Andronova, Bogomolova and Gulyakova (40) |
| " | 67.89 | " | Smirnov, Ivanov and Chechneva (78) |
| " | 67.92 | " | Andronova (84) |

(continued)

CRITICAL EVALUATION (continued)

Table 1 (continued)

| T/K | mass % | Solid Phase | Source |
|---|---|---|---|
| 298 | (68.84)[a] | $NaClO_4.H_2O$ | Druzhinina (67) |
| Mean: | 67.76 | | |
| 303 | | " | Abdukarimova, Nogoev and Sulaimankulov (74) |
| " | 68.71 | " | Freeth (7) |
| " | 68.25 | | Caven and Bryce (11) |
| Mean: | 68.56 | | |
| 308 | 69.8 | $NaClO_4.H_2O$ | Kudryakova and Karnaukhov (57) Karnaukhov and Guseva (31) |
| 311 | 70.38 | " | Cornec and Dickely (10) |
| 313 | 70.88 | " | Freeth (7) |
| " | 70.87 | " | Karnaukov (17) |
| " | 67.63 | " | Ivanov (44) |
| Mean: | 70.88 | | |
| 323 | (71.28)[a] | $NaClO_4.H_2O$ | Carlson (2) |
| 323 | 73.26 | " | Cornec and Dickely (10) |
| 323 | 73.16 | " | Freeth (7) |
| 323 | 73.15 | " | Zaitseva and Lepeshkov (50) |
| 323 | 73.53 | " | Andronova (62) |
| 323 | 73.75 | " | Andronova (75) |
| 323 | 73.2 | " | Molchanov (28) |
| 323.2 | 73.2 | " | Lepeshkov, Druzhinina and Troitskii (34); Druzhinina (30) |
| Mean: | 73.32 | std. dev.: 0.21 | |
| 323.95 | 73.3 | $NaClO_4$+$NaClO_4.H_2O$ | Freeth (7) |
| 328 | 73.94 | $NaClO_4$ | Cornec and Dickely (10) |
| 333 | 74.30 | " | Freeth (7) |
| " | 74.2 | " | Loseva (71) |
| Mean: | 74.25 | | |
| 335.8 | 74.33 | $NaClO_4$ | Molchanov (28) |
| 348 | 75.00 | " | Freeth (7) |

(continued)

CRITICAL EVALUATION (continued)

Table 1 (continued)

| T/K | mass % | Solid Phase | Source |
|---|---|---|---|
| 348 | 75.01 | $NaClO_4$ | Cornec and Dickely (10) |
| Mean | 75.01 | | |
| 363 | 76.27 | " | Kudryakova and Lepeshkov (46) |
| " | 75.85 | " | Kudryakova and Karnaukhov (57) |
| " | 75.85 | " | Karnaukhov and Troitskii (33) |
| Mean: | 75.99 | | |
| 373 K | 76.75 | " | Cornec and Dickely (10) |
| 416 | 79.08 | " | Carlson (2) |

[a] rejected in computation of the mean value

Based on theoretical treatments described in ref. (76), (90) a semi-empirical equation on the form given by Equation (1) was used to fit the combined original data given in table 1, after conversion of the mass % data to mole fractions. Data were selectively rejected until all the values were fitted to within ± 2s of the calculated value at each selected temperature, s being the standard error defined by $s^2 = (x_{abs}-x_{calc})^2/(N-3)$, where $N$ is the total number of data points.

$$F(x) = a_0 + a_1 (T/K)^{-1} + a_2 \ln (T/K) \qquad (1)$$

where $F(x) = \ln (x^v (1-x)^n/(1+(v-1)x)^{(n+v)})$, $T$ = temperature, $x$ = mole fraction, v = 2 for $NaClO_4$ (stoichiometric number for salt), the mole ratio water : salt is n = 1 when the solid phase is $NaClO_4.H_2O$ and n = 0 when the anhydrous salt is the solid phase.

$a_0$, $a_1$ and $a_2$ are the best-fit parameters, obtained using least-squares linear regression analysis. The results of the analysis are given in Table 2 for n = 1 and n = 0 in Eq. (1).

Table 2 Values of best-fit parameters with reference to Equation 1, pertaining to data in Table 1

| | | |
|---|---|---|
| Temperature : | 273 K - 323 K | |
| Solid phase : | $NaClO_4.H_2O$ | |
| Correlation coefficient : | 0.999 (37 data points) | |
| Parameters | $a_0 = -31.828$ | std. error in F(x) = 0.005 |
| | $a_1 = 6.3_3 \times 10^2$ | " " " $a_1 = 1.7 \times 10^2$ |
| | $a_2 = 4.55$ | " " " $a_2 = 0.57$ |

(continued)

CRITICAL EVALUATION (continued)

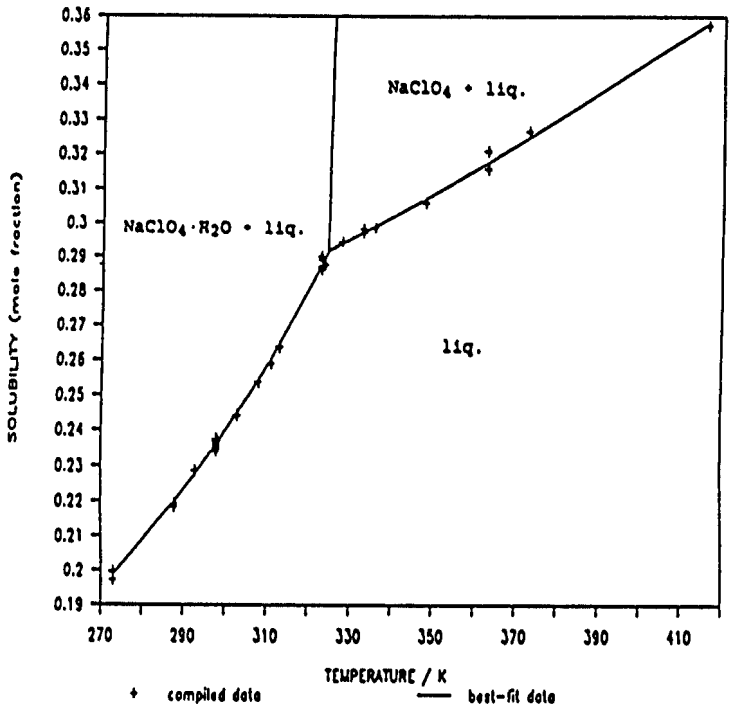
Table 2 (continued)

| | |
|---|---|
| Temperature : | 328 K - 416 K |
| Solid phase : | $NaClO_4$ |
| Correlation coefficient : | 0.996 (11 data points) |
| Parameters | $a_0 = -17.854$ std. error in F(x) = 0.008 |
| | $a_1 = 4.09 \times 10^2$ " " " $a_1 = 3.4 \times 10^2$ |
| | $a_2 = 2.355$ " " " $a_2 = 0.94$ |

The average difference between calculated and observed values was 0.38 % of the observed solubility value.

Figure 1. Solubility-temperature plot for the $NaClO_4$-water system

Solid line represents smoothed data based on Equation 1.




The solubility-temperature plot is shown in Figure 1 and Table 3 lists recommended and tentative smoothed values of the solubility of sodium perchlorate in water, computed based on Equation 1 using the appropriate parameters at the selected temperatures. The recommended values are for the solubilities at 298.15 K and 323.15 K, where original data were

(continued)

CRITICAL EVALUATION: (continued)

reported by more than three independent groups at each of these temperatures.

Table 3. Smoothed data calculated for the solubility of $NaClO_4$ in water at selected temperatures.

| *t*/°C | *T*/K | Solubility mol fraction | Solubility molality | Solid phase | Status |
|---|---|---|---|---|---|
| 0 | 273.15 | 0.1987 | 13.77 | $NaClO_4.H_2O$ | tentative |
| 5 | 278.15 | 0.2056 | 14.36 | " | " |
| 10 | 283.15 | 0.2126 | 14.99 | " | " |
| 15 | 288.15 | 0.2200 | 15.66 | " | " |
| 20 | 293.15 | 0.2278 | 16.38 | " | " |
| 25 | 298.15 | 0.2362 | 17.16 | " | recommended |
| 30 | 303.15 | 0.2451 | 18.02 | " | tentative |
| 35 | 308.15 | 0.2545 | 18.95 | " | " |
| 40 | 313.15 | 0.2647 | 19.98 | " | " |
| 45 | 318.15 | 0.2757 | 21.13 | " | " |
| 50 | 323.15 | 0.2876 | 22.41 | " | recommended |
| 51.8 | 324.95 | 0.2922 | 22.91 | $NaClO_4.H_2O$ + $NaClO_4$ | tentative |
| 55 | 328.15 | 0.2942 | 23.14 | $NaClO_4$ | " |
| 60 | 333.15 | 0.2975 | 23.50 | " | " |
| 65 | 338.15 | 0.3008 | 23.87 | " | " |
| 70 | 343.15 | 0.3041 | 24.26 | " | " |
| 75 | 348.15 | 0.3075 | 24.65 | " | " |
| 80 | 353.15 | 0.3109 | 25.04 | " | " |
| 85 | 358.15 | 0.3144 | 25.45 | " | " |
| 90 | 363.15 | 0.3179 | 25.87 | " | " |
| 95 | 368.15 | 0.3215 | 26.30 | " | " |
| 100 | 373.15 | 0.3251 | 26.74 | " | " |
| 105 | 378.15 | 0.3287 | 27.19 | " | " |
| 110 | 383.15 | 0.3324 | 27.64 | " | " |
| 115 | 388.15 | 0.3362 | 28.11 | " | " |

(continued)

CRITICAL EVALUATION (continued)

Table 3. (continued)

| t/°C | T/K | Solubility | | | Status |
|---|---|---|---|---|---|
| | | mol fraction | molality | Solid phase | |
| 120 | 393.15 | O.3400 | 28.59 | $NaClO_4$ | tentative |
| 125 | 398.15 | 0.3438 | 29.08 | " | " |
| 130 | 403.15 | 0.3477 | 29.59 | " | " |
| 135 | 408.15 | 0.3516 | 30.10 | " | " |
| 140 | 413.15 | 0.3556 | 30.63 | " | " |
| 145 | 418.15 | 0.3596 | 31.17 | | |

The peritectic transition from the monohydrate to anhydrous sodium perchlorate was determined to occur at 324.96 K and at this point the mole fraction of the salt in the solution at equilibrium is 0.2922, molality was 22.91 mol $kg^{-1}$, based on the evaluator's analysis. It should be noted that these values are slightly higher than the values of 0.2877 (mole fraction) and 22.42 mol $kg^{-1}$ calculated from Freeth's data (7) at 323.95 K for the saturated solution in equilibrium with both the anhydrous salt and its monohydrate. The peritectic temperature reported by Cornec and Dickely (10) was 325.90 K.

Cornec and Dickely (10) also reported solubility data in mamount concentration units and saturated solution densities (see compilation) over the temperature range 273 K - 373 K while similar data were reported by Carlson (2) at 288 K, 323 K and 416 K. Carlson's solubility values of 8.79 mol $dm^{-3}$ at 288 K and 10.08 mol $dm^{-3}$ at 323 K are somewhat lower than Cornec and Dickely's values of 8.91 mol $dm^{-3}$ and 10.46 mol $dm^{-3}$ at the corresponding temperatures. There is better agreement between Willard and Smith's value (6) of 9.301 mol $dm^{-3}$ at 298.15 K, Corned and Dickely's value of 9.33 mol $dm^{-3}$ and the value of 9.309 mol $dm^{-3}$ converted from the recommended molal solubility (Table 3) using Willard and Smith's value of 1.6821 g $cm^{-3}$ for the saturated solution density (6) at the same temperature. Table 4 compares solubility values (in mol $dm^{-3}$) reported by Cornec and Dickely (10) and those calculated using the smoothed data given in Table 3 and Cornec and Dickely's saturated solution density values at various temperatures.

(continued)

CRITICAL EVALUATION (continued)

Table 4. Comparison of data for the solubility (in mol $dm^{-3}$) of $NaClO_4$ in water at selected temperatures

| t/°C | T/K | Solubility / mol $dm^{-3}$ | |
|---|---|---|---|
| | | [calc. smoothed data] | [Cornec and Dickely (10)] |
| 15 | 288.15 | 8.94 | 8.91 |
| 25 | 298.15 | 9.309 | 9.33 |
| 50 | 323.15 | 10.47 | 10.46 |
| 75 | 348.15 | 10.78 | 10.76 |
| 100 | 373.15 | 11.00 | 10.86 |

I.2. Solubility in $D_2O$

There is only one report of the solubility of sodium perchlorate in water-$d_2$, $D_2O$, over the temperature range 298-363 K, that of Selecki and Tyminski (60), (see relevant compilation).

I.3. Solubility of sodium perchlorate in other solvents

Data have been reported for the solubility of sodium perchlorate in each of the following solvents :

$T$ = 240-323 K ammonia (22)

$T$ = 272 K perchloric acid (39), hydrogen peroxide (91), hydrazine (59) ;

$T$ = 298 K methanol (6), ethanol (8), 1-propanol (6), 1-butanol (6), 2-methyl,1-propanol (6), sulfinylbis-methane (DMSO) (23,83), 1,2-ethanediol (14), 1,2-ethanediamine (14), 2-amino-ethanol (14), hydrazine (72), ethyl acetate;

$T$ = 308 and 318 K sulfinylbis-methane (DMSO) (23);

$T$ = 313 K tetrahydrothiophene 1-1 dioxide (sulfolane) (73) ;

$T$ = 373 K acetamide (60);

$T$ = 283-323 K 2-butanone (methyl ethyl ketone) (69);

$T$ = 283-323 K acetone (48).

The only data compilations for the solubility of $NaClO_4$ in non-aqueous solvents that have a basis for comparison were those for solubility in acetone at 298 K. Willard and Smith (6) reported that the solubility of sodium perchlorate in acetone at 298.15 K was 4.228 mol $kg^{-1}$ while Krumgal'z et al. (69) reported a value of 4.422 mol $kg^{-1}$, the solid phase being the

(continued)

CRITICAL EVALUATION: (continued)

anhydrous salt in both reports. Information on source and purity of materials as well as details of method were available in the former's report but not in the second.

II. TERNARY SYSTEMS

Solubility in mixed solvents

*Systems $NaClO_4$ - organic solvent - $H_2O$*

Data for such systems were reported in which the organic omponent was one of the following

$T$ = 298 K dimethylurea (88), thiocarbamide (64), acetamide (70), benzamide (87) and hexamethylenetetramine (65).

$T$ = 303 K carbamide (74), thiocarbamide (74).

No critical evaluation could be carried out for lack of relevant information.

*Systems $NaClO_4$ - alcohol - ethyl acetate*

The solubility at 298 K in mixed solvent ethyl acetate -alcohols (methanol, ethanol, 1-butanol has been determined by Smith (8).

Systems $NaClO_4$ - inorganic compound - water

The data concerning isothermal sections of the ternary systems involving a second salt are summarized in the following pages and some anomalies are mentioned but a critical evaluation of data could not be carried out because of insufficient information.

*Systems involving the same anion*

*Systems $NaClO_4$-$MClO_4$-$H_2O$ with M = H, Li, K, Cs, $NH_4$, Tl*

*$NaClO_4$-$HClO_4$-$H_2O$*

At 298 K (51) the solid phases are $NaClO_4$, $NaClO_4 \cdot H_2O$ and $NaClO_4 \cdot HClO_4$.

*$NaClO_4$-$LiClO_4$-$H_2O$*

The results are presented in the critical analysis of $LiClO_4$.

*$NaClO_4$-$KClO_4$-$H_2O$*

$T$ = 273 K (20), the observed solid phases are $NaClO_4$, $NaClO_4 \cdot H2O$, $KClO_4$ and $mNaClO_4 \cdot nKClO_4$. The data are scattered; furthermore, the double saturation point cannot involve $KClO_4$ and $NaClO_4$

$T$ = 298 and 323 K (34, 30) the observed solid phases are $NaClO_4$, $NaClO_4 \cdot H2O$, $KClO_4$.

(continued)

CRITICAL EVALUATION (continued)

*$NaClO_4$-$CsClO_4$-$H_2O$ (81)*

At 298 K the solid phases in equilibrium with liquid are $NaClO_4 \cdot H_2O$, $CsClO_4$ and two double salts $3CsClO_4 \cdot NaClO_4$ and $CsClO_4 \cdot NaClO_4$.

At 348 K $3CsClO_4 \cdot NaClO_4$ is no longer observed.

*$NaClO_4$-$NH_4ClO_4$-$H_2O$ (57)*

Two intermediate compounds are mentioned C=n($NH_4ClO_4$)·m($NaClO_4 \cdot H_2O$) and D=n($NH_4ClO_4$)·m($NaClO_4$); their stoichiometry is not known; their fields of existence are small and the phase diagram presents some anomalies so that the data must be considered very cautiously.

$T$ = 298 K the observed solid phases are $NaClO_4 \cdot H_2O$, $NH_4ClO_4$ and C.

$T$ = 308 K the solid phases are $NaClO_4 \cdot H_2O$, $NH_4ClO_4$, C and D.

$T$ = 363 K three solid phases can crystallize $NaClO_4$, $NH_4ClO_4$ and D.

*$NaClO_4$-$TlClO_4$-$H_2O$ (78)*

At 298 K the observed solid phases are $NaClO_4 \cdot H_2O$ and $TlClO_4$.

*Systems $NaClO_4$-$M(ClO_4)_2$-$H_2O$ with M = Mg, Ca, Sr, Ba, Zn, Ni*

*$NaClO_4$-$Mg(ClO_4)_2$-$H_2O$*

$T$ = 298 K (32) the observed solid phases are $NaClO_4 \cdot H_2O$ and $Mg(ClO_4)_2 \cdot 6H_2O$.

$T$ = 363 K (46) the solid phases are $NaClO_4$ and $Mg(ClO_4)_2 \cdot 6H_2O$.

*$NaClO_4$-$Ca(ClO_4)_2$-$H_2O$*

$T$ = 298 K (27) the observed solid phases are $NaClO_4 \cdot H_2O$ and $Ca(ClO_4)_2 \cdot 4H_2O$.

$T$ = 313 K (44) the same phases can crystallize.

*$NaClO_4$-$Sr(ClO_4)_2$-$H_2O$ (67)*

At 298 K the observed solid phases are $NaClO_4 \cdot H_2O$ and $Sr(ClO_4)_2 \cdot 4H_2O$.

*$NaClO_4$-$Ba(ClO_4)_2$-$H_2O$ (50)*

$T$ = 298 K the solid phases are $NaClO_4 \cdot H_2O$ and $Ba(ClO_4)_2 \cdot 3H_2O$.

$T$ = 323 K two other salts can also crystallize according to the composition $NaClO_4$ and $Ba(ClO_4)_2 \cdot 2H_2O$.

*$NaClO_4$-$Zn(ClO_4)_2$-$H_2O$ (85)*

At 298 K the observed solid phases are $NaClO_4 \cdot H_2O$ and $Zn(ClO_4)_2 \cdot 6H_2O$.

(continued)

CRITICAL EVALUATION (continued)

*$NaClO_4$-$Ni(ClO_4)_2$-$H_2O$ (56)*

At 298 K the observed solid phases are $NaClO_4 \cdot H_2O$ and $Ni(ClO_4)_2 \cdot 6H_2O$.

*Systems $NaClO_4$-$M(ClO_4)_3$-$H_2O$ where M = Al, Ce, Tb*

*$NaClO_4$-$Al(ClO_4)_3$-$H_2O$ (11)*

At 303 K the solid phases are $NaClO_4 \cdot H_2O$, $Al(ClO_4)_3 \cdot nH_2O$.

*$NaClO_4$-$Ce(ClO_4)_3$-$H_2O$ (82)*

At 298 K three solid phases are observed $NaClO_4$, $NaClO_4 \cdot H_2O$ and $Ce(ClO_4)_3 \cdot 9H_2O$.

*$NaClO_4$-$Tb(ClO_4)_3$-$H_2O$ (84)*

At 298 K the solid phases $NaClO_4$, $NaClO_4 \cdot H_2O$ and $Tb(ClO_4)_3 \cdot 9H_2O$ can be observed.

*Systems involving the same cation*

*$NaClO_4$-NaCl-$H_2O$*

The solubilities have been measured between 273 and 373 K (10, 21, 32, 33, 71). At low temperatures the solid phases are $NaClO_4 \cdot H_2O$ and NaCl, at higher temperatures $NaClO_4$ and NaCl The nature of the solid perchlorate indicated on the 293 K data sheet (21) is erroneous.

*$NaClO_4$-$NaNO_3$-$H_2O$ (62)*

Two isothermal sections have been investigated,the solid phases are $NaClO_4 \cdot H_2O$ and $NaNO_3$.

*$NaClO_4$-$NaClO_3$-$H_2O$ (71)*

At 333 K the solubility curves of pure components, $NaClO_4$ and $NaClO_3$, are observed.

*$NaClO_4$-$Na_2SO_4$-$H_2O$ (7)*

Two isothermal sections have been investigated. At 298 K three solid phases are observed, $NaClO_4 \cdot H_2O$, $Na_2SO_4$ and $Na_2SO_4 \cdot 10H_2O$. At 333 K the solid phases are the anhydrous salts $NaClO_4$ and $Na_2SO_4$.

*$NaClO_4$-$Na_2CrO_4$-$H_2O$*

The system has been investigated at three temperatures.

$T$ = 298 K, Karnaukhov (31) mentions the existence of $NaClO_4 \cdot H_2O$ and $Na_2CrO_4 \cdot 4H_2O$ while Molchanov (28) indicates also a solubility field of $Na_2CrO_4$.

(continued)

CRITICAL EVALUATION (continued)

$T$ = 323 K (28, 30) the observed solid phases are $NaClO_4 \cdot H_2O$, $Na_2CrO_4 \cdot 4H_2O$ and $Na_2CrO_4$.

$T$ = 335 K (28) the dehydrated salts $NaClO_4$ and $Na_2CrO_4$ are observed.

*$NaClO_4$-$Na_2Cr_2O_7$-$H_2O$ (55)*

At 298K the solid phases are $NaClO_4 \cdot H_2O$ and $Na_2Cr_2O_7 \cdot 2H_2O$.

$NaClO_4$-$Na_3PO_4$-$H_2O$ (66)

At 298 K the solid phases are $NaClO_4.H_2O$, $Na_3PO_4.12H_2O$ and a solid solution is observed.

*Systems $NaClO_4$-inorganic compound-$NH_3$*

$NaClO_4$-NaCl-$NH_3$ (22)

In the range 240-323 K the solid phases are the pure components $NaClO_4$ and NaCl.

$NaClO_4$-$NH_4ClO_4$-$NH_3$ (22)

The system has been investigated between 240 and 323 K . The solid phases are the pure components $NaClO_4$ and $NH_4ClO_4$.

## III QUATERNARY SYSTEMS

Simple quaternary systems

*$NaClO_4$-Water-alcohols-ethyl acetate (8)*

The solubility of $NaClO_4 \cdot H_2O$ in alcohol-methyl acetate mixtures has been determined at 298 K . The investigated alcohols were methanol, ethanol (abs. and 93 %), 1-butanol. The solid phase was $NaClO_4.H_2O$.

*$NaClO_4$-$NH_4ClO_4$-Hexamethylenetetramine-Water (77)*

At 298 K eight solubility fields were found corresponding to:
$NH_4ClO_4$, $NaClO_4 \cdot H_20$, $C_6H_{12}N_4$, $NH_4ClO_4 \cdot C_6H_{12}N_4$, $NH_4ClO_4 \cdot 2C_6H_{12}N_4$, $NaClO_4 \cdot C_6H_{12}N_4$, $n(NH_4ClO_4) \cdot m(NaClO_4)$ and $5(NaClO_4) \cdot 2(C_6H_{12}N_4) \cdot 3H_2O$.

*$NaClO_4$-$Cu(ClO_4)_2$-Benzamide-Water (87)*

The system was investigated at 298 K. Three solid phases were observed, $NaClO_4 \cdot H_20$, $C_6H_5CONH_2$ and $Cu(ClO_4)_2$.

(continued)

CRITICAL EVALUATION (continued)

*$NaClO_4$-$Ni(ClO_4)_2$-Urea-Water (86)*

At 298 K seven solubility fields were found corresponding to $NaClO_4 \cdot H_2O$, $Ni(ClO_4)_2 \cdot 6H_2O$, $CH_4N_2O$, $NaClO_4 \cdot 2CH_4N_2O$, $2NaClO_4 \cdot 3CH_4N_2O$, $Ni(ClO_4)_2 \cdot 4CH_4N_2O \cdot 2H_2O$ and $Ni(ClO_4)_2 \cdot 6CH_4N_2O$.

*$NaClO_4$-$NaClO_3$-NaCl-Water (41-43)*

The isothermal section was investigated at two temperatures. At 313 K three compounds are observed $NaClO_4 \cdot H_2O$, $NaClO_3$, NaCl. At 333 K a solubility field of $NaClO_4$ is also observed

Reciprocal quaternary systems

The data concerning the reciprocal quaternary systems must be considered very cautiously , due to the incoherence of several data and to the erroneous attribution of the crystallization fields of some solid phases.

*$Na^+$, $K^+$ // $ClO_4^-$, $Cl^-$ - $H_2O$ (33)*

The isothermal section 363 K was investigated. According to the composition five solid phases can crystallize : NaCl, KCl, $NaClO_4$, $KClO_4$ and a double salt $mKCl \cdot nKClO_4$.

*$Na^+$, $K^+$ // $ClO_4^-$, $CrO_4^{2-}$ - $H_2O$ (30)*

At 323 K five salts are observed in the diagram $NaClO_4 \cdot H_2O$, $KClO_4$, $K_2CrO_4$, $Na_2CrO_4$ and a double salt $3K_2CrO_4 \cdot Na_2CrO_4$.

*$Na^+$, $Cs^+$ // $ClO_4^-$, $Cl^-$ - $H_2O$ (81)*

The isothermal section was investigated at two temperatures. At 298 K the components CsCl, NaCl, $CsClO_4$, a solid solution $Cs_{1-x}Na_xCl$ and two double salts $3CsClO_4 \cdot NaClO_4$, $CsClO_4 \cdot NaClO_4$ have a crystallization field. At 348 K $NaClO_4 \cdot H_2O$ and $3CsClO_4 \cdot NaClO_4$ are no longer observed and there is a solubility range for anhydrous $NaClO_4$·

*$Na^+$, $NH_4^+$ // $ClO_4^-$, $Cl^-$ - $H_2O$ (54,63)*

The system has been investigated by the same authors at three temperatures. The observed solid phases are indicated in the following table by x:

(continued)

CRITICAL EVALUATION (continued)

System $Na^+$, $NH_4^+$ // $ClO_4^-$, $Cl^-$ - $H_2O$

observed solid phases

| *T* / K | (I) | (II) | (III) | (IV) | (V) | (VI) |
|---|---|---|---|---|---|---|
| 298 | x | x | x | | x | x |
| 308 | x | x | x | | x | x |
| 363 | x | x | x | x | | x |

(I) = $NH_4Cl$, (II) = $NH_4ClO_4$, (III)= NaCl, (IV) = $NaClO_4$, (V) = $NaClO_4 \cdot H_2O$
(VI) = $mNH_4ClO_4 \cdot nNaClO_4 \cdot H_2O$

*$Na^+$, $NH_4^+$ // $ClO_4^-$, $SO_4^{2-}$ - $H_2O$ (7)*

Two isothermal sections of the diagram have been investigated. The observed solid phases are indicated in the following table by x:

System $Na^+$, $NH_4^+$ // $ClO_4^-$, $SO_4^{2-}$ - $H_2O$

observed solid phases

| *T* / K | A | B | C | D | E | F | G |
|---|---|---|---|---|---|---|---|
| 298 | x | x | x | x | x | x | |
| 333 | x | x | | | x | | x |

A=$NH_4ClO_4$, B=$(NH_4)_2SO_4$, C=$Na_2SO_4 \cdot (NH_4)_2SO_4 \cdot 10H_2O$, D=$Na_2SO_4 \cdot 10H_2O$
E=$Na_2SO_4$, F=$NaClO_4 \cdot H_2O$, G=$NaClO_4$

*$Na^+$, $NH_4^+$ // $ClO_4^-$, $CrO_4^{2-}$ - $H_2O$ (35, 52)*

Two sets of data are found in literature, at 298 and 308 K. The isothermal sections are very similar. At 298 K the solid phases shown in the corner closest to of $NaClO_4$ are $Na_2CrO_4$, $NaClO_4 \cdot H_2O$ and a mixture represented by $n(NH_4ClO_4) \cdot m(NaClO_4 \cdot H_2O)$. At 303 K the solubility surface of anhydrous $NaClO_4$ is no longer observed. This difference is surprising since the increase of temparature ought to favor the dehydration of $NaClO_4 \cdot H_2O$.

*$Na^+$, $NH_4^+$ // $ClO_4^-$, $Cr_2O_7^{2-}$ - $H_2O$ (58)*

The crystallization fields of mixtures are not clearly represented on the phase diagram. Furthermore, in the last column of the compilation sheet the double saturation lines are problably D+F and A+F instead of D+E and A+E.

(continued)

CRITICAL EVALUATION (continued)

*$Na^+$, $Mg^{2+}$ // $ClO_4^-$, $Cl^-$ - $H_2O$ (46)*

At 363 K the diagram shows four solubility ranges corresponding to NaCl, $NaClO_4$, $Mg(ClO_4)_2 \cdot 6H_2O$ and $MgCl_2 \cdot 6H_2O$

*$Na^+$, $Ba^{2+}$ / $ClO_4^-$, $Cl^-$ - $H_2O$ (50, 61)*

The assessment of phases in the last column of the data sheets is not correct. For example, at 298 K $Ba(ClO_4)_2$ crystallizes from aqueous solution as a dihydrate and it is not possible to pass continuously from an hydrated salt to an anhydrous solid solution, as shown on the diagram.

*$Na^+$, $Ni^{2+}$ // $ClO_4^-$, $NO_3^-$ - $H_2O$ (68)*

At 363 K the diagram shows four solubility ranges corresponding to $NaNO_3$, $NaClO_4 \cdot H_2O$, $Ni(ClO_4)_2 \cdot 6H_2O$ and $Ni(NO_3)_2 \cdot 6H_2O$

*$Na^+$, $Zn^{2+}$ // $ClO_4^-$, $SO_4^{2-}$ - $H_2O$ (89)*

$ZnSO_4 \cdot H_2O$ mentioned in the data sheet is not observed on the phase diagram

*$Na^+$, $NH_4^+$ // $ClO_4^-$, $Cl^-$ - $NH_3$ (22)*

At 240 and 323 K only the pure components are observed.

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78 Smirnov,V.N.; Ivanov, S. A.; Chechneva, I.V. *Uch. Zap. Yarosl. Gos. Ped. Inst.*, 1973, *120*, 13

79 Mikheeva, V. I.; Titova, K. V. *Zh. Neorg. Khim.*, 1974, *19*, 2900; **Russ. J. Inorg.Chem. (Engl. Transl.)*, 1974, *19*, 1586

80 Shklovskaya, R.M.; Arkhipov, S.M.; Kusina, V.A. *Zh. Neorg. Khim.*, 1974, *19*, 3128; **Russ. J. Inorg. Chem. (Engl. Transl.)*, 1974, *19*, 1711

81 Shklovskaya, R.M.; Arkhipov, S.M.; Kusina, V.A.; Kirgintsev, A.N. *Zh. Neorg. Khim.*, 1974, *19*, 846; **Russ. J. Inorg. Chem. (Engl. Transl.)*, 1974, *19*, 462

82 Druzinina, G. V.; Paraguzova, T. V. *Uch. Zap. Yarosl. Gos. Ped. Inst.*, 1975, *144*, 76

83 Ferroni, G.; Villetard, M. *Ann. Chim..* 1975, *10*, 33

84 Andronova, N. P. *Uch. Zap. Yarosl. Gos. Ped. Inst.*, 1976, *154*, 25


(continued)

CRITICAL EVALUATION (continued)

85 Leboshchina, V.I.; Kudryakova, S.A. *Uch. Zap. Yarosl. Gos. Ped. Inst.*, 1976, *154*, 68

86 Tarakanov, V.F. *Uch. Zap. Yarosl. Gos. Ped. Inst.*, 1976, *154*, 35

87 Bestuzheva, I. M.; Kinderov, A.P.; Karnaukhov, A.S.. *Sb. Tr. Yarosl. Gos. Ped. Inst.*, 1978, *169*, 37

88 Bestuzheva, I.M. *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1979, *178*, 67

89 Karnaukhov, A.S.; Leboschina, V.I. *Tr. Yarosl. Gos. Ped. Inst.*, 1979, *178*, 24

90 Counioux, J.J.; Tenu, R. *J. Chim. Phys.* 1981, *78*, 815

91 Titova, K. V.; Kolmakova, E. I.; Rosolovskii, V. Ya. *Zh. Neorg. Khim.*, 1986, *31*, 3213; **Russ. J. Inorg. Chem. (Engl. Transl.)*, 1986, *31*, 1846

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Willard, H.H.; Smith, G.F. |
| (2) Water; $H_2O$; [7732-18-5] | *J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES: | PREPARED BY: |
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of sodium perchlorate in water at 25.00°C :

| mass % | g/100 $cm^{-3}$ sln | mol % | mol $dm^{-3}$ | mol $kg^{-1}$ $m_1$ | satd sln. density/g $cm^{-3}$ |
|---|---|---|---|---|---|
| 67.70 | 113.88 | 23.57[b] | 9.301 [b] | 17.118[b] | 1.6821 |

[a] The solid phase was mixture of the anhydrous salt and that crystallized from the sat. sln (probably $NaClO_4.H_2O$).
[b] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles. The salt was dried to const. wt. at 250 °C in a current of air dried with $P_2O_5$. Duplicate soly determinations were made, those analyses in which chloride was found present being rejected.

SOURCE AND PURITY OF MATERIALS:
Anhyd. $NaClO_4$ was prepared from pure sodium carbonate and slight excess of purified $HClO_4$ (ref.1) by crystallization above 50 °C (ref. 2). After centrifugal separation, the solid was dried in a current of dry air at 250 °C.

ESTIMATED ERROR:
Precision in temp. was ± 0.01 °C
Precision in solubility data was ± 0.05 %.

REFERENCES:
1. Willard, H.H.; *J. Am. Chem. Soc.* 1912, *34*, 1480.
2. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Freeth, F.A. |
| (2) Water; $H_2O$; [7732-18-5] | *Rec. Trav. Chim. Pays-Bas* 1924, *43*, 475-507. |
| VARIABLES: | PREPARED BY: |
| Temperature: 241 - 348 K. | C.Y. Chan |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$H_2O$ at various temperatures :

| Solubility of $NaClO_4$ | | | | Solid |
|---|---|---|---|---|
| t/ °C | mass % | mol % [a] | molality[a]/ mol $kg^{-1}$ | phase |
| 75 | 75.0 | 30.62 | 24.50 | $NaClO_4$ |
| 60 | 74.3 | 29.84 | 23.61 | " |
| 50.8 | 73.3 | 28.77 | 22.42 | $NaClO_4$ + $NaClO_4.H_2O$ |
| 50 | 73.16 | 28.63 | 22.26 | $NaClO_4.H_2O$ |
| 40 | 70.88 | 26.37 | 19.88 | " |
| 30 | 68.71 | 24.42 | 17.94 | " |
| 15 | 65.51 | 21.84 | 15.51 | " |
| 0 | 62.54 | 19.72 | 13.64 | " |
| -0.3 | 10 | 1.6 | 0.91 | ice |
| -6.8 | 20 | 3.5 | 2.0 | " |
| -11.1 | 30 | 5.9 | 3.5 | " |
| -17.8 | 40 | 8.9 | 5.5 | " |
| -22 | 45 | 10.7 | 6.7 | " |
| -32 | 56 | 15.8 | 10.4 | $NaClO_4.H_2O$ + ice |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The saturation apparatus was similar to that used by Van't Hoff (ref.1) and samples of clear satd sln were taken using a weight-pipette. $Na^+$ was determined as $Na_2SO_4$ by addition of pure sulphuric acid to the sln in silica basins and evaporating at a low red heat. All analyses were carried out in duplicate.

Solid phase compositions were determined using Schreinemakers' method. Gas-heated thermostats were used and thermometers were checked against N.P.L. Standards.

SOURCE AND PURITY OF MATERIALS:

$NaClO_4$ was prepared from very pure ammonium perchlorate (% purity not stated) and an aqueous sln of pure NaOH. Source and other details not given.

ESTIMATED ERROR:

No estimation.

REFERENCES:

1. Van't Hoff, J.H. *Zur Bildung der Ozeanischen Salzablagerungen Wieweg, Braunschweig* 1905, *1*; 1902, *2*.

(continued next page)

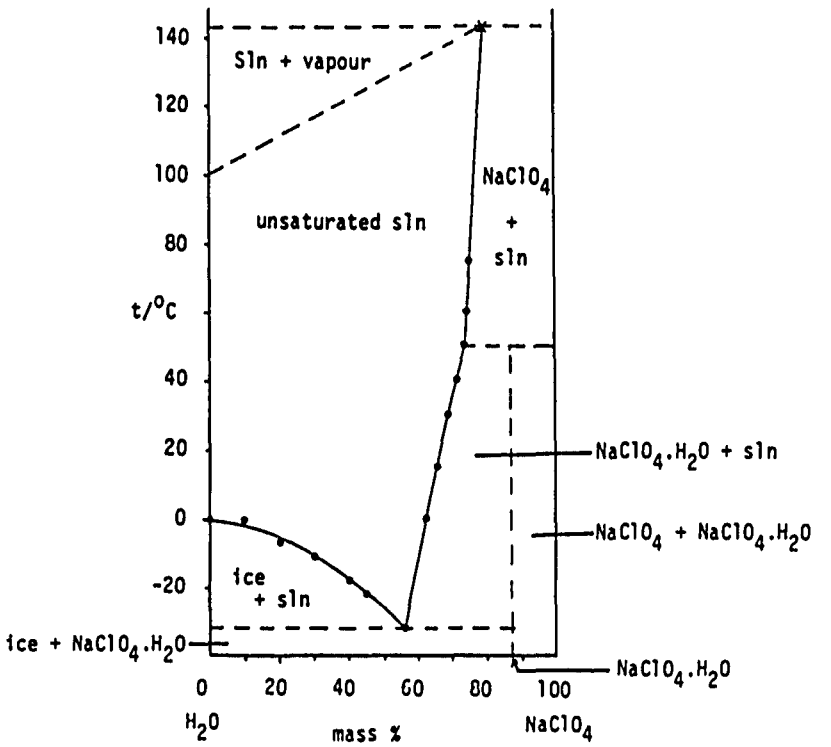
| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Freeth, F.A. |
| (2) Water; $H_2O$; [7732-18-5] | *Recl. Trav. Chim. Pays-Bas* 1924, *43*, 475-507. |

EXPERIMENTAL VALUES:(continued)

COMMENTS AND/OR ADDITION DATA:

The temperature-composition phase diagram for the system $NaClO_4$-$H_2O$ (Pressure = 101325 Pa) is given below.

x - ref. B. Carlson



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Cornec, E.; Dickely, J. |
| (2) Water; $H_2O$; [7732-18-5] | *Bul. Soc. Chim. (France)* 1927, *41*, 1017-27. |
| VARIABLES: Temperature: 273 - 373 K | PREPARED BY: C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of sodium perchlorate in water at various temperatures :

| | Solubility of $NaClO_4$ | | | | density/ | Solid |
|---|---|---|---|---|---|---|
| t/ °C | g $dm^{-3}$ | mass % | mol % [a] | mol $dm^{-3}$ | g $cm^{-3}$ | phase |
| 100 | 1330 | 76.75 | 32.69 | 10.86 | 1.758 | $NaClO_4$ |
| 75 | 1318 | 75.01 | 30.63 | 10.76 | 1.757 | " |
| 55 | 1298 | 73.94 | 29.45 | 10.60 | 1.756 | " |
| 50 | 1281 | 73.26 | 28.73 | 10.46 | 1.749 | $NaClO_4.H_2O$ |
| 38 | 1206 | 70.38 | 25.90 | 9.85 | 1.713 | " |
| 25 | 1142 | 67.63 | 23.67 | 9.33 | 1.683 | " |
| 15 | 1091 | 65.63 | 21.93 | 8.91 | 1.663 | " |
| 0 | - | 62.87 | 19.94 | - | - | " |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

No details of saturation method given. The saturated solutions were evaporated in a water-bath and the solids dried in an oven at 110 °C, cooled and weighed in stoppered flasks.

Transition temperature detn: A mixture (about 40g) of hydrated sodium perchlorate with a small amount of water was put in a wide test-tube placed inside a wider tube, and the whole apparatus was placed in a thermostated oven set at a few degrees under the transition temperature. Then some crystals of the salt were added. The transition temperature which remained steady to ± 0.03 °C for at least 20 min. was measured using a Baudin thermometer.

SOURCE AND PURITY OF MATERIALS: Commercial sodium perchlorate was purified by several recrystallizations before use.

| ESTIMATED ERROR: Not stated. | REFERENCES: |
|---|---|

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Cornec, E.; Dickely, J. |
| (2) Water; $H_2O$; [7732-18-5] | *Bul. Soc. Chim. (France)* 1927, *41*, 1017-27. |

EXPERIMENTAL VALUES: (continued)

COMMENTS AND/OR ADDITIONAL DATA

The transition temperature for the transformation $NaClO_4.H_2O$ = $NaClO_4$ was measured as 52.75 °C.

Compositions of super-saturated aqueous $NaClO_4$ solutions ( solid phase was the anhydrous salt) are as follows :

| t / °C | mass % (1) | mol % (1) [a] |
|---|---|---|
| 38 | 72.83 | 28.28 |
| 25 | 72.21 | 27.66 |
| 15 | 71.68 | 27.14 |

[a] Compiler's calculations.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Carlson, B. |
| (2) Water; $H_2O$; [7732-18-5] | *Festkrift* ed. Klason, P., (Norstedt, Stockholm, 1910), 262-3. |

| VARIABLES: | PREPARED BY: |
|---|---|
| Temperature: 288 K, 323 K and 416 K. | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$H_2O$ at various temperatures :

| | Solubility of $NaClO_4$ | | | | density/ | Solid |
|---|---|---|---|---|---|---|
| t/ °C | g $dm^{-3}$ | mass % | mol % [a] | mol $dm^{-3}$ | g $cm^{-3}$ | phase |
| 15 | 1076 | 64.63 | 21.19 | 8.79 | 1.666 | $NaClO_4.H_2O$ |
| 50 | 1234 | 71.28 | 26.75 | 10.08 | 1.731 | " |
| 143 | 1414 | 79.08 | 35.74 | 11.55 | 1.789 | $NaClO_4$ |

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details given. | Not stated. |
| **ESTIMATED ERROR:** | **REFERENCES:** |
| Not stated. | |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$ [7601-89-0] | Selecki, A.; Tyminski, B.; Mariankowska, B. |
| (2) Water-$d_2$; $D_2O$; [7789-20-0] | *J. Chem. Eng. Data* 1970, *15*, 130-4. |
| VARIABLES: | PREPARED BY: |
| One temperature: 298 - 363 K. | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of $NaClO_4$ in $D_2O$ at various temperatures :

| t/ °C | mol % | molality/ mol $kg^{-1}$ [a] | Refractive Index |
|---|---|---|---|
| 25 | 23.38 ± 0.04 | 15.24 | 1.3892 |
| 30 | 25.1 ± 0.1 | 16.7 | 1.3896 |
| 45 | 27.2 ± 0.2 | 18.7 | 1.3903 |
| 60 | 29.4 ± 0.2 | 20.8 | 1.3903 |
| 75 | 30.1 ± 0.3 | 21.5 | 1.3883 |
| 90 | 30.8 ± 0.2 | 22.2 | 1.3862 |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Method of saturation not given. Solubility was determined by plotting refractive indexes of standard and equilibrated solutions versus concentrations at each temperature, the saturation point being the concentration at which a break occurred in the curve, followed by observation of constant values of the refractive index. Refractive index measurements were made using a RL refractometer (PZO, Warsaw).

Accuracy of measurements was difficult to estimate because of indeterminate errors arising from solvent evaporation from the refractometer prisms.

SOURCE AND PURITY OF MATERIALS:

Heavy water, purity 99.8 mol %, was of Russian manufacture.

The salt was analytical grade and was used without further purification.

ESTIMATED ERROR:

Errors as tabulated were maximum deviations from mean values.

Temperature precision not stated.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Willard, H.H.; Smith, G.F. |
| (2) *Alcohols*: | *J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| (A) Methanol (*methyl alcohol*); $CH_4O$; [67-56-1] | |
| (B) Ethanol (*ethyl alcohol*); $C_2H_6O$; [64-17-5] | |
| (C) 1-Propanol (*n-propyl alcohol*); $C_3H_8O$; [71-23-8] | |
| (D) 1-Butanol (*n-butyl alcohol*); $C_4H_{10}O$; [71-36-3] | |
| (E) 2-Methyl-1-propanol (*iso-butyl alcohol*); $C_4H_{10}O$; [78-83-1] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of sodium perchlorate in various alcohols at 25.00°C, the solid phase being the anhydrous salt :

| soly in : | methanol | ethanol | 1-propanol | 1-butanol | 2-methyl-1-propanol |
|---|---|---|---|---|---|
| mass % | 33.93 | 12.82 | 4.66 | 1.83 | 0.76 |
| g/100 $cm^{-3}$ sln | 35.833 | 11.134 | 3.871 | 1.495 | 0.6264 |
| mol %[a] | 11.85 | 5.243 | 2.34 | 1.12 | 0.47 |
| mol $dm^{-3}$ [a] | 2.927 | 0.9093 | 0.3162 | 0.1221 | 0.05116 |
| mol $kg^{-1}$ [a] | 4.194 | 1.201 | 0.399 | 0.152 | 0.064 |

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the | Anhydrous (1) was prepared by neutralizing pure sodium carbonate with a slight excess of a dilute sln of purified $HClO_4$ and crystallization above 50 °C. After centrifugal separation, the solid was dried at 250 °C (ref.1) in a current of dry air. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) *Alcohols*:<br>(A) Methanol (*methyl alcohol*); $CH_4O$; [67-56-1]<br>(B) Ethanol (*ethyl alcohol*); $C_2H_6O$; [64-17-5]<br>(C) 1-Propanol (*n-propyl alcohol*); $C_3H_8O$; [71-23-8]<br>(D) 1-Butanol (*n-butyl alcohol*); $C_4H_{10}O$; [71-36-3]<br>(E) 2-Methyl-1-propanol (*iso-butyl alcohol*); $C_4H_{10}O$; [78-83-1] | Willard, H.H.; Smith, G.F.<br><br>*J. Am. Chem. Soc.* 1923, *45*, 286-96. |

EXPERIMENTAL VALUES:(continued)

| | methanol | ethanol | 1-propanol | 1-butanol | 2-methyl-1-propanol |
|---|---|---|---|---|---|
| satd sln density/g $cm^{-3}$ | 1.0561 | 0.8685 | 0.8308 | 0.8167 | 0.8031 |
| pure solvent density/g $cm^{-3}$ | 0.78705 | 0.78515 | 0.8026 | 0.8059 | 0.7981 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:(continued)
solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles, making sure that organic solvent was completely removed before the salt was dried to constant weight at 250°C in a current of air dried with $P_2O_5$. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:
Alcohols were purified by refluxing with calcium and fractional distillation.

ESTIMATED ERROR:
Precision in temp. was ±0.01°C.

REFERENCES:
1. Willard, H.H.; Smith, G.F. *J. Am. Chem.Soc.* 1922, *44*, 2816.

| COMPONENTS : | ORIGINAL MEASUREMENTS : |
|---|---|
| (1) Sodium Perchlorate ; $NaClO_4$ : [7601-89-0]<br>(2) a) 1,2-ethanediol (ethylene-glycol) ; $C_2H_6O_2$ ; [107-21-1]<br>b) 2-amino-ethanol *(monoethanol-amine)* ; $C_2H_7NO$ ; [141-43-5] | Isbin, H.S. ; Kobe, K.A.<br>*J. Am. Chem. Soc.* 1945, *67*, 464 |
| VARIABLES<br>one temperature 298 K | PREPARED BY :<br>C.C. Ho |

EXPERIMENTAL VALUES :

| Solvent | g salt/100g solvent[a] | molality[b]/mol kg$^{-1}$ |
|---|---|---|
| 1,2-ethanediol | 75.5 | 6.166 |
| 2-amino-ethanol | 90.8 | 7.416 |

[a] Solid phase at equilibrium not specified

[b] Compiler's calculation

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solvent and excess solid phase were sealed in soft-glass test-tubes and rotated for at least 7 days in a water thermostat at 25°C. Analyses of a series of tubes were made at regular intervals until values became consistent. All analyses were made on a weight basis by use of weighing pipets. The perchlorate was first reduced to the halide by fusing with sodium carbonate in a platinum crucible and determined with both the standard gravimetric detn of halide and the volumetric method, using dichlorofluorescein as indicator. The presence of (2) did not affect the accuracy of the detns. The solid phase was analysed by drying the excess solute on filter paper, weighing, titrating with $HNO_3$ to the methyl orange end-point and determining the halide with $AgNO_3$ to the dichlorofluorescein end-point.

SOURCE AND PURITY OF MATERIALS:

(1) was purified by recrystallization from water above 50°C and dehydrated at 250°C. (2) was technical grade chemical and purified by careful fractionation (ref. 1).

ESTIMATED ERROR:

Temperature: ± 0.08°C.

Solubility : Insufficient information for reliable estimates.

REFERENCES :

1. Reitmeier, R.E. ; Sivertz, V. ; Tartar, H.V. *J. Am. Chem. Soc.* 1940, *62*, 1943-4

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) 1,2-ethanediamine (*ethylenediamine*); $C_2H_8N_2$; [107-15-3] | Isbin, H.S.; Kobe, K.A.<br>*J. Am. Chem. Soc.* 1945, *67*, 464-5 |
| VARIABLES:<br>One temperature: 298 K | PREPARED BY:<br>C.C. Ho |

EXPERIMENTAL VALUES:

Solubility of (1) in (2) at 25°C :

| g salt/100g solvent[a] | molality[b]/mol $kg^{-1}$ |
|---|---|
| 30.1 | 2.458 |

[a] Solid phase at equilibrium is the solvate $NaClO_4.3C_2H_8N_2$.
[b] Compiler's calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
The solvent and excess solid phase were sealed in soft-glass test-tubes and rotated for at least 7 days in a water thermostat at 25°C. Analyses of a series of tubes were made at regular intervals until values were obtained which were within 0.5%. All analyses were made on a weight basis by use of weighing pipets. The perchlorate was first reduced to the halide by fusing with sodium carbonate in a platinum crucible and determined with both the standard gravimetric detn of halide and the volumetric method, using dichlorofluorescein as indicator. The presence of (2) after being neutralised with $HNO_3$, did not affect the accuracy of the detns. The solid phase was analysed by drying the excess solute on filter paper, weighing, titrating with $HNO_3$ to the methyl orange end-point and determining the halide with $AgNO_3$ to the dichlorofluorescein end-point.

SOURCE AND PURITY OF MATERIALS:
(1) was purified by recrystallization from water above 50°C and dehydrated at 250°C. (2) was dehydrated and purified by the method given in ref. 1.

ESTIMATED ERROR:
Temperature: ± 0.08°C.
Solubility : insufficient information for reliable estimates.

REFERENCES:
1. Putnam, G.L.; Kobe, K.A. *Trans. Electrochem. Soc.* 1938, *74*, 609-24.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$ [7601-89-0] | Willard, H.H.; Smith, G.F. |
| (2) Acetone; $C_3H_6O$; [67-64-1] | *J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES: | PREPARED BY: |
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of sodium perchlorate in acetone at 25.00°C :

| mass % | g/100 $cm^{-3}$ sln | mol % | mol $dm^{-3}$ | mol $kg^{-1}$ | satd sln density/g $cm^{-3}$ |
|---|---|---|---|---|---|
| 34.10 | 36.596 | 19.71[b] | 2.9889[b] | 4.226[b] | 1.0732 |

[a] The solid phase was the anhydrous salt.
[b] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles.

The salt was dried to constant wt. at 250°C in a current of air dried with $P_2O_5$, after ensuring that organic solvent was removed completely enough to avoid any danger of explosions.

Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:

Sodium perchlorate was prepared by neutralizing pure $Na_2CO_3$ with a slight excess of a dilute sln of purified $HClO_4$ (ref. 1) and crystallization above 50 °C.

After centrifugal separation, the solid was dried in a current of dry air at 250 °C (ref.2).

Acetone was purified using the bi-bisulfite process and refluxing with powdered potassium hydroxide. Density of (2) at 25°C was 0.7852 g $cm^{-3}$; b.p. 56.16 - 56.51 °C.

ESTIMATED ERROR:

Precision in temp. was ±0.01°C .

REFERENCES:

1. Willard, H.H. *J. Am. Chem. Soc.* 1912, *34*, 1480.
2. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$ [7601-89-0] | Willard, H.H.; Smith, G.F. |
| (2) Ethyl acetate; $C_4H_8O_2$; [141-78-6] | *J. Am. Chem. Soc.* 1923, *45*, 286-96. |
| VARIABLES: | PREPARED BY: |
| One temperature: 298.15 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility[a] of sodium perchlorate in ethyl acetate at 25.00°C :

| mass % | g/100 $cm^{-3}$ sln | mol % | mol $dm^{-3}$ | mol $kg^{-1}$ | satd sln density/g $cm^{-3}$ |
|---|---|---|---|---|---|
| 8.80 | 8.425 | 6.49[b] | 0.6881[b] | 0.788[b] | 0.9574 |

[a] The solid phase was the anhydrous salt.

[b] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 $cm^3$. This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles. The salt was dried to constant wt. at 250°C in a current of air dried with $P_2O_5$, after ensuring that organic solvent was removed completely enough to avoid any danger of explosions. Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.

SOURCE AND PURITY OF MATERIALS:

Sodium perchlorate was prepared by neutralizing pure $Na_2CO_3$ with purified $HClO_4$ (ref.1). The anhydrous salt was obtained by crystallization above 50°C and after centrifugal separation, dried in in a current of dry air at 250°C (ref.2). Ethyl acetate was purified by re-fluxing with $P_2O_5$ and fractional distillation. Its density at 25°C was 0.8945 g $cm^{-3}$ ; and b.p. 77.14 - 77.16 °C.

ESTIMATED ERROR:

Precision in temp. was ±0.01°C .

REFERENCES:

1. Willard, H.H.; *J. Am. Chem. Soc.* 1912, *34*, 1480.
2. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1922, *44*, 2816.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Krumgal'z, B.S.; Smirnova, V.A.; Gerzhvert, Yu. I. |
| (2) 2-butanone (*methyl ethyl ketone*); $C_4H_8O$; [78-93-3] | *Zh. Neorg. Khim.* 1972, *17*, 1778-80; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1972, *17*, 924. |
| VARIABLES: One temperature: 283-323 K | PREPARED BY: C.C. Ho |

EXPERIMENTAL VALUES:

Solubility of (1) in (2) at various temperatures:

| t/°C | Liquid phase composition[a] molality/mol $kg^{-1}$ |
|---|---|
| 10 | 1,944 |
| 20 | 1.836 |
| 25 | 1.790 |
| 30 | 1.749 |
| 40 | 1.682 |
| 50 | 1.630 |

[a] Solid phase at equilibrium was $NaClO_4$ over the temperature range investigated.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The method and apparatus were as described in ref. 1. | No details given. |
| | ESTIMATED ERROR: Nothing specified. |
| | REFERENCES: 1. Krumgal'z, B.S.; Gerzhvert, Yu. I; Nikitina, I.P.; Derevskaya, V.I.; Fedotova, G.F.; Traber, D.G. *Zh. Prikl. Khim.* 1969, *42*, 1414. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Acetone; $C_3H_6O$; [67-64-1] | Krumgal'z, B.S.; Smirnova, V.A.; Gerzhvert, Yu. I.<br><br>*Zh. Neorg. Khim.* 1972, *17*, 1778-80; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1972, *17*, 924. |
| VARIABLES:<br>One temperature: 283-323 K | PREPARED BY:<br>C.C. Ho |

EXPERIMENTAL VALUES:

Solubility of (1) in acetone at various temperatures:

| $t/^{\circ}C$ | Liquid phase composition[a] molality/mol $kg^{-1}$ |
|---|---|
| 10 | 4.242 |
| 20 | 4.346 |
| 25 | 4.422 |
| 30 | 4.508 |
| 40 | 4.604 |
| 50 | 4.666 |

[a] Solid phase at equilibrium was $NaClO_4$ over the temperature range investigated.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The method and apparatus were as described in ref. 1. | No details given. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES:<br>1. Krumgal'z, B.S.; Gerzhvert, Yu. I; Nikitina, I.P.; Derevskaya, V.I.; Fedotova, G.F.; Traber, D.G. *Zh. Prikl. Khim.* 1969, *42*, 1414. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Paul, R.C.; Dev, R. |
| (2) Acetamide; $C_2H_5NO$; [60-35-5] | *Res. Bulletin Panjab Univ.* 1969, *20*, 139-48. |
| VARIABLES: | PREPARED BY: |
| One temperature: 373 K. | C.C. Ho |

EXPERIMENTAL VALUES:

Solubility of (1) in (2) at 100°C :

| g salt/100g solvent | molality[a]/mol $kg^{-1}$ |
|---|---|
| 12.46 | 1.018 |

[a] Compiler's calculations.

COMMENTS/ADDITIONAL DATA:

The solubility of sodium perchlorate in molten acetamide is strikingly similar to that in water. A fairly high dielectric constant of acetamide is responsible for the ionization of the salt dissolved in it. The ions formed are assumed to be similar to those obtained in aqueous solution.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Solubility was determined by preparing the saturated solution in molten acetamide. The tubes containing the solvent and the solute were sealed and shaken continuously for several hours in a bath at 100°C. The cation and anion estimated using standard methods. No details were given.

SOURCE AND PURITY OF MATERIALS:

(1) and (2) were purified as described in ref. 1 and 2.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Paul, R.C.; Dev, R. *Ind. J. Chem.* 1965, *3*, 315.
2. Paul, R.C.; Dev, R. *Ind. J. Chem.* 1967, *5*, 267.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Kenttamaa, J. |
| (2) Sulfinylbis-methane ( *dimethyl sulphoxide, DMSO* ); $C_2H_6OS$; [67-68-5] | *Suomen Chemist* 1960, *33*, 179-82. |
| VARIABLES: | PREPARED BY: |
| Temperature: 298, 308 and 318 K. | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of sodium perchlorate in DMSO at various temperatures :

( Solid phase was the anhydrous salt. )

| t / °C | mol/100 g(2) | mol $kg^{-1}$ |
|---|---|---|
| 25 | 0.18 | 1.8 |
| 35 | 0.17 | 1.7 |
| 45 | 0.21 | 2.1 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The soly measurements and the preparation of solvates was carried out in glass-stoppered flasks immersed in a thermostat at an initial temperature of 50 °C. The flasks were shaken from time to time to mix the contents. About 2 weeks were allowed for the attainment of equilibrium, after which the temperature was lowered to 45, 35, or 25 °C as required. After one more week of equilibration, the solutions were analysed. After the soly measurements at 25 °C, the solids in the flasks were filtered and washed with pure DMSO. Excess DMSO was removed from the solids in a vacuum of about 0.05 torr and trapped in a U-tube immersed in a dry ice-acetone mixture, care being taken to isolate the solvates from atmospheric moisture. The solutions were analysed for sodium by flame photometry, using a Beckman 4100 flame photometer. All analyses were carried out in duplicate or triplicate to an accuracy of about ± 5 %. The solvates were also analysed using flame photometry and ion exchange methods (no details).

SOURCE AND PURITY OF MATERIALS:

DMSO of "practical quality" was purified by repeated recrystallization. The melting point of the final product was 18.5 °C. The salt was dried in a heating cabinet for a few days at a temperature high enough to remove any moisture. No details of salt purity.

ESTIMATED ERROR:

Soly precision: about ± 5 % .

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Starkovich, J.A.; Janghorbani, M. |
| (2) Tetrahydrothiophene 1,1-dioxide (*sulfolane*); $C_4H_8O_2S$; [126-33-0] | *J. Inorg. Nucl. Chem.* 1972, *34*, 789-91. |
| VARIABLES: | PREPARED BY: |
| One temperature: 313 K | C.C. Ho |

EXPERIMENTAL VALUES:

The solubility of (1) in sulfolane at 40°C was 0.981 mol $dm^{-3}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small amount of (1) sufficient to saturate 3 $cm^3$ of the solvent was placed in 5 $cm^3$ glass ampoules and (2) added. The ampoules were sealed and placed in a water bath maintained at 40.0 ± 0.5°C. The soln was allowed to equilibrate for 250-300h with periodic shaking. At the end of the equilibration period, the ampoules were opened and a 1 $cm^3$ aliquot of each supernatant soln was transferred in the open atmosphere to a clean 1/2 dram polyvial. The 1/2 dram polyvials were sealed and placed inside 2 dram polyvials for activation. Solubility was determined by subjecting the perchlorate sample to neutron irradiation and subsequently measuring the amount of induced $^{38}Cl$ activity. Samples were activated twice for 30 min. each time at thermal neutron fluxes of $2.8 \times 10^{10}$ and $5.6 \times 10^{9}$ neutrons $cm^{-2}$ $s^{-1}$ in the Oregon State University Triga reactor. A set of 3 $NH_4Cl$ standards was used to obtain a calibration plot of $^{38}Cl$ activity vs. perchlorate concentration. After each

SOURCE AND PURITY OF MATERIALS:

Sulfolane (Shell Chem.) was twice distilled below 100°C in vacuo. The solvent prepared in this way was found to contain <0.02% by wt water as determined by a Fisher titration (ref. 1). The salt used was of reagent grade quality or purified according to published procedures (ref. 2).

ESTIMATED ERROR:

Temperature: ± 0.5°C.

Solubility : relative standard deviations range between 2 and 6% which is what may be expected from INNA techniques without elaborate sample handling.

REFERENCES:

1. Mitchell, J.; Smith, D.M. *Aquametry*, Interscience, N.Y., 1948, 65-78.
2. Mann, C.K. *Electroanalytical Chemistry*, Marcel Dekker, N.Y., 1969, 132-4.

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Starkovich, J.A.; Janghorbani, M. |
| (2) Tetrahydrothiophene 1,1-dioxide (*sulfolane*); $C_4H_8O_2S$; [126-33-0] | *J. Inorg. Nucl. Chem.* 1972, *34*, 789-91. |

METHOD/APPARATUS/PROCEDURE: (continued)

activation the 1/2 dram polyvials were placed in new 2 dram polyvials and counted with a 3 in x 3 in NaI(Tl) well detector coupled to a 400 channel analyzer. The 1.64 and 2.16 MeV photopeak areas of 37-min $^{38}Cl$ were corrected for Compton scattering and decay and both peaks were used generally for analysis. Where interferences were noted, only one $\gamma$-ray was used. The number of perchlorate ions per formula unit was taken into consideration in calculating the salt solubilities.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$ [13454-84-7] | Rosolovskii, V.Ya.; Sakk, Zh.G. |
| (2) Hydrazine; $N_2H_4$; [302-01-2] | *Zh. Neorg. Khim.* 1970, *15*, 2262-4; **Russ. J. Inorg. Chem. (Engl. Transl.)* 1970, *15*, 1169-70. |
| VARIABLES: | PREPARED BY: |
| One temperature: 273.2 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of sodium perchlorate in hydrazine at 0.0 °C was reported as 74.3 g(1)/100 g(2). The corresponding mol % and molality values are 16.3 % and 6.07 mol $kg^{-1}$, respectively (calculated by compiler). The solid phase was reported to be $NaClO_4.N_2H_4$ [22475-89-4].

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| A sln of sodium perchlorate in hydrazine with the solid phase present was stirred for 2 h continuously in a thermostat at 0 °C. The liquid and solid phases were separated and analysed for $ClO_4^-$ by precipitation as nitron perchlorate. Hydrazine in both phases was analysed by titration with satd iodine solution in the presence of excess sodium bicarbonate. | Sodium perchlorate was obtained by reacting 70 % $HClO_4$ aq. sln with sodium carbonate and recrystallized twice. It was dried to constant wt. in a vacuum at 200 - 250 °C . Hydrazine was 99.5 - 98.8 % pure. |
| | ESTIMATED ERROR: Temperature precision: ± 0.1 °C. Insufficient details for soly error estimation. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$ [13454-84-7]<br>(2) Hydrazine; $N_2H_4$; [302-01-2] | Sakk, Zh.G.; Rosolovskii, V.Ya.<br><br>*Zh. Neorg. Khim.* 1972, *17*, 1783-4;<br>**Russ. J. Inorg. Chem. (Engl. Transl.)* 1972, *17*, 927-8. |
| VARIABLES: | PREPARED BY: |
| One temperature: 298.2 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of sodium perchlorate in hydrazine at 25.0 °C was reported as 85.1 g(1)/100 g(2). The corresponding mol % and molality values are 18.22 % and 6.95 mol $kg^{-1}$, respectively (calculated by compiler).

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| 4-6 g of the salt and 8-11 $cm^3$ of hydrazine were thermostated at 25°C for 7-8h with continuous stirring in a vessel isolated from atmospheric moisture. Samples for analysis were removed by withdrawing solution and part of the solid phase into another vessel fitted with a porosity no. 4 filter at reduced pressure. After separating the phases, the solution was analysed for hydrazine using the procedure given in ref. 1.<br>Replicate solubility determinations were carried out. | The methods of purification of the perchlorate and of the preparation of anhydrous hydrazine were as described in ref. 1.<br>Salt purity was about 99.5-99.9 %. |
| | ESTIMATED ERROR:<br>Absolute error in soly value was 0.4 %.<br>Precision in temp. was ±0.1 °C . |
| | REFERENCES:<br>1. Rosolovskii, V.Ya.; Sakk, Zh.G. *Zh. Neorg. Khim.* 1970, *15*, 2262. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Perchloric acid; $HClO_4$; [7601-90-3] | Rosolovskii, V.Ya.; Kristov, N.V.; Lemesheva, D.G.<br><br>*Zh. Neorg. Khim.* 1968, *13*, 1115-8; **Russ. J. Inorg. Chem. (Engl. Transl.)* 1968, *13*, 582-4. |
| VARIABLES:<br>Temperature: 273 K. | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of sodium perchlorate in anhydrous perchloric acid at 0 °C, the solid phase being the anhydrous salt :

| mass % (1) | g(1)/100 g(2) | mol %[a] | molality[a]/mol $kg^{-1}$ |
|---|---|---|---|
| 0.624 | 0.628 | 0.512 | 0.0513 |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Mixtures of 5-6 g of the salt with 10-12 g of the acid were kept in a thermostat at 0 °C (melting ice) for 10-15 h with continuous stirring in tubes isolated from atmospheric moisture. After equilibrium has been reached, the solid and liquid phases were separated on a glass filter. Perchlorate was determined gravimetrically as nitron perchlorate. The purity of the acid before and after saturation was determined by acid-base titration. Concentration of the salt in the satd sln was determined by first diluting a weighed sampple of the sln (5-8 g) in water and then evaporating to dryness. The dry residue was then dissolved in concentrated sulphuric acid, evaporated to dryness and the resulting sulfate heated to constant weight at 600 °C. Triplicate determinations were made.

SOURCE AND PURITY OF MATERIALS:

$NaClO_4$ was prepared by dissolving $Na_2CO_3$ in 70 % $HClO_4$, recrystalling twice from water, and drying to constant weight under vacuum at 200-250 °C. Analysis : Na 18.81 %, $ClO_4$ 81.45 %.

Anhydrous $HClO_4$ was distilled from a mixture of oleum and perchloric acid dihydrate at 100 °C under vacuum (ref. 1). Acid purity was 99.79 ± 0.01 % (w/w).

ESTIMATED ERROR:

Precision in soly value : ± 0.3 %.
Temperature precision not stated.

REFERENCES:

(1) Rosolovskii, Y. Ya. *Khimiya Bezvodnoi Khlornoi Kisloty*, ( Moscow, 1966 ).

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Titova, K.V.; Kolmakova, E.I.; Rosolovskii, V.Ya. |
| (2) Hydrogen peroxide; $H_2O_2$; [7722-84-1] | *Zh. Neorg. Khim.* 1986, *31*, 3213-5; **Russ. J. Inorg. Chem.* ( *Engl. Transl.* ) 1986, *31*, 1846-7. |
| VARIABLES: | PREPARED BY: |
| One temperature: 273 K | C.Y. Chan |

EXPERIMENTAL VALUES:

The solubility[a] of sodium perchlorate in hydrogen peroxide at 0 °C :

| g(1)/ 100 g(2) | mass % | mol % | molality/ mol $kg^{-1}$ |
|---|---|---|---|
| 42.12 | 29.64 | 10.48 | 3.440 |

[a] Mass%, mol% and molality values calculated by compiler.
The solid phase was an unstable solvate.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details of saturation method was given. Solubility equilibrium was established in 1-1.5 h. The concencentration of the solutions did not change noticeably during the next 3h but after that slow decomposition of peroxide began. The concentration of perchlorate in the satd solution was determined from the mass of the solid residue left after removal of the solvent from a sample of the solution under vacuum. | Sources not given.<br>The $H_2O_2$ was 99.8% ± 0.2% pure.<br>No information on purity of salt. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Marshall, P.R.; Hunt, H. |
| (2) Ammonia; $NH_3$; [7664-41-7] | *J. Chem. Eng. Data* 1959, *4*, 217-22. |
| VARIABLES: | PREPARED BY: |
| Temperature: 240 - 323 K. | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of $NaClO_4$ in $NH_3$ at various temperatures, the solid phase being the anhydrous salt :

| t/ °C | g(1)/ 100g(2) | mol %[a] | molality/ mol $kg^{-1}$ |
|---|---|---|---|
| -33 | 278.3 | 27.91 | 22.7 |
| 0 | 304.3 | 29.74 | 24.8 |
| 25 | 318.3 | 30.69 | 26.0 |
| 50 | 328.0 | 31.33 | 26.75 |

[a] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility determinations were carried out using a specially constructed apparatus (diagram given in original paper), involving a gas line connected to the saturation cell. The cell consisted of two compartments separated by a sintered glass partition, the larger one of which was connected to the gas line in such a way that the cell could be inverted, with either one of the compartments vertically above the other. Weighed amts of the salt were sealed in the smaller compartment of the cell which was then connected to the gas line via the larger compartment. Excess of dry ammonia was condensed in the cell until the salt had completely dissolved at the set temperature. Coolants used were dry ice and $CCl_4$. The cell was thermostated in a liquid $NH_3$ bath for -33 °C determinations, in an ice + water bath for 0 °C, and in a water bath for the other temperatures. Ammonia was bled from the solution until salt crystals were formed, and the cell inverted so that the solution filtered through the partition into the larger compartment. After filtration the ammonia in the solution was all removed by condensation into a reservoir in the apparatus and determined quantitatively by absorption in std. HCl sln and back-titrated with std. base. The cell was then opened and the solids removed for analysis.

SOURCE AND PURITY OF MATERIALS:

Not stated. Ammonia was dried with sodium.

ESTIMATED ERROR: Reproducibility (3 detn) is within ± 2 % of the mean value in most cases.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Abdukarimova, F.M.; Nogoev, K; Sulaimankulov, K. |
| (2) Carbamide (*urea*); $CH_4N_2O$; [57-13-6] | *Zh. Neorg. Khim.* 1973, *18*, 3102-6; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1973, *18*, 1651-3. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 303 K | C.C. Ho |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$CO(NH_2)_2$-$H_2O$ at 30°C:

| Liquid phase composition | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|
| mass % | | mol % | | molality[a]/mol kg$^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 57.50 | - | 28.87 | - | 22.53 | A |
| 7.86 | 54.09 | 1.91 (2.09)[c] | 28.61 (29.27) | 1.687 | 23.67 | A |
| 14.00 | 51.61 | 3.80 (3.97) | 29.74 (29.81) | 3.325 | 24.99 | A |
| 17.91 | 50.64 | 5.16 (5.35) | 30.98 (30.83) | 4.651 | 26.81 | A |
| 24.04 | 49.10 | 7.60 (7.84) | 32.79 (32.64) | 7.310 | 30.44 | A |
| 31.49 | 49.60 | 11.71 (12.06) | 38.96 (38.73) | 13.60 | 43.68 | A |
| 37.50 | 52.29 | 17.63 (17.56) | 50.00 (49.93) | 30.00 | 85.28 | A |
| 37.57 | 52.46 | 17.80 (17.70) | 50.82 (50.38) | 30.78 | 87.62 | A + B |
| 37.77 | 51.96 | 17.75 (17.69) | 49.47 (49.62) | 30.04 | 84.25 | B |
| 39.13 | 43.81 | 16.07 (16.01) | 36.62 (36.55) | 18.73 | 42.76 | B |
| 42.44 | 37.48 | 16.61 (16.62) | 30.00 (29.93) | 17.26 | 31.08 | B |
| 46.52 | 31.19 | 17.80 (17.78) | 24.38 (24.31) | 17.05 | 23.30 | B |

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details were given. | Nothing specified. |
| | ESTIMATED ERROR: Nothing specified. |
| | REFERENCES: |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Abdukarimova, F.M.; Nogoev, K; Sulaimankulov, K. |
| (2) Carbamide (*urea*); $CH_4N_2O$; [57-13-6] | *Zh. Neorg. Khim.* 1973, *18*, 3102-6; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1973, *18*, 1651-3. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

| Liquid phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|
| mass % | | mol % | | molality[a]/mol $kg^{-1}$ | | phase[b] |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 52.43 | 25.72 | 20.61 (20.69)[c] | 20.76 (20.70) | 19.60 | 19.60 | B |
| 62.40 | 19.02 | 37.62 (27.43) | 23.50 (17.05) | 27.43 | 17.05 | B |
| 62.76 | 19.34 | 28.30 (28.04) | 16.90 (17.61) | 28.64 | 17.99 | B + C |
| 62.35 | 19.28 | 27.40 (27.53) | 17.30 (17.35) | 27.72 | 17.48 | B + C |
| 62.55 | 19.36 | 28.00 (27.80) | 16.87 (17.54) | 28.24 | 17.82 | C |
| 64.26 | 9.96 | 24.83 (24.74) | 7.86 (7.82) | 20.36 | 6.43 | C |
| 66.12 | 3.83 | 23.88 (23.77) | 2.83 (2.81) | 17.97 | 2.12 | C |
| 68.71 | - | 24.4 | - | 17.94 | - | C |

[a] Compiler's calculations. [b] A = $CO(NH_2)_2$;
B = $NaClO_4.2CO(NH_2)_2$;
C = $NaClO_4.H_2O$.

[c] Values in brackets are compiler's calculations.

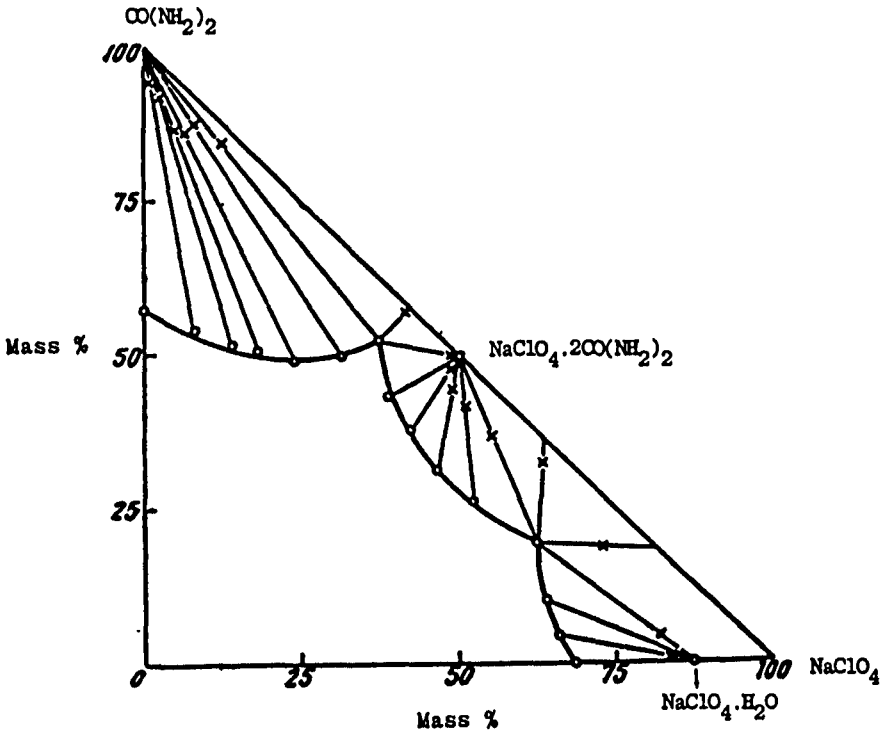
COMMENTS/ADDITIONAL DATA

The solubility curve of the ternary system (see Figure) shows that in addition to the initial components, an anhydrous compound with sodium perchlorate:urea ratio of 1:2 crystallises out. The branch of the curve which belongs to this compound occupies a large part of the diagram and extends in the range 19.02- 51.96% urea and 37.77-62.40% sodium perchlorate. The straight line rays which run from this branch of the solubility curve meet at a single point of the diagram corresponding to a solid phase with the composition $NaClO_4.2CO(NH_2)_2$.

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Abdukarimova, F.M.; Nogoev, K; Sulaimankulov, K. |
| (2) Carbamide (urea); $CH_4N_2O$; [57-13-6] | *Zh. Neorg. Khim.* 1973, *18*, 3102-6; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1973, *18*, 1651-3. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Abdukarimova, F.M.; Nogoev, K; Sulaimankulov, K. |
| (2) Thiocarbamide (*thiourea*); $CH_4N_2S$; [62-56-6] | *Zh. Neorg. Khim.* 1973, *18*, 3102-6; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1973, *18*, 1651-3. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 303 K | C.C. Ho |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$CS(NH_2)_2$-$H_2O$ at 30°C:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol % | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 18.20 | - | 5.27 (5.00)[b] | - | 2.923 | $CS(NH_2)_2$ |
| 12.67 | 16.35 | 2.42 (2.43) | 5.31 (5.04) | 1.458 | 3.026 | " |
| 23.52 | 12.03 | 4.87 (4.89) | 4.24 (4.02) | 2.981 | 2.452 | " |
| 33.78 | 9.06 | 7.71 (7.73) | 3.51 (3.34) | 4.827 | 2.082 | " |
| 47.89 | 8.01 | 13.24 (13.28) | 3.76 (3.57) | 8.869 | 2.386 | " |
| 60.56 | 5.07 | 19.97 (20.03) | 2.83 (2.70) | 14.39 | 1.938 | " |
| 67.02 | 3.60 | 24.54 (24.60) | 2.25 (2.13) | 18.63 | 1.610 | " |
| 67.50 | 3.51 | 25.06 (24.98) | 2.21 (2.09) | 19.02 | 1.591 | $CS(NH_2)_2$ + $NaClO_4.H_2O$ |
| 68.06 | 2.80 | 25.11 (25.15) | 1.76 (1.66) | 19.08 | 1.262 | $NaClO_4.H_2O$ |
| 68.45 | 1.23 | 24.73 (24.76) | 0.75 (0.72) | 18.44 | 0.533 | " |
| 68.71 | - | 24.40 (24.42) | - | 17.94 | - | " |

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details were given. | Nothing specified. |
| | ESTIMATED ERROR: Nothing specified. |
| | REFERENCES: (continued next page) |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Abdukarimova, F.M.; Nogoev, K; Sulaimankulov, K. |
| (2) Thiocarbamide (*thiourea*); $CH_4N_2S$; [62-56-6] | *Zh. Neorg. Khim.* 1973, *18*, 3102-6; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1973, *18*, 1651-3. |
| (3) Water; $H_2O$; [7732-18-5] | |

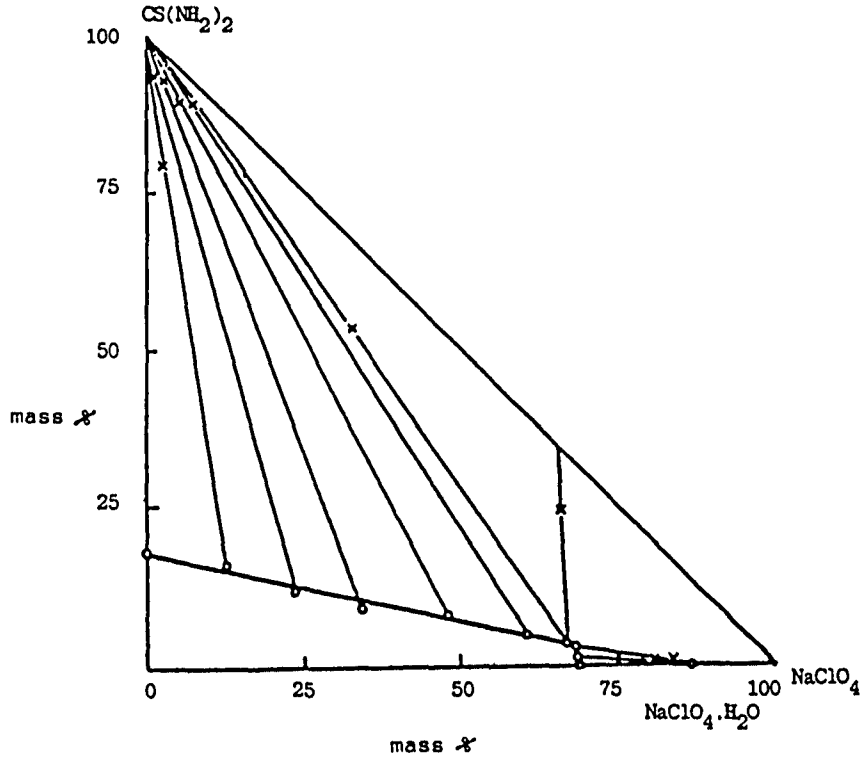
EXPERIMENTAL VALUES: (continued)

[a] Compiler's calculations.

[b] Values in brackets are compiler's calculations. The original values appeared to be in error.

COMMENTS/ADDITIONAL DATA

The solubility isotherm of the ternary system (see Figure) consists of two branches: one corresponding to $CS(NH_2)_2$ as solid phase in equilibrium and the other to $NaClO_4.H_2O$ as solid phase. The composition of the eutonic solution, where crystals of thiourea and sodium perchlorate monohydrate are in equilibrium, is characterized by the following mean composition of initial substances: 3.51% thiourea and 67.50% sodium perchlorate.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Karnaukhov, A.S.; Tarakanov, V.F. |
| (2) Thiocarbamide; $CS(NH_2)_2$; [62-56-6] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1971, *95*, 109-12. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298 K | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$CS(NH_2)_2$-$H_2O$ at 298 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol % | | molality/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 0.00 | 14.70 | 0.00 | 3.92 | 0.000 | 2.264 | $CS(NH_2)_2$ |
| 21.79 | 11.44 | 4.41 | 3.72 | 2.665 | 2.251 | " |
| 47.93 | 8.20 | 13.34 | 3.67 | 8.923 | 2.455 | " |
| 61.20 | 4.48 | 20.29 | 2.39 | 14.564 | 1.715 | " |
| 64.79 | 3.84 | 22.80 | 2.17 | 16.868 | 1.608 | $CS(NH_2)_2$ + $NaClO_4.H_2O$ |
| 67.89 | 0.00 | 23.73 | 0.00 | 17.268 | 0.000 | $NaClO_4.H_2O$ |

Solution composition at the isothermal double saturation point ( solid phases $NaClO_4.H_2O$ and $CS(NH_2)_2$ ):
64.79 mass % $NaClO_4$, 3.84 mass % $CS(NH_2)_2$, and 31.37 mass % $H_2O$.

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method. Details of saturation technique are not given. The composition of solid phases was determined by Schreinemakers' method of "residues". Thiocarbamide was determined by the Kjeldahl method; $ClO_4^-$ gravimetrically, by nitron precipitation. | Not stated. |
| | ESTIMATED ERROR: Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Lepeshkov, I.N.; Tarakanov, V.F. |
| (2) Acetamide; $C_2H_5NO$; [60-35-5] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1972, *103*, 78-82. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298 K | I.S. Bodnya |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$CH_3CONH_2$-$H_2O$ at 298 K:

| Liquid phase composition | | | | Solid phase |
|---|---|---|---|---|
| mass % | | mol % | | |
| (1) | (2) | (1) | (2) | |
| 67.89 | - | 23.73 | - | $NaClO_4.H_2O$ |
| 62.10 | 9.70 | 22.68 | 7.34 | " |
| 58.21 | 20.56 | 23.75 | 17.39 | " |
| 54.07 | 31.00 | 24.60 | 29.23 | " |
| 48.22 | 45.14 | 25.80 | 50.06 | $NaClO_4$ |
| 46.03 | 50.59 | 26.47 | 60.31 | " |
| 32.90 | 65.10 | 18.13 | 74.37 | $CH_3CONH_2$ |
| 23.75 | 65.16 | 10.14 | 57.67 | " |
| 11.00 | 68.35 | 3.75 | 48.35 | " |
| - | 71.24 | - | 43.03 | " |

[a] Compiler's calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The soly was studied by the method of isothermal recrystallization. Addition of sodium perchlorate monohydrate to the saturated solution of acetamide resulted in dissolution of the solid phase. To avoid this, dried acetamide was taken or anhydrous sodium perchlorate was added. Acetamide was determined by the Kjeldahl method; perchlorate ion by the gravimetric method with nitron. The density, viscosity and electric conductivity of the saturated solutions were measured. | Not stated. |
| | **ESTIMATED ERROR:** Not stated. |
| | **REFERENCES:** |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sulfinylbis-methane ( *dimethyl sulphoxide, DMSO* ); $C_2H_6OS$; [67-68-5]<br>(3) Water; $H_2O$; [7732-18-5] | Ferroni, G.; Villetard, M.<br><br>*Ann. Chim.* 1975, *10*, 33-5. |
| VARIABLES:<br>Temperature: 298.2 K .<br>Composition. | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of sodium perchlorate in mixtures of water and DMSO at 25.0 °C, the solid phase being $NaClO_4.H_2O$ :

| Solvent composition mol ratio, DMSO:$H_2O$ | Solubility g/100 $cm^3$ | Solubility mol $dm^{-3}$ |
|---|---|---|
| 2:1 | 23.27 | 1.90 |
| 1:1 | 25.59 | 2.09 |
| 1:2 | 34.53 | 2.82 |
| 1:5 | 60.12 | 4.91 |
| 1:10 | 82.12 | 6.71 |
| pure DMSO | 22.41 | 1.83 |
| pure $H_2O$ | 181.22 | 14.8 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
No details of saturation method. Excess of the salt was allowed to saturate in 50 $cm^3$ of solvent for 8 days at 25 °C. The saturated slns were first eluted through acidic cation exchange resins and the eluants analysed by potentiometric titration ( glass electrode used ) with NaOH sln.

SOURCE AND PURITY OF MATERIALS:
Anhydrous $NaClO_4$ was obtained by heating the monohydrate (Merck product) gradually, then by fusing to remove water of crystallization and further dried at 120 °C. Analytical grade DMSO was further purified (ref. 1) before use.

ESTIMATED ERROR:
Temperature precision: ± 0.1 °C.
Precision in soly detn not stated.

REFERENCES:
1. Butler, J.N. *J. Electroanalytical. Chem.* 1967, *14*, 89.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Bestuzheva, M.M. |
| (2) Dimethylurea; $C_3H_8ON_2$; [1320-50-9] | *Sb. Tr.Yarosl. Gos. Ped. Inst.* 1979, *178*, 67-9. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 298 K | I.S. Bodnya |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$C_3H_8ON_2$-$H_2O$ at 298 K:

| Liquid phase composition | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 67.84 | - | 23.69 | - | 17.228 | - | A |
| 67.24 | 1.89 | 24.04 | 0.94 | 17.790 | 0.695 | " |
| 65.27 | 3.81 | 23.25 | 1.89 | 17.240 | 1.398 | A + B |
| 65.19 | 3.85 | 23.20 | 1.90 | 17.197 | 1.411 | " |
| 65.04 | 4.16 | 23.22 | 2.06 | 17.247 | 1.533 | B |
| 55.99 | 6.89 | 17.62 | 3.01 | 12.319 | 2.107 | " |
| 49.50 | 10.50 | 14.73 | 4.34 | 10.107 | 2.979 | " |
| 43.29 | 14.38 | 12.33 | 5.69 | 8.352 | 3.855 | " |
| 39.43 | 16.51 | 10.90 | 6.34 | 7.309 | 4.253 | " |
| 37.05 | 23.56 | 10.98 | 9.70 | 7.682 | 6.788 | " |
| 37.07 | 23.50 | 10.98 | 9.67 | 7.678 | 6.764 | B + C |
| 37.01 | 23.42 | 10.93 | 9.61 | 7.639 | 6.717 | C |
| 28.08 | 22.83 | 7.14 | 8.06 | 4.672 | 5.278 | " |
| 24.31 | 22.60 | 5.84 | 7.54 | 3.740 | 4.831 | " |
| 16.64 | 22.42 | 3.60 | 6.74 | 2.230 | 4.175 | " |
| 12.94 | 22.30 | 2.67 | 6.40 | 1.632 | 3.908 | " |
| 9.90 | 21.71 | 1.96 | 5.98 | 1.182 | 3.603 | C |
| 5.43 | 22.16 | 1.03 | 5.83 | 0.612 | 3.473 | " |
| - | 23.55 | - | 5.92 | - | 3.496 | " |

[a] Compiler's calculations.

[b] A = $NaClO_4.H_2O$; B = $NaClO_4.2C_3H_8ON_2.H_2O$; C = $C_3H_8ON_2$

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal recrystallization was used. Periods of equilibration vary from 2 to 3 days. Perchlorate ion was determined gravimetrically with nitron, dimethylurea by Kjeldahl's method. The density and viscosity of saturated solutions were determined. | Not stated. |
| | ESTIMATED ERROR: |
| | Not stated. |

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Sal'nikova, L.N.; Karnaukhov, A.S.; Lepeshkov, I.N. |
| (2) Hexamethylenetetramine; $C_6H_{12}N_4$; [100-97-0] | *Zh. Neorg. Khim.* 1971, *16*, 2840-4; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1971, *16*, 1511-3. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 298 K | C.C. Ho |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$C_6H_{12}N_4$-$H_2O$ at 25°C:

| Liquid phase composition | | | | | | | Solid[c] |
|---|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | density[b] | phase |
| (1) | (2) | (1) | (2) | (1) | (2) | | |
| - | 46.52 | - | 10.05 | - | 6.205 | 1.1126 | A |
| 2.29 | 46.20 | 0.583 | 10.28 | 0.363 | 6.398 | 1.1250 | A |
| 6.83 | 45.18 | 1.834 | 10.60 | 1.162 | 6.716 | 1.1480 | A |
| 9.95 | 44.10 | 2.758 | 10.68 | 1.769 | 6.846 | 1.1832 | A |
| 16.35 | 43.46 | 4.993 | 11.59 | 3.323 | 7.714 | 1.3217 | A + B |
| 19.67 | 39.78 | 5.960 | 10.53 | 3.962 | 6.998 | 1.2510 | B |
| 21.43 | 38.70 | 6.569 | 10.36 | 4.390 | 6.924 | - | B |
| 22.94 | 37.02 | 7.007 | 9.88 | 4.679 | 6.595 | 1.2764 | B |
| 30.34 | 28.53 | 9.062 | 7.443 | 6.025 | 4.948 | 1.3241 | B |
| 36.63 | 23.97 | 11.26 | 6.435 | 7.593 | 4.340 | - | B |
| 38.44 | 21.86 | 11.74 | 5.83 | 7.908 | 3.928 | 1.3810 | B |
| 43.99 | 17.75 | 13.77 | 4.85 | 9.390 | 3.309 | - | B |
| 47.71 | 15.74 | 15.40 | 4.44 | 10.66 | 3.072 | 1.8382 | B |
| 52.72 | 13.50 | 17.93 | 4.01 | 12.75 | 2.851 | - | B |
| 54.47 | 13.67 | 19.25 | 4.22 | 13.96 | 3.061 | 2.2410 | B + C |
| 56.28 | 12.04 | 19.95 | 3.73 | 14.51 | 2.711 | 1.6005 | C |
| 59.50 | 9.37 | 21.31 | 2.93 | 15.61 | 2.147 | 1.5907 | C |

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Equilibrium was reached after 20 days. The solid phases were analysed for $ClO_4^-$ ion (determined as nitron perchlorate) and $C_6H_{12}N_4$, by acid hydrolysis followed by distillation of the ammonia into saturated boric acid soln and titration with $H_2SO_4$. The density was measured pyknometrically in benzene. | Nothing specified. |
| | ESTIMATED ERROR: Nothing specified. |
| | REFERENCES: |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Sal'nikova, L.N.; Karnaukhov, A.S.; Lepeshkov, I.N. |
| (2) Hexamethylenetetramine; $C_6H_{12}N_4$; [100-97-0] | *Zh. Neorg. Khim.* 1971, *16*, 2840-4; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1971, *16*, 1511-3. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

| Liquid phase composition | | | | | | | Solid[c] |
|---|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | density[b] | phase |
| (1) | (2) | (1) | (2) | (1) | (2) | | |
| 61.72 | 7.97 | 22.47 | 2.53 | 16.63 | 1.876 | 1.6127 | C |
| 64.95 | 5.55 | 24.03 | 1.79 | 17.98 | 1.342 | - | C |
| 67.11 | 5.44 | 25.97 | 1.84 | 19.97 | 1.414 | 1.7310 | C + D |
| 67.02 | 4.27 | 25.21 | 1.40 | 19.07 | 1.061 | - | D |
| 66.86 | 3.50 | 24.64 | 1.13 | 18.42 | 0.842 | - | D |
| 67.80 | - | 23.65 | - | 17.20 | - | 1.6840 | D |

[a] Compiler's calculation. [b] in $10^3$ kg $m^{-3}$

[c] A = $C_6H_{12}N_4$; B = $NaClO_4.C_6H_{12}N_4$; C = $5NaClO_4.2C_6H_{12}N_4.3H_2O$; D = $NaClO_4.H_2O$.

COMMENTS/ADDITIONAL DATA:

The solubility isotherm of the system has four crystallization branches (see Figure). The first and fourth correspond to the crystallization in the solid phase of the initial subtances. The second (the longest) branch corresponds to the crystallization of congruently soluble $NaClO_4.C_6H_{12}N_4$, the composition of which was confirmed by chemical and thermographic analyses:

Found % : $NaClO_4$ 46.45%; $C_6H_{12}N_4$ 53.32%

Calculated % : $NaClO_4$ 46.62%; $C_6H_{12}N_4$ 53.38%

The compound was isolated as elongated crystals and its density is $1.703 \times 10^3$ kg $m^{-3}$.

When the concn of sodium perchlorate was increased a complex with the formula $5NaClO_4.2C_6H_{12}N_4.3H_2O$ was formed. The composition of this compound was confirmed similarly:

Found % : $NaClO_4$ 64.69%; $C_6H_{12}N_4$ 29.49%; $H_2O$ 5.81%

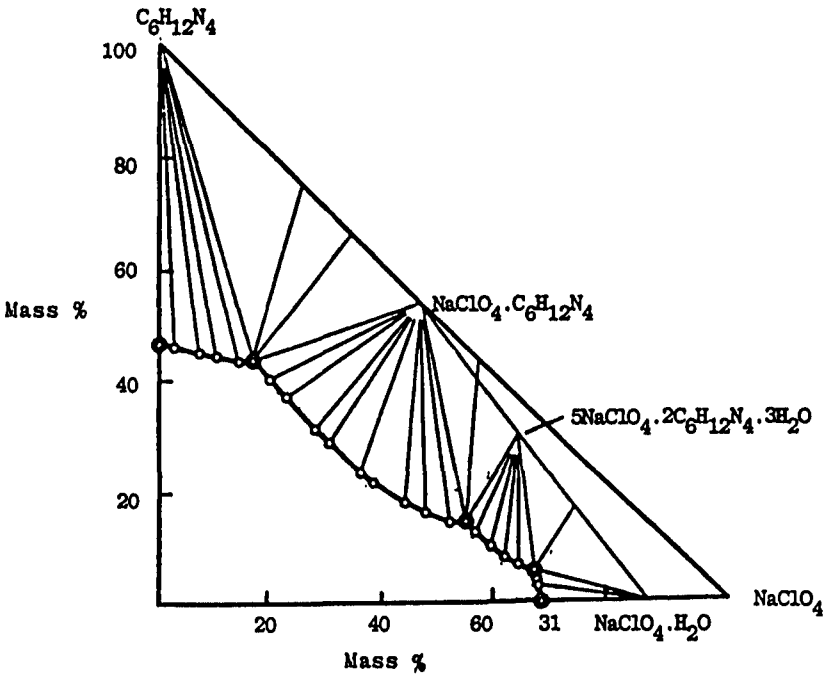
Calculated % : $NaClO_4$ 64.67%; $C_6H_{12}N_4$ 29.62%; $H_2O$ 5.71%

The compound crystallized out as rectangular plates and its density is $1.8639 \times 10^3$ kg $m^{-3}$.

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Sal'nikova, L.N.; Karnaukhov, A.S.; Lepeshkov, I.N. |
| (2) Hexamethylenetetramine; $C_6H_{12}N_4$; [100-97-0] | *Zh. Neorg. Khim.* 1971, *16*, 2840-4; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1971, *16*, 1511-3. |
| (3) Water; $H_2O$; [7732-18-5] | |

COMMENTS/ADDITIONAL DATA: (continued)



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Bestuzheva, M.M.; Kinderov, A.B.; Karnaukhov, A.S. |
| (2) Benzamide; $C_7H_7NO$; [55-21-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1978, *169*, 37-41. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 298 K | E.S. Gryzlova |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$C_6H_5CONH_2$-$H_2O$ at 298 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 1.57 | - | 0.24 | - | 0.132 | $C_6H_5CONH_2$ |
| 4.36 | 1.42 | 0.67 | 0.22 | 0.378 | 0.124 | " |
| 12.66 | 1.40 | 2.12 | 0.24 | 1.203 | 0.134 | " |
| 21.30 | 1.63 | 3.90 | 0.30 | 2.257 | 0.175 | " |
| 30.42 | 1.60 | 6.16 | 0.33 | 3.655 | 0.194 | " |
| 45.07 | 1.57 | 11.01 | 0.39 | 6.898 | 0.243 | " |
| 57.70 | 1.88 | 17.26 | 0.57 | 11.659 | 0.384 | " |
| 64.61 | 1.87 | 21.95 | 0.64 | 15.742 | 0.461 | $NaClO_4.H_2O$ + $C_6H_5CONH_2$ |
| 65.12 | 1.84 | 22.34 | 0.64 | 16.097 | 0.460 | " |
| 66.28 | 1.32 | 23.03 | 0.46 | 16.708 | 0.336 | $NaClO_4.H_2O$ |
| 67.84 | - | 23.69 | - | 17.228 | - | " |

[a] Compiler's calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Periods of equilibration varied from 2 to 3 days in the system and from 4 to 6 days at the eutonic points. Benzamide was determined by the Kjeldahl method (ref. 1); $ClO_4^-$ ion by nitron precipitation (ref. 2). The density and viscosity of saturated solutions were measured. The maximum density and viscosity values occurred at the eutonic composition. | Not stated. |
| | ESTIMATED ERROR: |
| | Not stated. |
| | REFERENCES: |
| | 1. Hillebrand, W. F.; Lundell G.E.F. *Applied Inorganic Analysis*, 2nd edit. Wiley, N.Y.-London 1963 |
| | 2. Loebich, O.L. *Anal. Chem.*, 1926, *68*, 34. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Chernykh, L.V.; Ivanov, V.V.; Alekseeva, E.A. |
| (2) Perchloric acid; $HClO_4$; [7601-90-3] | *Zh. Neorg. Khim.* 1970, *15*, 1922-27; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1970, *15*, 987-9. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298 K | C.C. Ho |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$HClO_4$-$H_2O$ at 25°C:

| Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 67.70 | - | 23.57 | - | 17.12 | - | A |
| 58.26 | 7.62 | 19.46 | 3.102 | 13.95 | 2.223 | A |
| 49.45 | 14.63 | 15.88 | 5.726 | 11.24 | 4.054 | A |
| 32.30 | 28.81 | 9.737 | 10.59 | 6.783 | 7.374 | A |
| 28.47 | 32.80 | 8.584 | 12.05 | 6.004 | 8.430 | A |
| 25.26 | 35.33 | 7.514 | 12.81 | 5.235 | 8.924 | A |
| 23.86 | 36.47 | 7.061 | 13.15 | 4.912 | 9.151 | A + B |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

(1) was analysed on columns of KU-2 ion exchange resin and gravimetrically. The solubility was found by isothermal saturation at 25°C. Equilibrium was reached in 6-8h. In analyses of specimens of liquid and solid phases, the $H^+$ ion concn was determined by titration with borax solution and the sum of $Na^+$ and $H^+$ cations was determined on columns of Ku-2 ion-exchange resin, then the concn of $Na^+$ ion in the specimen was found by difference. Solid phase composition was found by Schreinemakers' method.

SOURCE AND PURITY OF MATERIALS:

Chemically pure grade 57% $HClO_4$, anhydrous $HClO_4$, and repeatedly recrystallized $NaClO_4$ were used. The anhydrous acid was made by the recommended method (ref. 1). The $HClO_4$ solutions were tested for $Cl^-$ ion, the anhydrous acid for sulfur oxides and chloride oxides.

ESTIMATED ERROR:

Temperature: ± 0.05°C

Solubility : nothing specified.

REFERENCES:

1. Brauer, G. *Handbuch der Praparativen anorganischen chemie* (Transl. into Russ.), Inostr. Lit. Moscow, 1956.

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Chernykh, L.V.; Ivanov, V.V.; Alekseeva, E.A. |
| (2) Perchloric acid; $HClO_4$; [7601-90-3] | *Zh. Neorg. Khim.* 1970, *15*, 1922-27; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1970, *15*, 987-9. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

| Liquid phase composition | | | | | | Solid[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | phase |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 23.12 | 37.15 | 6.832 | 13.38 | 4.753 | 9.308 | B |
| 19.40 | 40.88 | 5.720 | 14.69 | 3.989 | 10.25 | B |
| 16.60 | 43.42 | 4.865 | 15.51 | 3.391 | 10.81 | B |
| 13.56 | 46.29 | 3.955 | 16.46 | 2.758 | 11.48 | B |
| 10.45 | 50.06 | 3.075 | 17.95 | 2.161 | 12.62 | B |
| 4.60 | 58.13 | 1.399 | 21.55 | 1.008 | 15.53 | B |
| 3.00 | 63.26 | 0.970 | 24.92 | 0.7262 | 18.66 | B |
| 2.33 | 66.45 | 0.788 | 27.41 | 0.6095 | 21.19 | B |
| 2.03 | 69.01 | 0.717 | 29.72 | 0.5725 | 23.72 | B |
| 1.78 | 69.30 | 0.622 | 29.85 | 0.4967 | 23.84 | B |
| 1.53 | 74.46 | 0.599 | 35.52 | 0.5204 | 30.87 | B |
| 1.57 | 76.34 | 0.641 | 38.02 | 0.5805 | 34.40 | B |
| 1.09 | 78.63 | 0.464 | 40.82 | 0.4390 | 38.60 | C |
| 1.40 | 78.51 | 0.599 | 40.96 | 0.5691 | 38.90 | C |
| 1.02 | 84.10 | 0.498 | 50.09 | 0.5599 | 56.26 | C |
| 1.00 | 91.60 | 0.614 | 68.52 | 1.104 | 123.22 | C |
| 1.26 | 93.80 | 0.845 | 76.65 | 2.083 | 189.01 | C |

[a] Compiler's claculations. [b] A = $NaClO_4.H_2O$; B = $NaClO_4$; C = $NaClO_4.HClO_4$.

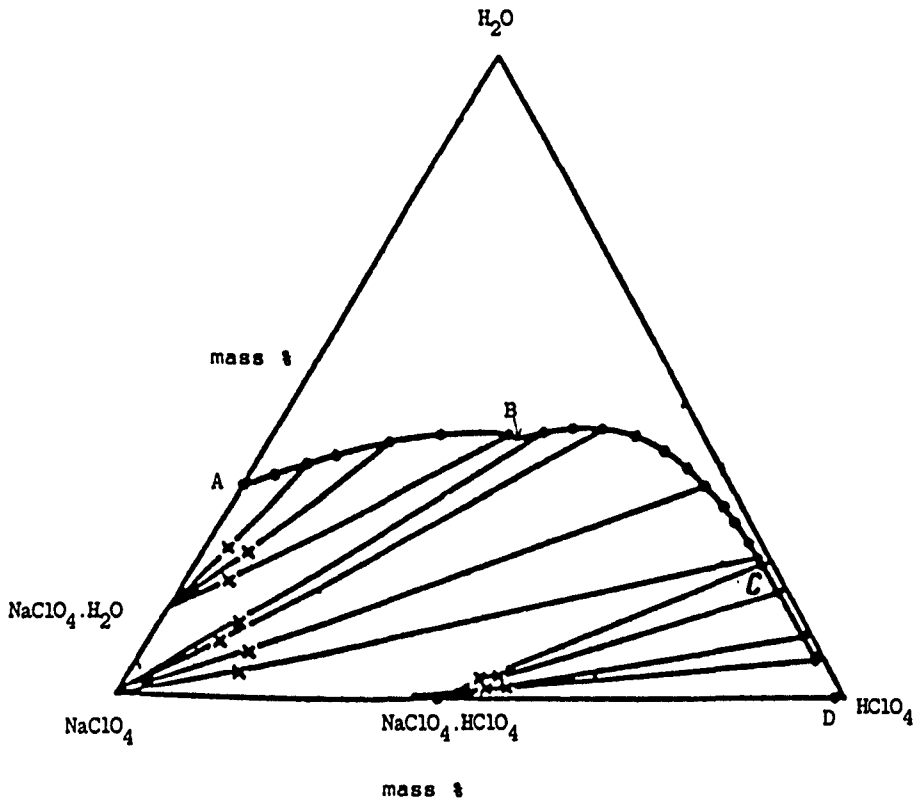
COMMENTS/ADDITIONAL DATA:

The solubility isotherm of the ternary system (see Figure) does not have a branch with a salt solubility minimum but there is a monotonic decrease in the solubility of $NaClO_4$. Evidently the $Na^+$ ion does not compete with the $H_3O^+$ as a salting-out agent and this part is played by the acid throughout the whole region. The isotherm has two eutonic points (B and C). Point B corresponds to the change of the solid phase from the crystal hydrate $NaClO_4.H_2O$ to the anhydrous salt and point C to the change from $NaClO_4$ to the compound $NaClO_4.HClO_4$. It was found that the solubility of $NaClO_4$ in the anhydrous acid is 0.624% (point D).

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Perchloric acid; $HClO_4$; [7601-90-3]<br>(3) Water; $H_2O$; [7732-18-5] | Chernykh, L.V.; Ivanov, V.V.; Alekseeva, E.A.<br><br>*Zh. Neorg. Khim.* 1970, *15*, 1922-27; **Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1970, *15*, 987-9. |

COMMENTS/ADDITIONAL DATA: (continued)



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Karnaukhov, A.S.; Makin, A.V. |
| (2) Potassium perchlorate; $KClO_4$; [7778-74-7] | *Zh. Neorg. Khim.* 1957, *2*, 910-4. |
| (3) Water; $H_2O$; [7732-18-5] | *Russ. J. Inorg. Chem.*, (*Engl. Transl.*) 1957, *2*, 311-6. |
| VARIABLES: Temperature: 273K and 298K Composition. | PREPARED BY: C.C. Ho |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$KClO_4$-$H_2O$ at 0°C:

| | Liquid Phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | Molality[a]/mol $kg^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | - | 0.75 | - | 0.098 | - | 0.0545 | $KClO_4$ |
| 2 | 5.15 | 0.63 | 0.797 | 0.0862 | 0.446 | 0.0483 | " |
| 3 | 6.94 | 0.55 | 1.091 | 0.0764 | 0.613 | 0.0429 | " |
| 4 | 9.23 | 0.45 | 1.480 | 0.0638 | 0.835 | 0.0360 | " |
| 5 | 11.71 | 0.49 | 1.923 | 0.0711 | 1.089 | 0.0403 | " |
| 6 | 11.85 | 0.48 | 1.949 | 0.0698 | 1.104 | 0.0395 | " |
| 7 | 19.54 | 0.51 | 3.468 | 0.0800 | 1.996 | 0.0460 | " |
| 8 | 21.56 | 0.50 | 3.908 | 0.0801 | 2.259 | 0.0463 | " |
| 9 | 31.82 | 0.49 | 6.463 | 0.0880 | 3.839 | 0.0522 | " |
| 10 | 36.58 | 0.45 | 7.868 | 0.0855 | 4.744 | 0.0516 | " |
| 11 | 41.24 | 0.46 | 9.418 | 0.0928 | 5.777 | 0.0569 | " |
| 12 | 50.00 | 0.51 | 12.93 | 0.117 | 8.251 | 0.0744 | " |
| 13 | 55.37 | 0.52 | 15.57 | 0.129 | 10.25 | 0.0851 | $KClO_4$ + $NaClO_4$[b] |
| 14 | 55.32 | 0.51 | 15.54 | 0.127 | 10.23 | 0.0833 | " |
| 15 | 55.35 | 0.51 | 15.56 | 0.127 | 10.24 | 0.0834 | " |
| 16 | 55.32 | 0.54 | 15.55 | 0.134 | 10.24 | 0.0883 | " |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
The isothermal method was used. Equilibrium was attained after 5-6 days at 25°C, but after 10-12 days at 0°C. $ClO_4^-$ was determined gravimetrically by precipitation from a satd sln of rubidium chloride, filtered, washed with 96% alcohol and dried to constant weight at 105°C. Potassium was determined by the gravimetric method of cobalt nitrate in the form of $K_2NaCo(NO_3)_6.H_2O$. $Na^+$ was determined by the zinc-uranyl-acetate gravimetric method.

SOURCE AND PURITY OF MATERIALS:
Chemically pure sodium perchlorate was purified by two recrystallizations. $KClO_4$ was obtained from 30% $HClO_4$ and chemically pure KCl. The salt obtained was twice recrystallized. Purities of salts obtained varied within the limits from 99.54-99.76%.

ESTIMATED ERROR:
Nothing specified.

REFERENCES: (continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Karnaukhov, A.S.; Makin, A.V. |
| (2) Potassium perchlorate; $KClO_4$; [7778-74-7] | *Zh. Neorg. Khim.* 1957, *2*, 910-4. |
| (3) Water; $H_2O$; [7732-18-5] | *Russ. J. Inorg. Chem.*, (*Engl. Transl.*) 1957, *2*, 311-6. |

EXPERIMENTAL VALUES: (continued)

Solubility system $NaClO_4$-$KClO_4$-$H_2O$ at 0°C:

| | Liquid Phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | Molality[a]/mol kg$^{-1}$ | | phase |
| Ponit | (1) | (2) | (1) | (2) | (1) | (2) | |
| 17 | 58.23 | 0.42 | 17.15 | 0.109 | 11.50 | 0.0733 | $nKClO_4$.$mNaClO_4$ |
| 18 | 60.15 | 0.28 | 18.26 | 0.0751 | 12.42 | 0.0511 | " |
| 19 | 61.46 | 0.16 | 19.06 | 0.0438 | 13.08 | 0.0301 | " |
| 20 | 62.89 | - | 19.96 | - | 13.84 | - | $NaClO_4$.$H_2O$ |

[a] Compiler's calculations.

[b] (evaluator's remark) the solid phase is not $NaClO_4$

Solubility system $NaClO_4$-$KClO_4$-$H_2O$ at 25°C:

| | Liquid Phase composition | | | | | | Solid |
|---|---|---|---|---|---|---|---|
| | mass % | | mol %[a] | | Molality[a]/mol kg$^{-1}$ | | phase |
| Point | (1) | (2) | (1) | (2) | (1) | (2) | |
| 1 | - | 2.30 | - | 0.305 | - | 0.170 | $KClO_4$ |
| 2 | 2.38 | 2.18 | 0.364 | 0.295 | 0.204 | 0.165 | " |
| 3 | 4.11 | 2.21 | 0.639 | 0.304 | 0.358 | 0.170 | " |
| 4 | 6.52 | 2.03 | 1.035 | 0.285 | 0.582 | 0.160 | " |
| 5 | 10.00 | 1.36 | 1.630 | 0.196 | 0.921 | 0.111 | " |
| 6 | 11.95 | 1.34 | 1.984 | 0.197 | 1.126 | 0.112 | " |
| 7 | 16.74 | 1.60 | 2.921 | 0.247 | 1.674 | 0.141 | " |
| 8 | 18.39 | 1.60 | 3.263 | 0.251 | 1.877 | 0.144 | " |
| 9 | 26.06 | 1.62 | 5.021 | 0.276 | 2.943 | 0.162 | " |
| 10 | 32.38 | 1.45 | 6.699 | 0.265 | 3.997 | 0.158 | " |
| 11 | 40.82 | 1.30 | 9.376 | 0.264 | 5.760 | 0.162 | " |
| 12 | 49.94 | 1.26 | 13.05 | 0.291 | 8.358 | 0.186 | " |
| 13 | 58.37 | 1.33 | 17.51 | 0.352 | 11.83 | 0.238 | " |
| 14 | 63.26 | 1.34 | 20.74 | 0.388 | 14.59 | 0.273 | " |
| 15 | 66.32 | 0.47 | 22.68 | 0.142 | 16.31 | 0.102 | $NaClO_4$.$H_2O$ |
| 16 | 67.82 | - | 23.67 | - | 17.21 | - | " |

[a] Compiler's calculations.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | a. Lepeshkov, I.N.; Druzhinina, G.V.; Troitskii, E.N. *Yarosl. kn. Izd.* 1966, 37-45 |
| (2) Potassium perchlorate; $KClO_4$; [7778-74-7] | b. Druzhinina, G.V. *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, 59, 73-82 |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** Temperatures: 298.2 and 323.2 K Composition | **PREPARED BY:** E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$KClO_4$-$H_2O$ at 298.2 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 2.00 | - | 0.26 | - | 0.147 | $KClO_4$ |
| 3.00 | 1.28 | 0.46 | 0.17 | 0.256 | 0.097 | " |
| 6.50 | 0.64 | 1.02 | 0.09 | 0.572 | 0.050 | " |
| 11.87 | 0.46 | 1.95 | 0.07 | 1.106 | 0.038 | " |
| 17.38 | 0.39 | 3.01 | 0.06 | 1.726 | 0.034 | " |
| 22.36 | 0.37 | 4.08 | 0.06 | 2.363 | 0.035 | " |
| 26.20 | 0.36 | 4.98 | 0.06 | 2.914 | 0.035 | " |
| 32.70 | 0.35 | 6.70 | 0.06 | 3.989 | 0.038 | " |
| 38.45 | 0.32 | 8.45 | 0.06 | 5.129 | 0.038 | " |
| 46.00 | 0.33 | 11.19 | 0.07 | 7.000 | 0.044 | " |
| 52.39 | 0.29 | 14.00 | 0.07 | 9.042 | 0.044 | " |
| 58.51 | 0.26 | 17.26 | 0.07 | 11.590 | 0.046 | " |
| 63.50 | 0.22 | 20.47 | 0.06 | 14.295 | 0.044 | " |
| 67.63 | 0.21 | 23.61 | 0.06 | 17.175 | 0.047 | $KClO_4$ + $NaClO_4.H_2O$ |
| 67.62 | 0.20 | 23.60 | 0.06 | 17.162 | 0.045 | " |
| 67.64 | 0.22 | 23.63 | 0.07 | 17.188 | 0.049 | " |
| 67.63 | 0.21 | 23.61 | 0.06 | 17.175 | 0.047 | " |
| 67.61 | 0.20 | 23.59 | 0.06 | 17.154 | 0.045 | " |
| 67.80 | - | 23.65 | - | 17.197 | - | $NaClO_4.H_2O$ |

[a] Compiler's calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Equilibrium of saturated solns was reached in 4 or 5 days for 298K and in 2 or 3 days for 323K. Sodium ion was determined gravimetrically with zinc uranyl acetate; potassium by precipitating with sodium tetraphenylacetate. | The salts were purified by double recrystallization. |
| | ESTIMATED ERROR: Temperature: ± 0.1 K |
| | REFERENCES: None. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS; |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | a.Lepeshkov, I.N.; Druzhinina, G.V.; Troitskii, E.N. *Yarosl. kn. Izd.* 1966, 37-45 |
| (2) Potassium perchlorate; $KClO_4$; [7778-74-7] | b.Druzhinina, G.V. *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, *59*, 73-82 |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system $KClO_4$-$NaClO_4$-$H_2O$ at 323.2 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol % | | molality/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 4.93 | - | 0.67 | - | 0.374 | $KClO_4$ |
| 3.11 | 3.60 | 0.49 | 0.50 | 0.272 | 0.279 | " |
| 7.47 | 2.38 | 1.20 | 0.34 | 0.677 | 0.191 | " |
| 12.31 | 2.02 | 2.06 | 0.30 | 1.174 | 0.170 | " |
| 17.23 | 1.79 | 3.03 | 0.28 | 1.738 | 0.160 | " |
| 26.20 | 1.18 | 5.03 | 0.20 | 2.947 | 0.117 | " |
| 30.73 | 0.99 | 6.20 | 0.18 | 3.676 | 0.105 | " |
| 36.50 | 0.87 | 7.88 | 0.17 | 4.760 | 0.100 | " |
| 40.53 | 0.65 | 9.19 | 0.13 | 5.628 | 0.080 | " |
| 45.28 | 0.62 | 10.95 | 0.13 | 6.836 | 0.083 | " |
| 51.43 | 0.64 | 13.61 | 0.15 | 8.764 | 0.096 | " |
| 56.91 | 0.61 | 16.44 | 0.16 | 10.942 | 0.104 | " |
| 60.61 | 0.62 | 18.67 | 0.17 | 12.768 | 0.115 | " |
| 67.00 | 0.60 | 23.29 | 0.18 | 16.889 | 0.134 | " |
| 71.05 | 0.62 | 26.90 | 0.21 | 20.483 | 0.158 | " |
| 72.52 | 0.61 | 28.36 | 0.21 | 22.043 | 0.164 | $KClO_4$ + $NaClO_4.H_2O$ |
| 72.65 | 0.59 | 28.49 | 0.20 | 22.173 | 0.159 | " |
| 72.70 | 0.60 | 28.54 | 0.21 | 22.238 | 0.162 | " |
| 72.54 | 0.61 | 28.38 | 0.21 | 22.065 | 0.164 | " |
| 72.81 | 0.60 | 28.66 | 0.21 | 22.364 | 0.163 | " |
| 73.20 | - | 28.67 | - | 22.308 | - | $NaClO_4.H_2O$ |

[a] Compiler's calculation.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Shklovskaya, R.M.; Arkhipov, S.M.; Kuzina, V.A.; Kirgintsev, A.N. |
| (2) Cesium perchlorate; $CsClO_4$; [13454-84-7] | *Zh. Neorg. Khim.* 1974, *19*, 846-9; **Russ. J. Inorg. Chem.* (Engl. Transl.) 1974, *19*, 462-3. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system $NaClO_4$-$CsClO_4$-$H_2O$ at 348.2 K:

| Liquid phase composition | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 11.667 | - | 1.0137 | - | 0.5684 | A |
| 10.950 | 5.046 | 1.8733 | 0.4549 | 1.0646 | 0.2585 | " |
| 25.725 | 3.816 | 5.0779 | 0,3969 | 2.9819 | 0.2331 | " |
| 38.600 | 3.517 | 8.8970 | 0.4272 | 5.4464 | 0.2615 | " |
| 51.497 | 3.417 | 14.3158 | 0.5006 | 9.3286 | 0.3262 | " |
| 55.502 | 3.413 | 16.4923 | 0.5344 | 11.033 | 0.3575 | " |
| 55.976 | 3.447 | 16.781 | 0.5445 | 11.267 | 0.3656 | A + C |
| 56.450 | 3.381 | 17.042 | 0.5379 | 11.478 | 0.3622 | C |
| 57.883 | 2.921 | 17.766 | 0.4724 | 12.061 | 0.3207 | " |
| 59.875 | 2.580 | 18.923 | 0.4297 | 13.025 | 0.2957 | " |
| 67.044 | 1.278 | 23.689 | 0.2380 | 17.285 | 0.1736 | " |
| 74.080 | 0.808 | 30.215 | 0.1737 | 24.093 | 0.1385 | " |
| 74.175 | 0.544 | 30.118 | 0.1164 | 23.963 | 0.0926 | C + E |
| 74.876 | - | 30.483 | - | 24.341 | - | E |

[a] Compiler's calculation.

[b] A = $CsClO_4$ ; B = $3CsClO_4.NaClO_4$ ; C = $CsClO_4.NaClO_4$
D = $NaClO_4.H_2O$ ; E = $NaClO_4$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: (continued)

ed gravimetrically with nitron (ref. 1); $Cs^+$ by precipitation as the tetraphenylborate (ref. 2). The solid phases were identified by the method of residues and by x-ray diffraction.

ESTIMATED ERROR:

Temperature: ± 0.1 °C.

REFERENCES:

1. Loebich, O. *Z. Analyt. Chem.* 1926, *68*, 34.
2. Yanson, E.Yu.; Levin'sh, A.F. *Uspekhi. Khim.* 1953, *28*, 980.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Shklovskaya, R.M.; Arkhipov, S.M.; Kuzina, V.A.; Kirgintsev, A.N. |
| (2) Cesium perchlorate; $CsClO_4$; [13454-84-7] | *Zh. Neorg. Khim.* 1974, *19*, 846-9; **Russ. J. Inorg. Chem.* (Engl. Transl.) 1974, *19*, 462-3. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| Temperature: 298.2 K and 348.2 K. | W.L. Ng |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$CsClO_4$-$H_2O$ at 298.2 K:

| Liquid phase composition | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 1.96 | - | 0.1548 | - | 0.086 | A |
| 6.262 | 0.598 | 0.9791 | 0.0493 | 0.549 | 0.0276 | " |
| 13.230 | 0.440 | 2.2043 | 0.0386 | 1.252 | 0.0219 | " |
| 20.042 | 0.379 | 3.5719 | 0.0356 | 2.057 | 0.0205 | " |
| 32.377 | 0.468 | 6.6205 | 0.0504 | 3.938 | 0.0300 | " |
| 37.010 | 0.502 | 8.0113 | 0.0573 | 4.837 | 0.0346 | " |
| 39.359 | 0.528 | 8.7817 | 0.0621 | 5.348 | 0.0378 | " |
| 43.492 | 0.562 | 10.257 | 0.0698 | 6.349 | 0.0432 | " |
| 43.540 | 0.563 | 10.275 | 0.0700 | 6.362 | 0.0433 | A + B |
| 43.588 | 0.565 | 10.294 | 0.0703 | 6.374 | 0.0435 | B |
| 44.504 | 0.565 | 10.643 | 0.0712 | 6.617 | 0.0443 | " |
| 44.957 | 0.565 | 10.820 | 0.0717 | 6.740 | 0.0446 | " |
| 46.587 | 0.556 | 11.471 | 0.0721 | 7.198 | 0.0453 | " |
| 47.114 | 0.556 | 11.689 | 0.0727 | 7.353 | 0.0457 | " |
| 47.138 | 0.552 | 11.698 | 0.0722 | 7.356 | 0.0454 | B + C |
| 47.162 | 0.548 | 11.707 | 0.0717 | 7.366 | 0.0451 | C |
| 48.919 | 0.488 | 12.447 | 0.0654 | 7.897 | 0.0415 | " |
| 54.996 | 0.385 | 15.343 | 0.0566 | 10.067 | 0.0371 | " |
| 57.436 | 0.383 | 16.681 | 0.0586 | 11.121 | 0.0391 | " |
| 58.746 | 0.292 | 17.417 | 0.0456 | 11.713 | 0.0307 | " |
| 60.413 | 0.237 | 18.420 | 0.0381 | 12.539 | 0.0259 | " |
| 62.117 | 0.201 | 19.514 | 0.0333 | 13.463 | 0.0230 | " |
| 67.246 | 0.168 | 23.284 | 0.0307 | 16.854 | 0.0222 | " |
| 68.388 | 0.128 | 24.214 | 0.0239 | 17.741 | 0.0175 | C + D |
| 68.464 | 0.129 | 24.279 | 0.0241 | 17.804 | 0.0177 | C + D |
| 68.516 | - | 24.254 | - | 17.774 | - | D |

[a] Compiler's calculation.

[b] A = $CsClO_4$ ; B = $3CsClO_4.NaClO_4$ ; C = $CsClO_4.NaClO_4$
D = $NaClO_4.H_2O$ ; E = $NaClO_4$

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| In the isothermal method used, the mixtures were equilibrated in a thermostated bath for 30 days. In the analysis of the solid and liquid phases, $ClO_4^-$ was determin- | "Chemically pure" grade salts were recrystallized twice from doubly-distilled water. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Kudryakova, S.A.; Karnaukhov, A.S. |
| (2) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 36-42. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 298.2, 308.2, 363.2 K. | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$NH_4ClO_4$-$H_2O$ at 298.2 K:

| Liquid phase composition | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 18.60 | - | 3.39 | - | 1.945 | A |
| 4.77 | 16.28 | 0.85 | 3.04 | 0.493 | 1.755 | " |
| 8.20 | 15.58 | 1.51 | 2.99 | 0.879 | 1.740 | " |
| 13.37 | 13.58 | 2.55 | 2.70 | 1.495 | 1.582 | " |
| 19.07 | 10.82 | 3.76 | 2.22 | 2.221 | 1.314 | " |
| 23.90 | 9.47 | 4.91 | 2.03 | 2.930 | 1.210 | " |
| 28.18 | 7.76 | 5.97 | 1.71 | 3.593 | 1.031 | " |
| 35.71 | 6.36 | 8.19 | 1.52 | 5.035 | 0.934 | " |
| 39.47 | 5.62 | 9.43 | 1.40 | 5.871 | 0.871 | " |
| 45.06 | 4.87 | 11.54 | 1.30 | 7.350 | 0.828 | " |
| 49.59 | 4.40 | 13.52 | 1.25 | 8.803 | 0.814 | " |
| 52.62 | 3.82 | 14.92 | 1.13 | 9.866 | 0.746 | " |
| 61.89 | 2.75 | 20.29 | 0.94 | 14.295 | 0.662 | " |
| 61.92 | 2.70 | 20.29 | 0.92 | 14.294 | 0.650 | A + B |
| 63.33 | 2.59 | 21.28 | 0.91 | 15.177 | 0.647 | B |
| 64.73 | 2.11 | 22.15 | 0.75 | 15.943 | 0.542 | " |
| 66.52 | 1.63 | 23.37 | 0.60 | 17.058 | 0.436 | B + C |
| 66.82 | 1.49 | 23.55 | 0.55 | 17.221 | 0.400 | " |
| 66.58 | 1.70 | 23.45 | 0.62 | 17.143 | 0.456 | C |
| 67.27 | 0.68 | 23.54 | 0.25 | 17.142 | 0.181 | " |
| 67.64 | - | 23.52 | - | 17.071 | - | " |

[a] Compiler's calculation.

[b] A = $NH_4ClO_4$ ; B = n($NH_4ClO_4$).m($NaClO_4.H_2O$) ; C = $NaClO_4.H_2O$ ; D = $NaClO_4$ ; E = n($NH_4ClO_4$).m($NaClO_4$)

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Equilibrium in the system in the region of solid solutions was reached in 30 days. Sodium ion was determined gravimetrically as sodium zinc uranyl acetate; ammonium ion by the volumet- | The salts were purified by two recrystallizations and then ground to fine powder. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Kudryakova, S.A.; Karnaukhov, A.S. |
| (2) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 36-42. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system $NaClO_4$-$NH_4ClO_4$-$H_2O$ at 308.2 K:

| Liquid phase composition | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 22.43 | - | 4.25 | - | 2.461 | A |
| 3.16 | 21.67 | 0.59 | 4.21 | 0.343 | 2.454 | " |
| 6.03 | 20.13 | 1.14 | 3.97 | 0.667 | 2.320 | " |
| 8.34 | 18.50 | 1.59 | 3.67 | 0.931 | 2.152 | " |
| 15.88 | 15.63 | 3.19 | 3.27 | 1.894 | 1.942 | " |
| 20.90 | 13.33 | 4.34 | 2.88 | 2.595 | 1.725 | " |
| 23.64 | 12.08 | 5.00 | 2.66 | 3.004 | 1.600 | " |
| 26.05 | 11.46 | 5.63 | 2.58 | 3.405 | 1.561 | " |
| 28.46 | 10.78 | 6.29 | 2.48 | 3.826 | 1.510 | " |
| 34.11 | 8.41 | 7.87 | 2.02 | 4.847 | 1.245 | " |
| 36.20 | 7.88 | 8.53 | 1.93 | 5.287 | 1.199 | " |
| 43.57 | 5.89 | 11.08 | 1.56 | 7.041 | 0.992 | " |
| 50.05 | 5.36 | 13.95 | 1.56 | 9.167 | 1.023 | " |
| 56.18 | 4.71 | 17.19 | 1.50 | 11.732 | 1.025 | " |
| 60.60 | 4.38 | 19.99 | 1.51 | 14.133 | 1.065 | " |
| 60.75 | 4.50 | 20.14 | 1.55 | 14.278 | 1.102 | A + B |
| 64.45 | 3.12 | 22.37 | 1.13 | 16.231 | 0.819 | B |
| 66.74 | 2.23 | 23.84 | 0.83 | 17.566 | 0.612 | " |
| 67.64 | 2.33 | 24.67 | 0.89 | 18.396 | 0.660 | B + C |
| 67.58 | 2.27 | 24.59 | 0.86 | 18.307 | 0.641 | C |
| 67.68 | 2.35 | 24.72 | 0.89 | 18.444 | 0.667 | " |
| 69.20 | 0.85 | 25.29 | 0.32 | 18.871 | 0.242 | " |
| 69.80 | - | 25.38 | - | 18.877 | - | " |

[a] Compiler's calculation.

[b] A = $NH_4ClO_4$ ; B = n($NH_4ClO_4$).m($NaClO_4.H_2O$) ; C = $NaClO_4.H_2O$ ;
D = $NaClO_4$ ; E = n($NH_4ClO_4$).m($NaClO_4$)

AUXILIARY INFORMATION

METHOD: (continued)

ric Formalin method; perchlorate ion by difference and occasionally by nitron precipitation. Solid solutions were studied by the differential thermal, microphotographic and X-ray powder analytical methods.

ESTIMATED ERROR:

Temperature: ± 0.1 K

REFERENCES:

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Kudryakova, S.A.; Karnaukhov, A.S. |
| (2) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 36-42. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system $NaClO_4$-$NH_4ClO_4$-$H_2O$ at 363.2 K:

| Liquid phase composition | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 43.37 | - | 10.51 | - | 6.518 | A |
| 4.18 | 40.45 | 0.99 | 9.97 | 0.617 | 6.218 | " |
| 9.82 | 35.37 | 2.34 | 8.79 | 1.463 | 5.493 | " |
| 14.14 | 32.86 | 3.46 | 8.38 | 2.179 | 5.277 | " |
| 20.69 | 27.65 | 5.16 | 7.19 | 3.271 | 4.556 | " |
| 25.40 | 24.80 | 6.52 | 6.63 | 4.166 | 4.239 | " |
| 30.75 | 21.80 | 8.18 | 6.04 | 5.293 | 3.910 | " |
| 37.50 | 18.40 | 10.52 | 5.38 | 6.945 | 3.551 | " |
| 40.85 | 16.93 | 11.83 | 5.11 | 7.902 | 3.413 | " |
| 43.60 | 14.93 | 12.79 | 4.56 | 8.587 | 3.064 | " |
| 46.76 | 13.20 | 14.06 | 4.14 | 9.538 | 2.806 | " |
| 51.38 | 11.20 | 16.19 | 3.68 | 11.214 | 2.548 | " |
| 58.76 | 9.20 | 20.54 | 3.35 | 14.978 | 2.444 | " |
| 67.99 | 6.14 | 27.17 | 2.56 | 21.465 | 2.020 | " |
| 68.28 | 6.43 | 27.66 | 2.71 | 22.051 | 2.164 | A + E |
| 70.85 | 5.27 | 29.69 | 2.30 | 24.232 | 1.878 | E |
| 71.94 | 4.56 | 30.43 | 2.01 | 25.002 | 1.652 | " |
| 73.67 | 3.67 | 31.82 | 1.65 | 26.553 | 1.379 | E + D |
| 73.78 | 3.56 | 31.87 | 1.60 | 26.592 | 1.337 | " |
| 73.58 | 3.65 | 31.70 | 1.64 | 26.392 | 1.364 | D |
| 74.40 | 2.40 | 31.72 | 1.07 | 26.191 | 0.880 | " |
| 75.20 | 0.86 | 31.49 | 0.38 | 25.655 | 0.306 | " |
| 75.85 | - | 31.61 | - | 25.652 | - | " |

[a] Compiler's calculation.

[b] A = $NH_4ClO_4$ ; B = $n(NH_4ClO_4).m(NaClO_4.H_2O)$ ; C = $NaClO_4.H_2O$ ;
D = $NaClO_4$ ; E = $n(NH_4ClO_4).m(NaClO_4)$

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Thallium perchlorate; $TlClO_4$; [13453-40-2]<br>(3) Water; $H_2O$; [7732-18-5] | Smirnov, V.N.; Ivanov, S.A.; Chechneva, I.V.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1973, *120*, 13-5. |
| VARIABLES:<br>One temperature: 298 K<br>Composition | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$TlClO_4$-$H_2O$ at 298 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 14.27 | - | 0.98 | - | 0.548 | $TlClO_4$ |
| 14.62 | 6.43 | 2.64 | 0.47 | 1.512 | 0.268 | " |
| 35.51 | 4.65 | 8.00 | 0.42 | 4.847 | 0.256 | " |
| 53.13 | 2.81 | 15.02 | 0.32 | 9.849 | 0.210 | " |
| 66.41 | 2.38 | 23.76 | 0.34 | 17.379 | 0.251 | $NaClO_4.H_2O$ + $TlClO_4$ |
| 66.35 | 2.41 | 23.73 | 0.35 | 17.346 | 0.254 | $NaClO_4.H_2O$ |
| 67.89 | - | 23.73 | - | 17.268 | - | " |

[a] Compiler's calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Equilibrium was attained in 5-7 days. Thallium ion was determined by bromate method; perchlorate ion gravimetrically by precipitation with nitron and sodium ion by difference. | Not stated. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Karnaukhov, A.S.; Kudryakova, S.A. |
| (2) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, *59*, 119-36. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298.2 K | E.S. Gryzlova |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system : $NaClO_4$-$Mg(ClO_4)_2$-$H_2O$ at 25°C

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 49.80 | - | 7.413 | - | 4.444 | $Mg(ClO_4)_2.6H_2O$ |
| 2.95 | 47.88 | 0.812 | 7.228 | 0.490 | 4.363 | " |
| 9.12 | 43.76 | 2.581 | 6.793 | 1.581 | 4.161 | " |
| 15.13 | 40.51 | 4.465 | 6.558 | 2.786 | 4.091 | " |
| 20.26 | 37.75 | 6.208 | 6.345 | 3.941 | 4.028 | " |
| 25.57 | 34.59 | 8.109 | 6.018 | 5.242 | 3.890 | " |
| 26.73 | 33.75 | 8.517 | 5.899 | 5.524 | 3.826 | " |
| 28.05 | 33.13 | 9.047 | 5.861 | 5.901 | 3.823 | " |
| 27.80 | 33.13 | 8.924 | 5.834 | 5.811 | 3.799 | $NaClO_4.H_2O$ + $Mg(ClO_4)_2.6H_2O$ |
| 27.85 | 33.18 | 8.958 | 5.854 | 5.837 | 3.815 | " |
| 28.08 | 33.43 | 9.116 | 5.954 | 5.958 | 3.891 | " |
| 28.00 | 33.56 | 9.101 | 5.984 | 5.949 | 3.911 | " |
| 28.12 | 33.60 | 9.168 | 6.009 | 6.000 | 3.932 | " |
| 28.20 | 33.73 | 9.232 | 6.058 | 6.050 | 3.969 | " |
| 28.30 | 33.73 | 9.283 | 6.069 | 6.087 | 3.980 | $NaClO_4.H_2O$ |
| 31.48 | 30.27 | 10.22 | 5.390 | 6.722 | 3.545 | " |
| 35.49 | 27.04 | 11.64 | 4.863 | 7.736 | 3.233 | " |
| 40.48 | 22.77 | 13.37 | 4.126 | 8.996 | 2.776 | " |
| 45.59 | 18.61 | 15.24 | 3.413 | 10.40 | 2.329 | " |
| 50.27 | 14.72 | 16.97 | 2.725 | 11.73 | 1.884 | " |
| 54.38 | 11.01 | 18.39 | 2.043 | 12.83 | 1.425 | " |

[a] Editors' calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Periods of equilibration varied from 20 to 70 h. $Mg^{2+}$ was determined by complexometric titration, $Na^+$ gravimetrically as sodium zinc uranyl acetate, and $ClO_4^-$ gravimetrically by nitron precipitation. | The salts were recrystallized twice. Purity: 95.58-99.75%. |
| | ESTIMATED ERROR: Temperature: ±0.1°C. |
| | REFERENCES: None. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Karnaukhov, A.S.; Kudryakova, S.A. |
| (2) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, *59*, 119-36. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system : $NaClO_4$-$Mg(ClO_4)_2$-$H_2O$ at 25°C (continued)

[Solid phase : $NaClO_4.H_2O$]

Liquid phase composition

| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | |
|---|---|---|---|---|---|
| (1) | (2) | (1) | (2) | (1) | (2) |
| 57.58 | 8.35 | 19.60 | 1.559 | 13.80 | 1.098 |
| 60.37 | 6.12 | 20.71 | 1.152 | 14.71 | 0.818 |
| 64.51 | 2.38 | 22.18 | 0.449 | 15.91 | 0.322 |
| 67.86 | - | 23.70 | - | 17.24 | - |

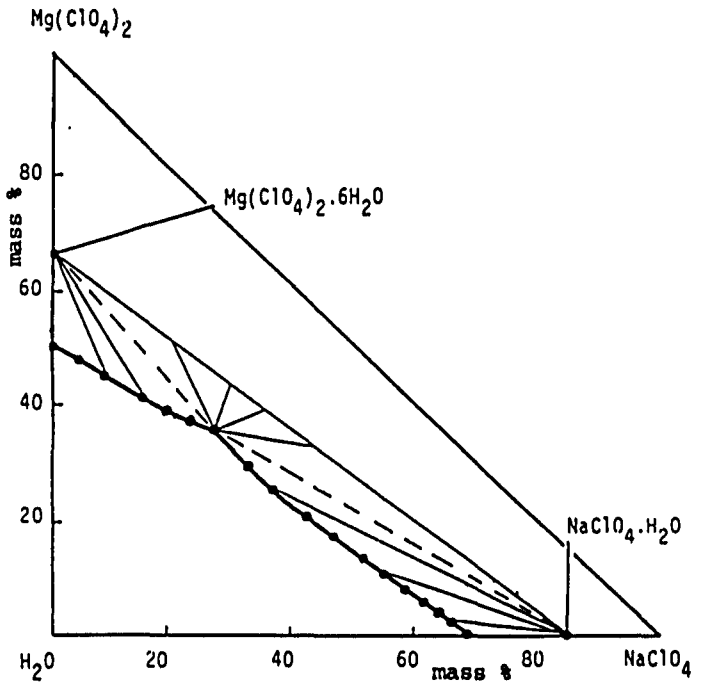
[a] Editors' calculations.

COMMENTS AND/OR ADDITIONAL DATA:

The solubility isotherm (mass%) is shown below.

Solution composition at the isothermal double saturation point ( solid phases $NaClO_4.H_2O$ and $Mg(ClO_4)_2.6H_2O$ ):

28.05 mass % $NaClO_4$, 33.43 mass % $Mg(ClO_4)_2$, and 38.52 mass % $H_2O$.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Kudryakova, S.A.; Lepeshkov, I.N. |
| (2) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8] | *Sb. Tr. Yarosl. Gos. Ped. Inst.*, 1969, *66*, 40-50. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 363 K | I.S. Bodnya |
| Composition | |

EXPERIMENTAL VALUES:

Solubility System : $NaClO_4$-$Mg(ClO_4)_2$-$H_2O$ at 90°C

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 55.57 | - | 9.167 | - | 5.603 | $Mg(ClO_4)_2.6H_2O$ |
| 4.40 | 52.15 | 1.340 | 8.713 | 0.827 | 5.377 | " |
| 8.07 | 49.67 | 2.502 | 8.448 | 1.560 | 5.266 | " |
| 14.54 | 45.18 | 4.644 | 7.916 | 2.948 | 5.025 | " |
| 19.87 | 42.03 | 6.582 | 7.638 | 4.259 | 4.942 | " |
| 24.22 | 39.21 | 8.230 | 7.309 | 5.409 | 4.804 | " |
| 28.63 | 36.39 | 9.999 | 6.972 | 6.685 | 4.661 | " |
| 34.01 | 33.10 | 12.34 | 6.586 | 8.445 | 4.509 | " |
| 34.08 | 32.87 | 12.32 | 6.516 | 8.422 | 4.456 | $NaClO_4$ + $Mg(ClO_4)_2.6H_2O$ |
| 34.27 | 32.75 | 12.40 | 6.500 | 8.487 | 4.449 | " |
| 34.57 | 32.56 | 12.53 | 6.475 | 8.590 | 4.438 | " |
| 35.10 | 31.89 | 12.67 | 6.317 | 8.684 | 4.328 | " |
| 34.57 | 32.18 | 12.43 | 6.345 | 8.491 | 4.336 | " |
| 34.60 | 32.28 | 12.47 | 6.383 | 8.532 | 4.367 | $NaClO_4$ |
| 37.58 | 29.81 | 13.64 | 5.934 | 9.412 | 4.095 | " |
| 41.69 | 26.57 | 15.33 | 5.359 | 10.73 | 3.750 | " |
| 45.56 | 23.78 | 17.06 | 4.886 | 12.14 | 3.475 | " |
| 49.37 | 20.81 | 18.74 | 4.333 | 13.52 | 3.126 | " |
| 55.45 | 15.49 | 21.21 | 3.250 | 15.58 | 2.388 | " |
| 61.75 | 11.03 | 24.43 | 2.393 | 18.53 | 1.815 | " |
| 69.13 | 5.45 | 28.23 | 1.221 | 22.21 | 0.961 | " |
| 74.51 | 1.33 | 31.12 | 0.305 | 25.19 | 0.247 | " |
| 76.27 | - | 32.11 | - | 26.25 | - | " |

[a] Editors's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. $Mg^{2+}$ was determined volumetrically by titration with Trilon B ; $Na^+$ gravimetrically by precipitation with zinc uranyl acetate; and $ClO_4^-$ gravimetrically by nitron precipitation. The densities and relative viscosities of the saturated solutions were measured. | Not given. |
| | **ESTIMATED ERROR:** Not given. |
| | **REFERENCES:** None. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Lilich, L.S.; Ovtrakht, N.V. |
| (2) Calcium perchlorate; $Ca(ClO_4)_2$; [13477-36-6] | *Vestn. LGU, Ser. Fiz. Khim.* 1965, *10*, 115-9. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 298 K | N.A. Kozyreva |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility of the system $NaClO_4$-$Ca(ClO_4)_2$-$H_2O$ at 25°C:

| Liquid phase composition | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 65.40 | - | 12.47 | - | 7.909 | $Ca(ClO_4)_2.4H_2O$ |
| 1.92 | 64.14 | 0.72 | 12.38 | 0.462 | 7.908 | " |
| 3.28 | 62.91 | 1.24 | 12.15 | 0.792 | 7.786 | " |
| 5.61 | 61.76 | 2.17 | 12.22 | 1.404 | 7.920 | " |
| 5.82 | 61.47 | 2.24 | 12.13 | 1.453 | 7.864 | " |
| 5.84 | 61.47 | 2.25 | 12.14 | 1.459 | 7.868 | " |
| 6.01 | 61.26 | 2.31 | 12.08 | 1.500 | 7.832 | " |
| 6.45 | 61.16 | 2.50 | 12.15 | 1.626 | 7.901 | $NaClO_4$ |
| 6.77 | 60.30 | 2.59 | 11.82 | 1.679 | 7.662 | " |
| 9.15 | 57.59 | 3.46 | 11.15 | 2.247 | 7.245 | " |
| 13.50 | 51.48 | 4.858 | 9.491 | 3.148 | 6.151 | " |
| 13.77 | 51.17 | 4.948 | 9.421 | 3.208 | 6.107 | " |
| 15.71 | 48.36 | 5.518 | 8.703 | 3.571 | 5.632 | $NaClO_4.H_2O$ |
| 17.16 | 46.73 | 5.989 | 8.356 | 3.881 | 5.415 | " |
| 17.32 | 46.90 | 6.087 | 8.445 | 3.954 | 5.485 | " |
| 18.16 | 45.72 | 6.326 | 8.160 | 4.106 | 5.297 | " |
| 23.13 | 39.56 | 7.789 | 6.825 | 5.063 | 4.437 | " |
| 35.68 | 27.02 | 11.77 | 4.568 | 7.813 | 3.031 | " |
| 44.94 | 18.59 | 14.86 | 3.150 | 10.06 | 2.133 | " |
| 53.88 | 11.15 | 18.13 | 1.922 | 12.58 | 1.334 | " |
| 67.79 | - | 23.64 | - | 17.19 | - | " |

[a] Compiler's calculations.

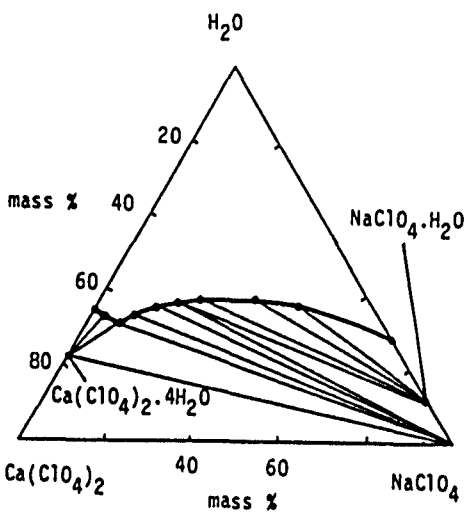
AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal. Equilibrium attained in 3-4h. $ClO_4^-$ was determined by an ion-exchange method using a KU-2 resin; $Ca^{2+}$ by complexometric titration. The composition of the solid phase was determined by Schreinemakers' method of "residues".

SOURCE AND PURITY OF MATERIALS:

Not stated.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Calcium perchlorate; $Ca(ClO_4)_2$; [13477-36-6]<br>(3) Water; $H_2O$; [7732-18-5] | Ivanov, S.A.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* <u>1969</u>, *66*, 82-95. |
| **VARIABLES:**<br>One temperature: 313 K<br>Composition. | **PREPARED BY:**<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility of the system $NaClO_4$-$Ca(ClO_4)_2$-$H_2O$ at 40°C:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 68.40 | - | 14.03 | - | 9.057 | $Ca(ClO_4)_2.4H_2O$ |
| 2.50 | 67.00 | 1.02 | 14.06 | 0.669 | 9.192 | " |
| 6.00 | 64.98 | 2.54 | 14.08 | 1.689 | 9.370 | " |
| 6.40 | 65.08 | 2.74 | 14.28 | 1.833 | 9.548 | $Ca(ClO_4)_2.4H_2O$ + $NaClO_4$ |
| 6.23 | 65.14 | 2.66 | 14.25 | 1.777 | 9.516 | " " |
| 5.90 | 64.90 | 2.48 | 13.99 | 1.650 | 9.300 | " " |
| 6.10 | 65.30 | 2.61 | 14.30 | 1.742 | 9.554 | " " |
| 6.15 | 65.20 | 2.63 | 14.26 | 1.753 | 9.523 | $NaClO_4$ |
| 10.30 | 59.80 | 4.219 | 12.55 | 2.813 | 8.369 | " |
| 13.20 | 55.90 | 5.241 | 11.37 | 3.489 | 7.570 | " |
| 15.20 | 50.04 | 5.486 | 9.253 | 3.571 | 6.024 | " |
| 20.17 | 45.70 | 7.320 | 8.497 | 4.827 | 5.603 | " |
| 23.04 | 42.20 | 8.202 | 7.697 | 5.413 | 5.080 | " |
| 29.82 | 35.00 | 10.40 | 6.251 | 6.923 | 4.163 | " |
| 36.05 | 31.71 | 13.28 | 5.986 | 9.132 | 4.116 | " |

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method. Periods of equilibrium varied from 3-5 days. The solid phases were studied thermographically. The densities, viscosities and refractive indexes of saturated solutions were measured. | Not stated. |
| | ESTIMATED ERROR: Not stated. |
| | REFERENCES: |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Ivanov, S.A. |
| (2) Calcium perchlorate; $Ca(ClO_4)_2$; [13477-36-6] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 82-95. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

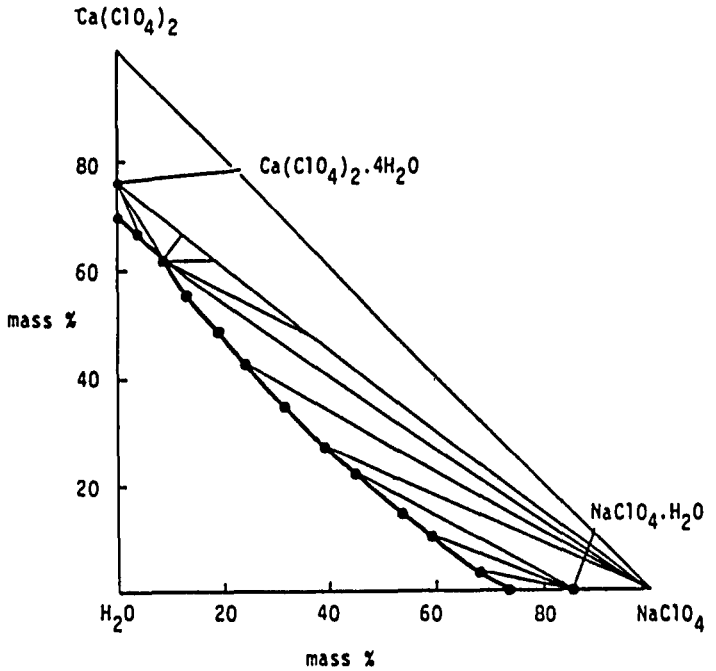
Solubility of the system $NaClO_4$-$Ca(ClO_4)_2$-$H_2O$ at 40°C:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 40.60 | 26.70 | 14.68 | 4.947 | 10.14 | 3.417 | $NaClO_4.H_2O$ |
| 47.10 | 19.80 | 16.69 | 3.595 | 11.62 | 2.503 | " |
| 53.70 | 14.05 | 19.17 | 2.570 | 13.60 | 1.823 | " |
| 59.40 | 8.90 | 21.26 | 1.63 | 15.30 | 1.175 | " |
| 66.07 | 4.14 | 24.41 | 0.78 | 18.11 | 0.582 | " |
| 70.87 | - | 26.36 | - | 19.87 | - | " |

[a] Compiler's calculations.

COMMENTS/ADDITIONAL DATA:

The isotherm shows the branches of crystallization of $Ca(ClO_4)_2.4H_2O$, $NaClO_4$ and $NaClO_4.H_2O$.




Solution composition at the isothermal double saturation point ( solid phases $NaClO_4$ and $Ca(ClO_4)_2.4H_2O$ ):

6.18 mass % $NaClO_4$, 65.10 mass % $Ca(ClO_4)_2$, and 28.77 mass % $H_2O$.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$ ; [7601-89-0] | Druzhinina, G.V. |
| (2) Strontium perchlorate; $Sr(ClO_4)_2$; [13450-97-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1972, *103*, 39-41. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: One temperature, 298 K. Composition. | PREPARED BY: I.S. Bodnya |

EXPERIMENTAL VALUES:

Solubility System $NaClO_4$-$Sr(ClO_4)_2$-$H_2O$ at 25°C:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 68.84 | - | 24.53 | - | 18.04 | - | $NaClO_4.H_2O$ |
| 55.51 | 12.80 | 20.09 | 1.979 | 14.31 | 1.410 | " |
| 40.04 | 28.45 | 15.03 | 4.564 | 10.38 | 3.151 | " |
| 23.80 | 47.06 | 9.836 | 8.311 | 6.671 | 5.636 | " |
| 12.30 | 58.39 | 5.202 | 10.55 | 3.427 | 6.953 | " |
| 5.81 | 69.39 | 2.848 | 14.54 | 1.913 | 9.765 | " + $Sr(ClO_4)_2.4H_2O$ |
| - | 75.01 | - | 15.88 | - | 10.48 | $Sr(ClO_4)_2.4H_2O$ |

[a] Compiler's calculations.

COMMENTS / ADDITIONAL DATA

The solubility isotherm consists of two branches, corresponding to solid phases $Sr(ClO_4)_2.4H_2O$ and $NaClO_4.H_2O$. Average solution composition at the isothermal double saturation point:

5.81 mass % $NaClO_4$, 69.39 mass % $Sr(ClO_4)_2$, and 24.80 mass % $H_2O$

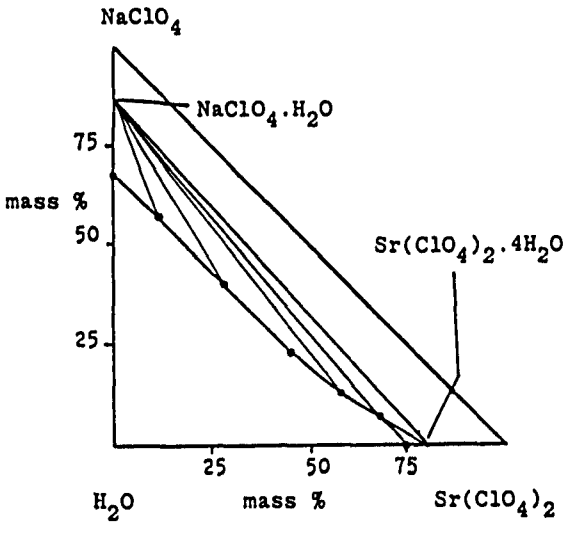
AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
No details.

SOURCE AND PURITY OF MATERIALS:
Not stated.

ESTIMATED ERROR: Not stated.

REFERENCES: None.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Barium perchlorate; $Ba(ClO_4)2$; [13465-95-7]<br>(3) Water; $H_2O$; [7732-18-5] | Zaitseva, S.N.; Lepeshkov, I.N.<br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 113-21. |
| VARIABLES:<br>Temperature/K: 298 and 323<br>Composition | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system : $Ba(ClO_4)_2$-$NaClO_4$-$H_2O$

| Liquid phase composition | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| | | | At 25°C | | | |
| - | 66.89 | - | 9.767 | - | 6.008 | A |
| 0.96 | 66.34 | 0.388 | 9.766 | 0.240 | 6.034 | A |
| 6.74 | 60.85 | 2.705 | 8.893 | 1.698 | 5.584 | A |
| 19.68 | 50.08 | 8.084 | 7.491 | 5.315 | 4.925 | A |
| 29.40 | 42.20 | 12.36 | 6.462 | 8.455 | 4.419 | A |
| 35.95 | 36.59 | 15.24 | 5.648 | 10.69 | 3.963 | A |
| 39.89 | 33.00 | 16.89 | 5.088 | 12.02 | 3.620 | A + B |
| 40.67 | 32.19 | 17.17 | 4.949 | 12.24 | 3.527 | A + B |
| 40.67 | 32.73 | 17.43 | 5.107 | 12.49 | 3.659 | A + B |
| 41.32 | 32.90 | 18.08 | 5.243 | 13.09 | 3.795 | A + B |
| 41.30 | 32.02 | 17.63 | 4.977 | 12.64 | 3.569 | A + B |
| 41.30 | 32.33 | 17.78 | 5.068 | 12.79 | 3.646 | B |
| 42.22 | 29.15 | 17.06 | 4.290 | 12.04 | 3.028 | B |
| 47.04 | 23.69 | 18.48 | 3.388 | 13.13 | 2.407 | B |
| 56.45 | 12.92 | 20.96 | 1.747 | 15.05 | 1.254 | B |
| 61.46 | 4.45 | 20.85 | 0.550 | 14.73 | 0.388 | B |
| 67.48 | - | 23.39 | - | 16.95 | - | B |
| | | | At 50°C | | | |
| - | 72.52 | - | 12.39 | - | 7.849 | A |
| 0.13 | 71.65 | 0.060 | 11.97 | 0.038 | 7.551 | A |
| 4.50 | 68.27 | 2.099 | 11.59 | 1.350 | 7.456 | A |
| 8.20 | 65.63 | 3.905 | 11.38 | 2.559 | 7.458 | A |
| 14.69 | 59.31 | 6.897 | 10.14 | 4.614 | 6.784 | A |
| 21.67 | 53.17 | 10.22 | 9.132 | 7.034 | 6.285 | A |
| 28.32 | 48.04 | 13.72 | 8.472 | 9.784 | 6.044 | A |

[a] Editors' calculations ; [b] A = $Ba(ClO_4)_2.3H_2O$ ; B = $NaClO_4.H_2O$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used [1]. The densities and viscosities of the saturated solutions were measured. | Not stated. |
| ESTIMATED ERROR: | REFERENCES: |
| Not stated. | 1. Karnaukhov, A.S. *Izv. SFKhA AN SSSR*, 1954, *25*, 335. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Zaitseva, S.N.; Lepeshkov, I.N. |
| (2) Barium perchlorate; $Ba(ClO_4)_2$; [13465-95-7] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 113-21. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system : $Ba(ClO_4)_2$-$NaClO_4$-$H_2O$ at 50°C (continued)

| Liquid phase composition | | | | | | Solid phase[a] |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 30.39 | 46.21 | 14.73 | 8.158 | 10.61 | 5.873 | B |
| 34.05 | 43.02 | 16.56 | 7.621 | 12.13 | 5.580 | B |
| 34.67 | 42.89 | 17.10 | 7.701 | 12.62 | 5.684 | B |
| 35.35 | 42.74 | 17.69 | 7.789 | 13.18 | 5.802 | B |
| 35.85 | 41.87 | 17.70 | 7.528 | 13.14 | 5.589 | B + C |
| 35.83 | 40.90 | 17.15 | 7.130 | 12.58 | 5.227 | B + C |
| 35.40 | 41.71 | 17.17 | 7.367 | 12.63 | 5.419 | B + C |
| 35.14 | 41.84 | 16.99 | 7.366 | 12.47 | 5.405 | B + C |
| 36.68 | 41.76 | 18.49 | 7.664 | 13.90 | 5.761 | C |
| 37.43 | 40.19 | 18.33 | 7.168 | 13.66 | 5.341 | C |
| 43.50 | 33.34 | 20.42 | 5.699 | 15.34 | 4.281 | C |
| 47.97 | 28.00 | 21.66 | 4.603 | 16.30 | 3.465 | C |
| 51.46 | 22.79 | 21.92 | 3.535 | 16.32 | 2.632 | C |
| 61.01 | 13.31 | 25.38 | 2.016 | 19.40 | 1.541 | C |
| 64.15 | 10.01 | 26.35 | 1.497 | 20.28 | 1.152 | D |
| 68.79 | 4.14 | 27.05 | 0.593 | 20.76 | 0.455 | D |
| 71.50 | 2.23 | 28.50 | 0.324 | 22.23 | 0.252 | D |
| 73.15 | - | 28.62 | - | 22.25 | - | D |

[a] Editors' calculations.

[b] B = $Ba(ClO_4)_2.2H_2O$ ; C = $NaClO_4$ ; D = $NaClO_4.H_2O$.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Zinc perchlorate; $Zn(ClO_4)_2$; [13637-61-1]<br>(3) Water; $H_2O$; [7732-18-5] | Leboshchina, V.I.; Kudryakova, S.A.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1976, *154*, 68-71. |
| VARIABLES:<br>One temperature: 298 K<br>Composition | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Zn(ClO_4)_2$-$H_2O$ at 298 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 53.17 | - | 7.18 | - | 4.296 | $Zn(ClO_4)_2.6H_2O$ |
| 5.02 | 47.60 | 1.44 | 6.32 | 0.865 | 3.801 | " |
| 11.68 | 43.00 | 3.44 | 5.87 | 2.105 | 3.590 | " |
| 20.29 | 38.71 | 6.40 | 5.66 | 4.042 | 3.573 | " |
| 23.64 | 35.20 | 7.39 | 5.10 | 4.691 | 3.236 | $Zn(ClO_4)_2.6H_2O$ + $NaClO_4.H_2O$ |
| 29.38 | 30.71 | 9.33 | 4.52 | 6.012 | 2.912 | $NaClO_4.H_2O$ |
| 36.14 | 26.38 | 11.92 | 4.03 | 7.875 | 2.663 | " |
| 39.58 | 23.00 | 13.00 | 3.50 | 8.639 | 2.326 | " |
| 51.70 | 12.48 | 17.18 | 1.92 | 11.788 | 1.318 | " |
| 63.35 | 3.87 | 22.00 | 0.62 | 15.784 | 0.447 | " |
| 67.65 | - | 23.53 | - | 17.079 | - | " |

[a] Compiler's calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Equilibrium was reached in 3 or 4 days. $Zn^{2+}$ was determined by complexometric titration with the indicator eriochrome black T at pH 9.7; $ClO_4^-$ gravimetrically as nitron perchlorate. | Not stated. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Karnaukhov, A.S.; Tarakanov, V.F. |
| (2) Nickel perchlorate; $Ni(ClO_4)_2$; [13637-71-3] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 28-31. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298.2 K | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Ni(ClO_4)_2$-$H_2O$ at 298.2 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol % | | molality/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| - | 52.42 | - | 7.15 | - | 4.277 | $Ni(ClO_4)_2.6H_2O$ |
| 5.08 | 47.63 | 1.46 | 6.48 | 0.877 | 3.910 | " |
| 6.50 | 46.58 | 1.87 | 6.37 | 1.131 | 3.854 | " |
| 9.33 | 44.58 | 2.71 | 6.16 | 1.653 | 3.755 | " |
| 12.32 | 41.81 | 3.58 | 5.78 | 2.194 | 3.538 | " |
| 17.04 | 38.81 | 5.08 | 5.50 | 3.152 | 3.412 | " |
| 20.59 | 36.53 | 6.25 | 5.27 | 3.922 | 3.307 | " |
| 24.94 | 33.31 | 7.69 | 4.88 | 4.879 | 3.097 | " |
| 31.66 | 28.94 | 10.11 | 4.39 | 6.563 | 2.851 | $Ni(ClO_4)_2.6H_2O + NaClO_4.H_2O$ |
| 30.73 | 29.42 | 9.74 | 4.43 | 6.298 | 2.866 | " |
| 30.44 | 29.56 | 9.62 | 4.44 | 6.215 | 2.869 | " |
| 30.38 | 29.76 | 9.63 | 4.48 | 6.225 | 2.898 | $NaClO_4.H_2O$ |
| 32.61 | 27.59 | 10.31 | 4.15 | 6.692 | 2.691 | " |
| 44.72 | 17.43 | 14.41 | 2.67 | 9.650 | 1.788 | " |
| 51.52 | 11.88 | 16.84 | 1.85 | 11.497 | 1.260 | " |
| 64.71 | 1.98 | 22.16 | 0.32 | 15.866 | 0.231 | " |
| 67.89 | - | 23.73 | - | 17.268 | - | " |

[a] Compiler's calculations.

Solution composition at the isothermal double saturation point ( solid phases $NaClO_4.H_2O$ and $Ni(ClO_4)_2).6H_2O$ : 30.80 mass %, 29.42 mass % $Ni(ClO_4)_2$, and 39.78 mass % $H_2O$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. The time of equilibration varied from 4 to 5 days. $Ni^{2+}$ was determined by titrating with Trilon B; $ClO_4^-$ gravimetrically, by nitron precipitation. The composition of the true solid phase was determined by Schreinemakers' method of "residues". | Not stated. |
| | ESTIMATED ERROR: |
| | Temperature: ±0.1 K |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Aluminium perchlorate; $Al(ClO_4)_3$; [14452-39-2]<br>(3) Water; $H_2O$; [7732-18-5] | Caven, R.M.; Bryce, G.<br><br>*J. Chem. Soc.* 1934, 514-7. |
| VARIABLES:<br>One temperature: 303.2 K<br>Composition | PREPARED BY:<br>K.H. Khoo |

EXPERIMENTAL VALUES:

Solubility system : $NaClO_4$-$Al(ClO_4)_3$-$H_2O$ at 30.2°C

| Liquid phase composition g/100 g(3) | | molality[a]/mol $kg^{-1}$ | | Solid phase |
|---|---|---|---|---|
| (1) | (2) | (1) | (2) | |
| 215.0 | - | 17.6 | - | $NaClO_4.H_2O$ |
| 135.4 | 27.76 | 11.1 | 0.85 | " |
| 116.6 | 37.30 | 9.52 | 1.15 | " |
| 100.7 | 45.59 | 8.22 | 1.40 | " |
| 50.64 | 72.46 | 4.14 | 2.23 | " |
| 43.84 | 76.26 | 3.58 | 2.34 | $NaClO_4.H_2O$+$Al(ClO_4)_3.nH_2O$ |
| 30.02 | 83.37 | 2.45 | 2.58 | $Al(ClO_4)_3.nH_2O$ |
| - | 110.1 | - | 3.38 | " |

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The isothermal method was used. Aluminium was determined as the oxide. Sodium was determined in the presence of aluminium as sodium magnesium uranyl acetate. The method of analysis of the solid phase was not mentioned. | Nothing specified. |
| | ESTIMATED ERROR:<br>Temperature: ±0.1°C |
| | REFERENCES:<br>None. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Druzhinina, G.V.; Paraguzova, T.V. |
| (2) Cerium perchlorate; $Ce(ClO_4)_3$; [14017-47-1] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1975, *144*, 76-85. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 298 K | I.S. Bodnya |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $Ce(ClO_4)_3$-$NaClO_4$-$H_2O$ at 298 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 67.80 | - | 23.65 | - | 17.197 | - | $NaClO_4.H_2O$ |
| 60.56 | 6.12 | 20.97 | 0.59 | 14.844 | 0.419 | " |
| 47.28 | 16.30 | 15.79 | 1.52 | 10.603 | 1.021 | " |
| 31.98 | 28.99 | 10.47 | 2.65 | 6.692 | 1.694 | " |
| 19.56 | 42.34 | 6.74 | 4.07 | 4.193 | 2.535 | " |
| 12.15 | 51.33 | 4.42 | 5.22 | 2.717 | 3.206 | " |
| 11.04 | 52.41 | 4.03 | 5.34 | 2.467 | 3.270 | " |
| 8.31 | 56.48 | 3.16 | 5.99 | 1.928 | 3.659 | " |
| 7.78 | 58.17 | 3.05 | 6.36 | 1.866 | 3.896 | " |
| 6.38 | 60.94 | 2.60 | 6.93 | 1.594 | 4.253 | $Ce(ClO_4)_3.9H_2O$ + $NaClO_4$ |
| 6.07 | 60.60 | 2.43 | 6.78 | 1.487 | 4.147 | " |
| 2.32 | 63.01 | 0.91 | 6.89 | 0.547 | 4.145 | $Ce(ClO_4)_3.9H_2O$ |
| - | 64.33 | - | 6.90 | - | 4.113 | " |

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. $ClO_4^-$ was determined gravimetrically as nitron perchlorate; $Ce^{3+}$ trilonometrically with the indicator xylenol orange. The composition of solid phases was determined graphically by Schreinemakers' method. The heating curves were recorded using Kurnakov's pyrometer to confirm the character of solid phases. I.R. spectra were studied using a IKS-14A spectrophotometer. | Not stated. |
| | ESTIMATED ERROR: Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Andronova, N.P. |
| (2) Terbium perchlorate; $Tb(ClO_4)_3$; [14014-09-6] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1976, *154*, 25-7. |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 298 K | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Tb(ClO_4)_3$-$H_2O$ at 298 K :

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 67.92 | - | 23.75 | - | 17.292 | - | $NaClO_4.H_2O$ |
| 51.42 | 13.84 | 17.66 | 1.27 | 12.089 | 0.871 | " |
| 33.27 | 30.59 | 11.59 | 2.85 | 7.519 | 1.851 | " |
| 18.52 | 45.37 | 6.71 | 4.40 | 4.189 | 2.748 | " |
| 17.75 | 47.57 | 6.67 | 4.79 | 4.180 | 3.000 | " |
| 16.36 | 49.46 | 6.25 | 5.06 | 3.909 | 3.165 | " |
| 13.85 | 51.90 | 5.32 | 5.33 | 3.303 | 3.314 | $NaClO_4$ |
| 12.50 | 53.98 | 4.91 | 5.67 | 3.046 | 3.522 | " |
| 11.81 | 55.40 | 4.73 | 5.95 | 2.942 | 3.695 | " |
| 7.99 | 59.31 | 3.25 | 6.45 | 1.996 | 3.967 | " |
| 6.96 | 61.34 | 2.91 | 6.88 | 1.793 | 4.232 | " |
| 6.68 | 63.39 | 2.94 | 7.48 | 1.823 | 4.632 | $NaClO_4$ + $Tb(ClO_4)_3.9H_2O$ |
| 3.53 | 63.70 | 1.45 | 7.01 | 0.880 | 4.251 | $Tb(ClO_4)_3.9H_2O$ |
| - | 64.30 | - | 6.63 | - | 3.939 | " |

[a] Compiler's calculation

Average solution composition at the isothermal double saturation point ( solid phases $NaClO_4$ and $Tb(ClO_4)_3.9H_2O$ ):
6.68 mass % $NaClO_4$, 63.39 mass % $Tb(ClO_4)_3$, and 29.93 mass % $H_2O$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details given. The solid phases were studied under a microscope and by differential thermal analysis. | Not stated. |
| | ESTIMATED ERROR: Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium chloride; NaCl; [7647-14-5]<br>(3) Water; $H_2O$; [7732-18-5] | Cornec, E.; Dickeley, J.<br>*Bul. Soc. Chim. France* 1927, *41*, 1017-27. |
| **VARIABLES:**<br>Temperature/K: 273-373<br>Composition | **PREPARED BY:**<br>K.H. Khoo |

EXPERIMENTAL VALUES:

Solubility of sodium perchlorate monohydrate at various temperatures (t)

| t/°C | 0 | 15 | 25 | 38 | 50 |
|---|---|---|---|---|---|
| g(2)/100 g(3) | 169 | 191 | 211 | 238 | 274 |
| molality[a]/ mol $kg^{-1}$ | 13.80 | 15.60 | 17.23 | 19.44 | 22.38 |

Solubility of anhydrous sodium perchlorate at various temperatures (t)

| t/°C | 15 | 25 | 38 | 55 | 75 | 100 |
|---|---|---|---|---|---|---|
| g(2)/100 g(3) | 256 | 260 | 268 | 284 | 300 | 330 |
| molality[a]/ mol $kg^{-1}$ | 20.91 | 21.23 | 21.89 | 23.19 | 24.50 | 26.95 |

Solubility of sodium perchlorate in aqueous sodium chloride

(i) At 0°C

| g(2)/100 g(3) | 0 | 6.70 | 10.21 | 17.15 | 25.44 |
|---|---|---|---|---|---|
| g(1)/100 g(3) | 169.4 | 158.0 | 123.5 | 80.2 | 39.3 |
| Solid phase[b] | A | A+B | B | B | B |

(ii) At 100°C

| g(2)/100 g(3) | 0 | 3.87 | 4.93 | 8.11 | 17.75 |
|---|---|---|---|---|---|
| g(1)/100 g(3) | 330.1 | 324.9 | 237.1 | 157.1 | 83.3 |
| Solid phase[b] | C | C+B | B | B | B |

(iii) At other temperatures

| Temperature/°C | 25 | 38 | 50 | 55 | 75 |
|---|---|---|---|---|---|
| g(2)/100 g(3) | 207.7 | 235.0 | 271.5 | 278.3 | 296.7 |
| g(1)/100 g(3) | 4.27 | 3.57 | 3.03 | 2.97 | 3.32 |
| Solid phase[b] | A+B | A+B | A+B | C+B | C+B |

[a] Compiler's calculations.

[b] A = $NaClO_4.H_2O$; B = NaCl; C = $NaClO_4$

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium chloride; NaCl; [7647-14-5]<br>(3) Water; $H_2O$; [7732-18-5] | Cornec, E.; Dickely, J.<br><br>*Bull. Soc. Chim. (France)* 1927, *41*, 1017-27. |
| VARIABLES:<br>Temperature: 273 - 373 K<br>Composition. | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility system $NaCl-NaClO_4-H_2O$ at various temperatures :

| t/ °C | Liquid phase composition mass% (2) | mass% (1) | mol % [a] (2) | mol % [a] (1) | mol kg$^{-1}$ [a] (2) | mol kg$^{-1}$ [a] (1) | Sln density g cm$^{-3}$ | Solid phase |
|---|---|---|---|---|---|---|---|---|
| 100 | - | 76.75 | - | 32.69 | - | 26.96 | 1.758 | $NaClO_4$ |
| " | 0.88 | 75.79 | 0.78 | 32.09 | 0.65 | 26.53 | 1.757 | $NaClO_4$ + NaCl |
| " | 1.44 | 69.32 | 1.11 | 25.57 | 0.84 | 19.36 | 1.664 | NaCl |
| " | 3.06 | 59.23 | 1.99 | 18.40 | 1.39 | 12.83 | 1.532 | " |
| " | 8.81 | 41.44 | 4.64 | 10.41 | 3.03 | 6.80 | 1.367 | " |
| 75 | 0.83 | 74.15 | 0.71 | 30.15 | 0.57 | 24.21 | 1.757 | $NaClO_4$ + NaCl |
| 55 | 0.78 | 73.00 | 0.65 | 28.87 | 0.51 | 22.74 | 1.755 | " " |
| 50 | 0.81 | 72.46 | 0.66 | 28.32 | 0.52 | 22.14 | 1.749 | $NaCl+NaClO_4.H_2O$ |
| 38 | 1.05 | 69.41 | 0.81 | 25.48 | 0.61 | 19.19 | 1.713 | " " |
| 25 | 1.37 | 66.58 | 1.00 | 23.18 | 0.73 | 16.97 | 1.683 | " " |
| 0 | - | 62.87 | - | 19.94 | - | 13.83 | - | $NaClO_4.H_2O$ |
| 0 | 2.53 | 59.69 | 1.65 | 18.55 | 1.15 | 12.90 | - | $NaCl+NaClO_4.H_2O$ |
| 0 | 4.37 | 52.82 | 2.59 | 14.97 | 1.746 | 10.08 | - | NaCl |
| 0 | 8.63 | 40.65 | 4.48 | 10.08 | 2.911 | 6.55 | - | " |
| 0 | 15.44 | 23.86 | 6.90 | 5.09 | 4.352 | 3.210 | - | " |

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details of saturation method given. The saturated solutions were evaporated in a water-bath and the solids dried at 110 °C in an oven, cooled and weighed in stoppered flasks. | Commercial sodium perchlorate was purified by several recrystallizations before use. |
| | ESTIMATED ERROR:<br>Not stated. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium chloride; NaCl; [7647-14-5]<br>(3) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.<br><br>*Izv. Vyssh. Uch. Zap. Khim. i. Khim. Tekhnolog.* 1958, *3*, 34-9. |
| VARIABLES:<br>One temperature: 293 K<br>Composition | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-NaCl-$H_2O$ at 293 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| - | 66.84 | - | 22.87 | - | 16.463 | $NaClO_4$ [b] |
| 0.82 | 66.30 | 0.59 | 22.75 | 0.427 | 16.469 | " |
| 1.62 | 65.55 | 1.16 | 22.44 | 0.844 | 16.307 | " |
| 1.78 | 64.80 | 1.26 | 21.92 | 0.911 | 15.836 | " |
| 1.83 | 64.26 | 1.28 | 21.52 | 0.923 | 15.477 | $NaClO_4$[b] + NaCl |
| 1.77 | 64.23 | 1.24 | 21.48 | 0.891 | 15.429 | " |
| 1.85 | 64.11 | 1.29 | 21.42 | 0.930 | 15.382 | NaCl |
| 3.97 | 53.43 | 2.37 | 15.21 | 1.595 | 10.244 | " |
| 10.64 | 38.19 | 5.46 | 9.35 | 3.558 | 6.096 | " |
| 13.53 | 31.86 | 6.57 | 7.39 | 4.239 | 4.765 | " |
| 14.83 | 25.12 | 6.69 | 5.41 | 4.226 | 3.417 | " |
| 21.68 | 10.32 | 8.77 | 1.99 | 5.455 | 1.239 | " |
| 22.46 | 8.18 | 8.93 | 1.55 | 5.541 | 0.963 | " |
| 23.59 | 5.39 | 9.19 | 1.00 | 5.684 | 0.620 | " |
| 26.50 | - | 10.00 | - | 6.169 | - | " |

[a] Compiler's calculation.

[b] probably $NaClO_4 \cdot H_2O$

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method (ref. 1) was used. Equilibrium of the saturated solutions was reached in 2 days. $Na^+$ was determined gravimetrically as sodium zinc uranyl acetate; $Cl^-$ by Mohr's method and in the transition points, gravimetrically as silver chloride. | Chemically pure salts were further purified by double recrystallization. Analysis gave 99.41 % to 99.63 % purity. |
| | ESTIMATED ERROR:<br>Not stated. |

REFERENCES:

1. Karnaukhov, A.S. *Zh. Neorg. khim.* 1957, *2*, 915.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium chloride; NaCl; [7647-14-5]<br>(3) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Kudryakova, S.A.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* **1966**, *59*, 119-36. |
| VARIABLES:<br>One temperature: 298.2 K<br>Composition | PREPARED BY:<br>I.S. Bodnya |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-NaCl-$H_2O$ at 298.2 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 26.45 | - | 9.98 | - | 6.153 | - | NaCl |
| 24.33 | 4.27 | 9.43 | 0.79 | 5.831 | 0.488 | " |
| 21.09 | 11.39 | 8.59 | 2.21 | 5.345 | 1.378 | " |
| 18.32 | 17.50 | 7.80 | 3.56 | 4.884 | 2.227 | " |
| 14.73 | 25.49 | 6.67 | 5.51 | 4.216 | 3.482 | " |
| 11.20 | 33.18 | 5.40 | 7.63 | 3.446 | 4.872 | " |
| 8.49 | 40.41 | 4.39 | 9.97 | 2.843 | 6.459 | " |
| 5.19 | 49.88 | 2.97 | 13.62 | 1.977 | 9.067 | " |
| 1.31 | 66.34 | 0.95 | 22.96 | 0.693 | 16.749 | " |
| 1.33 | 66.29 | 0.96 | 22.93 | 0.703 | 16.720 | NaCl + $NaClO_4.H_2O$ |
| 1.35 | 66.18 | 0.98 | 22.85 | 0.711 | 16.646 | " |
| 1.38 | 66.11 | 1.00 | 22.80 | 0.726 | 16.608 | " |
| 1.36 | 66.11 | 0.98 | 22.79 | 0.715 | 16.598 | " |
| 1.41 | 65.69 | 1.01 | 22.48 | 0.733 | 16.307 | " |
| 1.41 | 65.92 | 1.02 | 22.66 | 0.738 | 16.479 | " |
| 1.42 | 66.03 | 1.03 | 22.75 | 0.746 | 16.568 | $NaClO_4.H_2O$ |
| 0.91 | 66.66 | 0.66 | 23.07 | 0.480 | 16.788 | " |
| - | 67.84 | - | 23.69 | - | 17.228 | " |

[a] Compiler's calculation.

COMMENTS AND/OR ADDITIONAL DATA:

Solution composition at the isothermal double saturation point ( solid phases $NaClO_4.H_2O$ and NaCl ):

1.36 mass % NaCl, 66.09 mass % $NaClO_4$, and 32.55 mass % $H_2O$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Equilibrium was reached in 20-70 hours. $Na^+$ was determined gravimetrically as sodium zinc uranyl acetate; $Cl^-$ mercurimetrically; $ClO_4^-$ gravimetrically by nitron precipitation. | The initial salts were recrystallized twice. Purity of the salts varied from 95.58 to 99.75%. |
| | ESTIMATED ERROR:<br>Temperature: ± 0.1 K |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium chloride; NaCl; [7647-14-5]<br>(3) Water; $H_2O$; [7732-18-5] | Loseva, G.K.<br>*Tr. Novocherkassk. Politekhn. Inst.* 1972, *266*, 78-81 |
| VARIABLES:<br>One temperature: 333 K<br>Composition | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility, system $NaClO_4$-NaCl-$H_2O$

| liquid phase composition | | | | | | solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 13.70 | 20.40 | 2.72 | 8.47 | 1.698 | 5.297 | NaCl |
| 31.70 | 12.40 | 7.24 | 5.94 | 4.632 | 3.796 | " |
| 41.30 | 8.50 | 10.32 | 4.45 | 6.719 | 2.897 | " |
| 49.50 | 5.60 | 13.51 | 3.20 | 9.004 | 2.134 | " |
| 55.70 | 3.70 | 16.41 | 2.28 | 11.205 | 1.559 | " |
| 66.00 | 2.40 | 23.09 | 1.76 | 17.058 | 1.300 | " |
| 66.80 | 1.60 | 23.45 | 1.18 | 17.265 | 0.866 | " |
| 69.60 | 1.20 | 25.72 | 0.93 | 19.467 | 0.703 | " |
| 71.70 | 0.99 | 27.64 | 0.80 | 21.442 | 0.620 | " |
| 72.80 | 0.79 | 28.67 | 0.65 | 22.513 | 0.512 | NaCl + $NaClO_4$ |
| 74.20 | - | 29.73 | - | 23.489 | - | $NaClO_4$ |

[a] Compiler's calculation

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method was used. The composition of the liquid phase was determined by chemical analysis. The composition of the solid phase was determined using Schreinemaker's method.

SOURCE AND PURITY OF MATERIALS:

The reagents were purified by recrystallization ; final components were 99.6-99.6 % pure.

ESTIMATED ERROR:

not stated

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Karnaukhov, A S., Troitskii, E.N. |
| (2) Sodium chloride; NaCl; [7647-14-5] | *Uch Zap. Yarosl Gos. Ped. Inst.* 1966, *59*, 8-21. |
| (3) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 363 K | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-NaCl-$H_2O$ at 363 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 27.80 | - | 10.61 | - | 6.588 | - | NaCl |
| 25.11 | 5.05 | 9.88 | 0.95 | 6.152 | 0.591 | " |
| 22.95 | 8.72 | 9.23 | 1.67 | 5.747 | 1.042 | " |
| 18.97 | 18.96 | 8.27 | 3.95 | 5.229 | 2.495 | " |
| 14.80 | 23.85 | 6.57 | 5.05 | 4.128 | 3.175 | " |
| 12.10 | 29.94 | 5.64 | 6.66 | 3.572 | 4.219 | " |
| 9.12 | 38.03 | 4.59 | 9.13 | 2.953 | 5.877 | " |
| 6.80 | 45.20 | 3.69 | 11.72 | 2.424 | 7.691 | " |
| 6.00 | 47.99 | 3.37 | 12.86 | 2.231 | 8.519 | " |
| 5.53 | 49.97 | 3.18 | 13.73 | 2.126 | 9.171 | " |
| 4.60 | 52.62 | 2.73 | 14.91 | 1.840 | 10.046 | " |
| 3.78 | 55.77 | 2.34 | 16.47 | 1.599 | 11.260 | " |
| 3.15 | 58.64 | 2.03 | 18.05 | 1.411 | 12.534 | " |
| 1.85 | 63.20 | 1.27 | 20.75 | 0.906 | 14.769 | " |
| 0.90 | 68.09 | 0.67 | 24.25 | 0.497 | 17.933 | " |
| 0.87 | 67.34 | 0.64 | 23.61 | 0.468 | 17.300 | NaCl + $NaClO_4$ |
| 0.88 | 66.93 | 0.64 | 23.28 | 0.468 | 16.981 | " |
| 0.85 | 67.31 | 0.62 | 23.58 | 0.457 | 17.266 | " |
| 0.86 | 68.06 | 0.64 | 24.21 | 0.473 | 17.885 | " |
| 0.87 | 67.17 | 0.64 | 23.47 | 0.466 | 17.165 | " |
| 0.90 | 66.42 | 0.65 | 22.87 | 0.471 | 16.599 | " |
| 0.89 | 66.95 | 0.65 | 23.30 | 0.474 | 17.002 | $NaClO_4$ |
| - | 75.85 | - | 31.61 | - | 25.652 | " |

[a] Compiler's calculation.

Average solution composition at the isothermal double saturation point ( solid phases $NaClO_4$ and NaCl ):

0.87 mass % NaCl, 67.21 mass % $NaClO_4$, and 31.92 mass% $H_2O$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE | SOURCE AND PURITY OF MATERIALS. |
|---|---|
| Isothermal method was used. Conditions of saturation were not given. $Na^+$ was determined gravimetrically as sodium zinc uranyl acetate; $ClO_4^-$ gravimetrically by precipitating with nitron; $Cl^-$ mercurimetrically. | Not stated. |
| | ESTIMATED ERROR: Not stated. |
| | REFERENCES |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium nitrate; $NaNO_3$; [7631-99-4]<br>(3) Water; $H_2O$; [7732-18-5] | Andronova, N.P.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1971, *95*, 142-6. |
| VARIABLES:<br>Temperature: 298 and 323 K<br>Composition | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system $NaNO_3$-$NaClO_4$-$H_2O$ at 298 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 47.82 | - | 16.27 | - | 10.782 | - | $NaNO_3$ |
| 42.88 | 8.12 | 15.33 | 2.02 | 10.296 | 1.353 | " |
| 33.68 | 20.38 | 12.73 | 5.35 | 8.626 | 3.623 | " |
| 29.47 | 24.85 | 11.24 | 6.58 | 7.590 | 4.443 | " |
| 25.82 | 35.56 | 11.10 | 10.61 | 7.866 | 7.520 | " |
| 19.37 | 47.52 | 9.29 | 15.82 | 6.883 | 11.722 | " |
| 15.50 | 55.50 | 8.12 | 20.19 | 6.288 | 15.630 | " |
| 11.59 | 59.19 | 6.08 | 21.56 | 4.667 | 16.544 | $NaNO_3$ + $NaClO_4.H_2O$ |
| 11.53 | 59.28 | 6.06 | 21.61 | 4.647 | 16.586 | " |
| 7.84 | 62.32 | 4.09 | 22.55 | 3.091 | 17.057 | $NaClO_4.H_2O$ |
| 4.98 | 64.13 | 2.55 | 22.80 | 1.897 | 16.956 | " |
| - | 67.89 | - | 23.73 | - | 17.268 | " |

[a] Compiler's calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Saturation conditions were not given. The composition of solid phases was determined graphically by Schreinemakers' method of "residues". The solutions and solid "residues" were analysed for the nitrate ion by Devarda's method. The perchlorate ion was precipitated with nitron. | Not stated. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES:<br><br>(continued next page) |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Andronova, N.P. |
| (2) Sodium nitrate; $NaNO_3$; [7631-99-4] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1971, *95*, 142-6. |
| (3) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system $NaNO_3$-$NaClO_4$-$H_2O$ at 323 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 53.27 | - | 19.46 | - | 13.412 | - | $NaNO_3$ |
| 48.42 | 6.50 | 18.23 | 1.70 | 12.637 | 1.178 | " |
| 41.70 | 18.30 | 17.15 | 5.23 | 12.265 | 3.737 | " |
| 33.41 | 31.78 | 15.21 | 10.04 | 11.292 | 7.456 | " |
| 24.40 | 43.67 | 11.88 | 14.76 | 8.991 | 11.170 | " |
| 18.14 | 57.23 | 10.42 | 22.82 | 8.665 | 18.977 | " |
| 16.98 | 59.07 | 9.93 | 23.98 | 8.341 | 20.144 | $NaNO_3$ + $NaClO_4$ |
| 16.98 | 59.07 | 9.93 | 23.98 | 8.341 | 20.144 | " |
| 14.81 | 60.82 | 8.61 | 24.55 | 7.150 | 20.383 | $NaClO_4$ |
| 10.05 | 65.17 | 5.84 | 26.27 | 4.772 | 21.479 | " |
| 5.70 | 69.20 | 3.31 | 27.90 | 2.672 | 22.517 | " |
| 3.37 | 70.84 | 1.93 | 28.23 | 1.537 | 22.434 | $NaClO_4.H_2O$ |
| - | 73.53 | - | 29.01 | - | 22.687 | " |

[a] Compiler's calculation.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Freeth, F.A. |
| (2) Sodium sulfate; $Na_2SO_4$; [7757-82-6] | *Rec. Trav. Chim. Pays-Bas* 1924, 43, 475 |
| (3) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Two temperatures: 298 K and 333 K. | C.Y. Chan |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Na_2SO_4$-$H_2O$ at 25 °C :

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol % [a] | | molality[a]/ mol kg$^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 67.60 | - | 23.49 | - | 17.04 | - | $NaClO_4.H_2O$ |
| 67.67 | 0.26 | 23.67 | 0.08 | 17.23 | 0.06 | $NaClO_4.H_2O$ + $Na_2SO_4$ |
| 53.58 | 1.24 | 14.81 | 0.296 | 9.69 | 0.193 | $Na_2SO_4$ |
| 41.68 | 4.28 | 10.10 | 0.894 | 6.30 | 0.558 | " |
| 31.27 | 9.07 | 7.034 | 1.759 | 4.281 | 1.070 | $Na_2SO_4$ + $Na_2SO_4.10H_2O$ |
| 31.21 | 9.07 | 7.015 | 1.757 | 4.268 | 1.069 | " + " |
| 18.72 | 12.56 | 3.761 | 2.180 | 2.225 | 1.287 | $Na_2SO_4.10H_2O$ |
| 5.79 | 18.15 | 1.075 | 2.906 | 0.622 | 1.671 | " |
| - | 21.71 | - | 3.398 | - | 1.952 | " |

[a] Compiler's calculations.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The saturation apparatus was similar to that used by Van't Hoff (ref.1) and samples of clear satd sln were taken using a weight-pipette. $Na^+$ was determined as $Na_2SO_4$ by addition of pure sulphuric acid to the sln in silica basins and evaporating at a low red heat. Sulfate was determined gravimetrically as $BaSO_4$. Solid phase compositions were determined using Schreinemakers' method. Gas-heated thermostats were used and thermometers were checked against N.P.L. Standards. All analyses were carried out in duplicates. | $NaClO_4$ was prepared from very pure ammonium perchlorate (% purity not stated) and an aqueous sln of pure NaOH. Source and other details not given. $NaSO_4$ was recrystallized from the reagent grade salt. |
| | **ESTIMATED ERROR:** |
| | No estimation. |
| | **REFERENCES:** |
| | 1. Van't Hoff, J.H. *Zur Bildung der Ozeanischen Salzablagerungen Wieweg, Braunschweig* 1905, *1*; 1902, *2*. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium chlorate; $NaClO_3$; [7775-09-9]<br>(3) Water; $H_2O$; [7732-18-5] | Loseva, G.K.<br><br>*Tr. Novocherkassk. Politekhn. Inst.* 1972, *266*, 78-81. |
| VARIABLES:<br>One temperature: 333 K<br>Composition | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$NaClO_3$-$H_2O$ at 333 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol % | | molality/mol $kg^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 51.40 | 9.20 | 17.59 | 2.74 | 12.256 | 1.907 | $NaClO_3$ |
| 40.10 | 23.00 | 14.42 | 7.19 | 10.210 | 5.091 | " |
| 30.90 | 35.90 | 11.96 | 12.08 | 8.744 | 8.831 | " |
| 23.40 | 47.70 | 9.93 | 17.60 | 7.607 | 13.480 | " |
| 20.10 | 55.10 | 9.37 | 22.33 | 7.614 | 18.146 | " |
| 16.80 | 59.90 | 8.13 | 25.21 | 6.774 | 20.996 | " |
| 15.30 | 62.70 | 7.66 | 27.28 | 6.534 | 23.277 | $NaClO_3$ + $NaClO_4$ |
| 14.80 | 62.90 | 7.35 | 27.17 | 6.235 | 23.037 | $NaClO_4$ |
| 14.10 | 63.50 | 6.99 | 27.38 | 5.914 | 23.153 | " |
| 7.90 | 68.30 | 3.80 | 28.56 | 3.118 | 23.438 | " |

[a] Compiler's calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. The composition of the liquid phase was determined by chemical analysis. The composition of the solid phase was determined by Schreinemakers' method. The viscosity, density and electrical conductivity were measured. X-ray powder analysis and optical crystallography methods were used. | The reagents were purified by recrystallization until the content of the main substance was 99.6-99 % pure. |
| | ESTIMATED ERROR:<br>Not given. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Freeth, F.A. |
| (2) Sodium sulfate; $Na_2SO_4$; [7757-82-6] | *Rec. Trav. Chim. Pays-Bas* 1924, 43, 475 |
| (3) Water; $H_2O$; [7732-18-5] | |

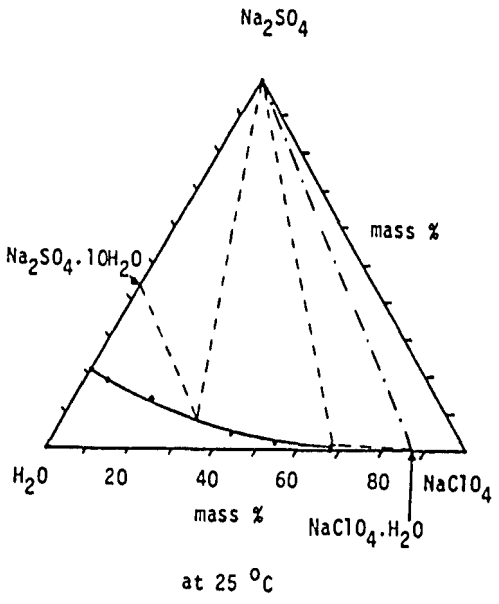
EXPERIMENTAL VALUES:(continued)

Solubility system $NaClO_4$-$Na_2SO_4$-$H_2O$ at 60 °C :

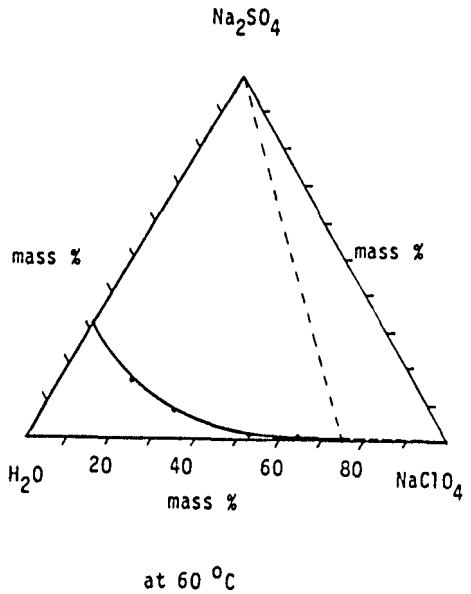
| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol % [a] | | molality[a]/ mol $kg^{-1}$ | | |
| (1) | (2) | (1) | (2) | (1) | (2) | |
| 74.30 | - | 29.84 | - | 23.61 | - | $NaClO_4$ |
| 74.40 | 0.290 | 30.16 | 0.101 | 24.01 | 0.081 | $NaClO_4$ + $Na_2SO_4$ |
| 64.65 | 0.370 | 21.36 | 0.105 | 15.095 | 0.074 | $Na_2SO_4$ |
| 52.47 | 1.11 | 14.22 | 0.259 | 9.232 | 0.168 | " |
| 31.55 | 6.95 | 6.93 | 1.315 | 4.181 | 0.796 | " |
| 17.70 | 14.90 | 3.622 | 2.629 | 2.145 | 1.556 | " |
| - | 31.2 | - | 5.44 | - | 3.19 | " |

[a] Compiler's calculations.

COMMENTS AND/OR ADDITIONAL DATA The solubility isotherms for the system at 25 °C and 60 °C are reproduced below.




at 25 °C




at 60 °C

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium chromate; $Na_2CrO_4$; [7775-11-3]<br>(3) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Guseva, A.D.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, *59*, 96-103 |
| VARIABLES:<br>One temperature: 298 K<br>Composition | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Na_2CrO_4$-$H_2O$ at 308 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol % | | molality/mol $kg^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 47.63 | 0.00 | 9.19 | 0.00 | 5.615 | 0.000 | $Na_2CrO_4.4H_2O$ |
| 45.69 | 2.82 | 8.92 | 0.73 | 5.478 | 0.447 | " |
| 40.63 | 10.20 | 8.19 | 2.72 | 5.101 | 1.694 | " |
| 38.07 | 13.39 | 7.73 | 3.60 | 4.842 | 2.253 | " |
| 33.14 | 20.35 | 6.93 | 5.63 | 4.399 | 3.573 | " |
| 30.60 | 24.04 | 6.51 | 6.76 | 4.165 | 4.328 | " |
| 27.84 | 28.22 | 6.05 | 8.11 | 3.912 | 5.245 | " |
| 24.81 | 33.26 | 5.57 | 9.87 | 3.653 | 6.478 | " |
| 20.43 | 39.60 | 4.73 | 12.12 | 3.156 | 8.092 | " |
| 15.82 | 46.45 | 3.80 | 14.75 | 2.589 | 10.055 | " |
| 13.80 | 49.77 | 3.39 | 16.17 | 2.339 | 11.158 | " |
| 12.02 | 53.15 | 3.04 | 17.78 | 2.131 | 12.463 | " |
| 10.44 | 56.41 | 2.72 | 19.48 | 1.944 | 13.898 | " |
| 9.38 | 59.27 | 2.54 | 21.21 | 1.847 | 15.441 | " |
| 8.63 | 61.62 | 2.41 | 22.79 | 1.791 | 16.916 | $Na_2CrO_4.4H_2O$ + $NaClO_4.H_2O$ |
| 8.61 | 61.48 | 2.40 | 22.66 | 1.777 | 16.788 | " |
| 8.62 | 61.52 | 2.40 | 22.70 | 1.782 | 16.827 | " |
| 8.60 | 61.68 | 2.41 | 22.83 | 1.786 | 16.950 | " |
| 8.61 | 61.63 | 2.41 | 22.79 | 1.786 | 16.914 | " |
| 5.63 | 64.32 | 1.56 | 23.58 | 1.157 | 17.481 | $NaClO_4.H_2O$ |
| 2.40 | 67.01 | 0.66 | 24.22 | 0.484 | 17.891 | " |
| 0.00 | 69.80 | 0.00 | 25.38 | 0.000 | 18.877 | " |

[a] Compiler's calculation.

Solution composition at the isothermal double saturation point ( solid phases $NaClO_4.H_2O$ and $Na_2CrO_4.4H_2O$ ):
61.58 mass % $NaClO_4$, 8.61 mass % $Na_2CrO_4$, and 29.81 mass % $H_2O$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Equilibrium was reached in 2-3 days. $Na^+$ was determined gravimetrically as zinc uranyl acetate; $CrO_4^{-2}$ iodimetrically; $ClO_4^-$ by difference. | The initial salts (reagent grade) were purified by recrystallization. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium chromate; $Na_2CrO_4$; [7775-11-3]<br>(3) Water; $H_2O$; [7732-18-5] | Druzhinina, G.V.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, *59*, 73-82. |
| VARIABLES:<br>One temperature: 323 K<br>Composition | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility in the system $NaClO_4$-$Na_2CrO_4$-$H_2O$ at 323K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 50.76 | - | 10.29 | - | 6.364 | - | $Na_2CrO_4.4H_2O$ |
| 49.38 | 2.51 | 10.18 | 0.68 | 6.337 | 0.426 | " |
| 48.19 | 4.58 | 10.06 | 1.27 | 6.299 | 0.792 | " |
| 46.11 | 8.73 | 9.94 | 2.49 | 6.303 | 1.579 | " |
| 41.94 | 12.71 | 8.99 | 3.60 | 5.709 | 2.289 | " |
| 38.84 | 17.05 | 8.48 | 4.92 | 5.436 | 3.157 | " |
| 35.40 | 21.83 | 7.89 | 6.43 | 5.110 | 4.169 | " |
| 31.91 | 26.44 | 7.23 | 7.92 | 4.730 | 5.185 | " |
| 27.05 | 33.55 | 6.35 | 10.43 | 4.238 | 6.955 | " |
| 23.02 | 40.88 | 5.73 | 13.46 | 3.937 | 9.249 | " |
| 19.47 | 47.02 | 5.08 | 16.24 | 3.587 | 11.460 | $Na_2CrO_4$ |
| 15.69 | 51.35 | 4.13 | 17.88 | 2.939 | 12.724 | " |
| 12.95 | 56.15 | 3.55 | 20.35 | 2.587 | 14.841 | " |
| 10.82 | 58.98 | 3.00 | 21.65 | 2.212 | 15.950 | " |
| 9.69 | 61.16 | 2.75 | 22.94 | 2.052 | 17.136 | " |
| 7.71 | 64.58 | 2.25 | 24.96 | 1.718 | 19.034 | $Na_2CrO_4$ |
| 6.41 | 67.19 | 1.93 | 26.72 | 1.499 | 20.786 | " |
| 6.38 | 67.23 | 1.92 | 26.74 | 1.493 | 20.806 | $Na_2CrO_4$ + $NaClO_4.H_2O$ |
| 6.42 | 67.28 | 1.93 | 26.82 | 1.507 | 20.893 | " |
| 6.37 | 67.20 | 1.91 | 26.70 | 1.488 | 20.766 | " |
| 6.40 | 67.21 | 1.92 | 26.73 | 1.497 | 20.800 | $NaClO_4.H_2O$ |
| 2.40 | 70.73 | 0.71 | 27.72 | 0.551 | 21.499 | " |
| - | 78.20 | - | 34.55 | - | 29.297 | " |

[a] Compiler's calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The solubility was studied by the method of isothermal recrystallization from supersaturated solutions. Conditions of saturation are not stated. $Na^+$ was determined as sodium zinc uranyl acetate and by flame photometry $CrO_4^{-2}$ iodimetrically and $ClO_4^-$ by difference. | Chemically pure grade (1) and (2) were purified by recrystallization and dehydrated. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES: None. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium chromate; $Na_2CrO_4$; [7775-11-3]<br>(3) Water; $H_2O$; [7732-18-5] | Molchanov, S.N.<br><br>*Tr. po Kimii i Khim. Tekhnolog.* 1965, *3(14)*, 27-9. |
| VARIABLES:<br>Temperature: 298, 323, 335.8 K<br>Composition | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Na_2CrO_4$-$H_2O$:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol kg$^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| **At 298 K** | | | | | | |
| 45.78 | - | 8.58 | - | 5.213 | - | $Na_2CrO_4.4H_2O$ |
| 7.82 | 53.91 | 1.85 | 16.85 | 1.261 | 11.505 | $Na_2CrO_4$ |
| 6.18 | 60.71 | 1.61 | 20.90 | 1.152 | 14.975 | $Na_2CrO_4$ + $NaClO_4.H_2O$ |
| - | 67.70 | - | 23.57 | - | 17.118 | $NaClO_4.H_2O$ |
| **At 323 K** | | | | | | |
| 50.76 | - | 10.29 | - | 6.364 | - | $Na_2CrO_4.4H_2O$ |
| 15.98 | 52.15 | 4.30 | 18.57 | 3.096 | 13.364 | $Na_2CrO_4$ |
| 6.39 | 67.82 | 1.95 | 27.35 | 1.530 | 21.477 | $Na_2CrO_4$ + $NaClO_4.H_2O$ |
| - | 73.20 | - | 28.67 | - | 22.308 | $NaClO_4.H_2O$ |
| **At 335.8 K** | | | | | | |
| mass % | | mol % | | molality/mol kg$^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 55.01 | - | 11.97 | - | 7.549 | - | $Na_2CrO_4$ |
| 4.55 | 73.13 | 1.51 | 32.04 | 1.259 | 26.759 | $Na_2CrO_4$ + $NaClO_4$ |
| - | 74.33 | - | 29.88 | - | 23.649 | $NaClO_4$ |

[a] Compiler's calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Periods of equilibrium varied from several hours at 62.8°C to 15-25 days at 25°C. $Na^+$ was determined as sodium zinc uranyl acetate; $CrO_4^{2-}$ iodimetrically. Solid phase was analysed microscopically, the decomposition curves were recorded with Kurnakov's pyrometer. | (1) was reagent grade and (2) was pure (no details). The salts were twice recrystallized. Analysis gave: (1) 99.78 %;<br>(2) 99.92 - 100.00 %. |
| | ESTIMATED ERROR:<br>Not stated. |

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium dichromate; $Na_2Cr_2O_7$; [10558-01-9]<br>(3) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Sal'nikova, L.N.<br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *78*, 65-70. |
| VARIABLES:<br>One temperature: 298.2 K<br>Composition | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Na_2Cr_2O_7$-$H_2O$ at 298.2 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol % | | molality/mol $kg^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 65.16 | - | 11.40 | - | 7.139 | - | $Na_2Cr_2O_7.2H_2O$ |
| 64.06 | 1.78 | 11.35 | 0.67 | 7.158 | 0.426 | " |
| 62.68 | 4.69 | 11.45 | 1.83 | 7.332 | 1.174 | " |
| 53.26 | 15.50 | 9.85 | 6.13 | 6.508 | 4.052 | " |
| 45.63 | 27.95 | 9.32 | 12.21 | 6.592 | 8.640 | " |
| 36.16 | 36.87 | 7.13 | 15.55 | 5.118 | 11.165 | " |
| 33.37 | 40.80 | 6.72 | 17.59 | 4.931 | 12.901 | " |
| 31.89 | 42.90 | 6.50 | 18.72 | 4.829 | 13.898 | " |
| 32.02 | 42.98 | 6.57 | 18.86 | 4.889 | 14.041 | $Na_2Cr_2O_7.2H_2O$ + $NaClO_4.H_2O$ |
| 23.23 | 49.04 | 4.37 | 19.75 | 3.198 | 14.444 | $NaClO_4.H_2O$ |
| 18.04 | 53.26 | 3.28 | 20.74 | 2.399 | 15.156 | " |
| 12.74 | 57.94 | 2.26 | 22.02 | 1.659 | 16.139 | " |
| 4.15 | 59.90 | 0.63 | 19.56 | 0.441 | 13.608 | " |
| 0.87 | 67.14 | 0.14 | 23.56 | 0.104 | 17.141 | " |
| - | 67.80 | - | 23.65 | - | 17.197 | " |

[a] Compiler's calculation

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Equilibrium was reached in 4-6 days. $Na^+$ was determined with zinc uranyl acetate. $Cr_2O_7^{2-}$ iodimetrically; $ClO_4^-$ by difference. Solid compositions were determined by Schreinemakers' method. | Not stated |
| | ESTIMATED ERROR:<br>Temperature: ± 0.1 K |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium phosphate; $Na_3PO_4$; [7601-54-9]<br>(3) Water; $H_2O$; [7732-18-5] | Babayan, G.G.; Darbinyan, G.M.<br><br>*Arm. Khim. Zh.* ,1972, *25*, 482-7. |
| VARIABLES:<br>One temperature: 293 K<br>Composition | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Na_3PO_4$-$H_2O$ at 293 K:

| Liquid phase composition | | | | | | Solid phase |
|---|---|---|---|---|---|---|
| mass % | | mol %[a] | | molality[a]/mol $kg^{-1}$ | | |
| (2) | (1) | (2) | (1) | (2) | (1) | |
| 0.39 | 58.40 | 0.09 | 17.24 | 0.058 | 11.574 | $NaClO_4.H_2O$ |
| 0.40 | 52.80 | 0.08 | 14.23 | 0.052 | 9.214 | solid solutions[b] |
| 0.47 | 47.70 | 0.09 | 11.92 | 0.055 | 7.516 | " |
| 2.14 | 41.60 | 0.38 | 9.78 | 0.232 | 6.039 | " |
| 1.80 | 41.35 | 0.31 | 9.64 | 0.193 | 5.940 | " |
| 2.54 | 35.87 | 0.42 | 7.86 | 0.252 | 4.757 | " |
| 2.90 | 34.70 | 0.47 | 7.53 | 0.283 | 4.542 | " |
| 3.20 | 26.00 | 0.47 | 5.10 | 0.276 | 2.999 | $Na_3PO_4.12H_2O$ |
| 4.80 | 21.80 | 0.68 | 4.16 | 0.399 | 2.426 | " |
| 12.46 | 9.80 | 1.70 | 1.79 | 0.978 | 1.030 | " |
| 18.42 | 4.48 | 2.54 | 0.83 | 1.457 | 0.475 | " |
| 14.46 | 8.20 | 1.98 | 1.51 | 1.140 | 0.866 | " |
| 23.90 | 1.60 | 3.39 | 0.30 | 1.957 | 0.175 | " |

[a] Compiler's calculation.

[b] Composition not known.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Isothermal method was used. Periods of equilibration were 5-7 days. Phosphate ion was detrmined by precipitating with ammonium molybdate in acidic solution; perchlorate ion gravimetrically. The composition of the solid phase was determined by Schreinemakers' method of residues . The composition of the solid phase was confirmed by thermographic, optical crystallographic and X-ray diffraction methods. | The original salts were reagent grade and were chemically pure. |
| | ESTIMATED ERROR:<br>Not stated. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium Chloride; [7647-14-5]<br>(3) Ammonia; $NH_3$; [7664-41-7] | Marshall, P.R.; Hunt, H.<br><br>*J. Chem. Eng. Data* 1959, *4*, 217-22. |
| VARIABLES: | PREPARED BY: |
| Temperature: 240 - 323 K. | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility system $NaCl-NaClO_4-H_2O$ at various temperatures, the solid phase being a mixture of the anhydrous salts :

| t/ °C | g / 100g(3) | | molality/ mol $kg^{-1}$ | | mol % (compiler) | |
|---|---|---|---|---|---|---|
| | (2) | (1) | (2) | (1) | (2) | (1) |
| -33 | 1.88 | 236 | 0.322 | 19.25 | 0.411 | 24.61 |
| 0 | 0.20 | 298.4 | 0.0343 | 24.4 | 0.041 | 29.32 |
| 25 | 0.222 | 299 | 0.038 | 24.4 | 0.046 | 29.36 |
| 50 | - | 310.0 | - | 25.3 | - | 30.13 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility determinations were carried out using a specially constructed apparatus (diagram given in original paper), involving a gas line connected to the saturation cell. The cell consisted of two compartments separated by a sintered glass partition, the larger one of which was connected to the gas line in such a way that the cell could be inverted, with either one of the compartments vertically above the other. Weighed amts of the salts were sealed in the smaller compartment of the cell which was then connected to the gas line via the larger compartment. Excess of dry ammonia was condensed in the cell until the salts had all dissolved at the set temperature. The coolants used were dry ice and $CCl_4$. The cell was thermostated in a liquid $NH_3$ bath for -33 °C determinations, in an ice + water bath for 0 °C, and in a water bath for the other temperatures. Ammonia was bled from the solution until salt crystals were formed, and the cell inverted so that the solution filtered through the partition into the larger compartment. After filtration the ammonia in the solution was all removed by condensation into a reservoir in the apparatus and determined quantitatively by absorption in std. HCl sln and back-titrated with std. base. The cell was then opened and the solids removed for analysis. Chloride was determined by titration with $AgNO_3$ using dichlorofluorescein as indicator.

SOURCE AND PURITY OF MATERIALS:

Not stated. Ammonia was dried with sodium.

ESTIMATED ERROR: Reproducibility (3 detn) is within ± 2 % of the mean value in most cases.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-07] | Marshall, P.R.; Hunt, H. |
| (2) Ammonium perchlorate; $NH_4ClO_4$ [7790-98-9] | *J. Chem. Eng. Data* 1959, *4*, 217-22. |
| (3) Ammonia; $NH_3$; [7664-41-7] | |
| VARIABLES: | PREPARED BY: |
| Temperature: 240 - 323 K. | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$NH_4ClO_4$-$NH_3$ at various temperatures, the solid phase being a mixture of the anhydrous salts :

| t/ °C | g / 100g(3) | | molality/ mol $kg^{-1}$ | | mol % (compiler) | |
|---|---|---|---|---|---|---|
| | (1) | (2) | (1) | (2) | (1) | (2) |
| -33 | 210.3 | 42.5 | 17.1 | 3.6 | 21.60 | 4.55 |
| 0 | 241.7 | 45.7 | 19.75 | 3.89 | 23.97 | 4.72 |
| 25 | 267.0 | 47.0 | 21.8 | 4.00 | 25.80 | 4.73 |
| 50 | 292.2 | 49.6 | 23.8 | 4.22 | 27.49 | 4.86 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility determinations were carried out using a specially constructed apparatus (diagram given in original paper), involving a gas line connected to the saturation cell. The cell consisted of two compartments separated by a sintered glass partition, the larger one of which was connected to the gas line in such a way that the cell could be inverted, with either one of the compartments vertically above the other. Weighed amts of the salts were sealed in the smaller compartment of the cell which was then connected to the gas line via the larger compartment. Excess of dry ammonia was condensed in the cell until the salts had all dissolved at the set temperature. The coolants used were dry ice and $CCl_4$. The cell was thermostated in a liquid $NH_3$ bath for -33 °C determinations, in an ice + water bath for 0 °C, and in a water bath for the other temperatures. Ammonia was bled from the solution until salt crystals were formed, and the cell inverted so that the solution filtered through the partition into the larger compartment. After filtration the ammonia in the solution was all removed by condensation into a reservoir in the apparatus and determined quantitatively by absorption in std. HCl sln and back-titrated with std. base. The cell was then opened and the solids removed for analysis. Ammonium ion was determined by a standard Kjeldahl procedure.

SOURCE AND PURITY OF MATERIALS:

Not stated. Ammonia was dried with sodium.

ESTIMATED ERROR: Reproducibility (3 detn) is within ± 2 % of the mean value in most cases.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$ [7601-89-0] | Smith, G.F. |
| (2) Water; $H_2O$; [7732-18-5] | *J. Am. Chem. Soc.* 1925, *47*, 762-9. |
| (3) *Alcohols*: | |
| (A) Methanol (*methyl alcohol*); $CH_4O$; [67-56-1] | |
| (B) Ethanol (*ethyl alcohol*); $C_2H_6O$; [64-17-5] | |
| (C) 1-Butanol (*n-butyl alcohol*): $C_4H_{10}O$; [71-36-3] | |
| (4) Ethyl acetate; $C_4H_8O_2$; [141-78-6] | |

| VARIABLES: | PREPARED BY: |
|---|---|
| One temperature: 298.2 K | C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility of sodium perchlorate in alcohol-ethyl acetate mixtures at 25.0 °C :

| | Volume % | | | | | | |
|---|---|---|---|---|---|---|---|
| Alcohol : | 0 | 5 | 10 | 20 | 30 | 40 | 50 |
| Ethyl acetate : | 100 | 95 | 90 | 80 | 70 | 60 | 50 |
| Alcohol used | mass %($NaClO_4$)[b] | | | | | | |
| methanol | 8.80 | - | 19.39 | 23.37 | 25.85 | 27.81 | 29.38 |
| ethanol (abs.) | " | 13.83 | 16.05 | 18.55 | 19.66 | 20.08 | 20.07 |
| ethanol (93 %) | " | 14.12 | 16.92 | 20.00 | 21.75 | 22.79 | 23.30 |
| 1-butanol | " | - | 12.34 | 13.16 | - | 12.82 | 11.99 |
| 1-butanol[a] | " | 11.97 | 13.00 | 14.32 | 14.75 | 14.71 | 13.98 |
| | mass %($NaClO_4.H_2O$)[c] | | | | | | |
| ethanol (abs.) | 26.32 | - | 29.32 | 31.16 | 32.44 | 33.26 | 33.63 |

(continued next page)

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The experimental technique used was essentially the same as that reported in ref.2 ( see compilation ). Duplicate measurements were made. | Sodium perchlorate monohydrate was prepared by recrystallization of the salt from its aqueous sln below 50 °C, filtered centrifugally and dried at room temp. in contact with excess of the anhydrous salt. The anhydrous salt was prepared in the same manner |

(continued next page)

COMPONENTS:

(1) Sodium perchlorate; $NaClO_4$ [7601-89-0]

(2) Water; $H_2O$; [7732-18-5]

(3) *Alcohols*:

(A) Methanol (*methyl alcohol*); $CH_4O$; [67-56-1]

(B) Ethanol (*ethyl alcohol*); $C_2H_6O$; [64-17-5]

(C) 1-Butanol (*n-butyl alcohol*); $C_4H_{10}O$; [71-36-3]

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

ORIGINAL MEASUREMENTS:

Smith, G.F.

*J. Am. Chem. Soc.* 1925, *47*, 762-9.

EXPERIMENTAL VALUES: (continued)

Solubility of sodium perchlorate in alcohol-ethyl acetate mixtures at 25.0 °C :

| | Volume % | | | | | |
|---|---|---|---|---|---|---|
| Alcohol : | 60 | 70 | 80 | 90 | 95 | 100 |
| Ethyl acetate : | 40 | 30 | 20 | 10 | 5 | 0 |
| Alcohol used | mass %($NaClO_4$)[b] | | | | | |
| methanol | 30.50 | 31.61 | 32.56 | 33.44 | - | 34.33 |
| ethanol (abs.) | 19.67 | 18.78 | 17.53 | 15.82 | 14.80 | 12.83 |
| ethanol (93 %) | 23.39 | 22.73 | 21.92 | 20.96 | 21.31 | 20.60 |
| 1-butanol | 10.54 | - | 6.70 | 4.35 | - | 2.19 |
| 1-butanol[a] | 13.09 | 11.66 | 9.85 | 7.87 | 6.97 | 4.27 |
| | mass %($NaClO_4.H_2O$)[c] | | | | | |
| ethanol (abs.) | 33.61 | 33.20 | 32.56 | 31.56 | 31.10 | 29.80 |

[a] Same quality as that reported in ref. 1.

[b] Solute and solid phase were the anhydrous salt.

[c] Solute and solid phase were the monohydrate.

AUXILIARY INFORMATION

SOURCE AND PURITY OF MATERIALS: (continued)

as that reported in ref.2. Analysis yielded 12.87 % water of crystallization for the hydrate prepared, compared to the theoretical value of 12.82 % for the monohydrate. Ethyl acetate and 1-butanol were prepared as reported in ref.2. Technical methyl alcohol was dried over solid KOH and distilled before used (2). 93 % ethanol was dehydrated with lime to 99.6 % before being made anhydrous by reaction with a slight excess of Ca metal. B.p. of the ethanol thus obtained was 78.29 - 78.31 °C.

ESTIMATED ERROR: Precision in soly not stated. Temperature ± 0.1 °C.

REFERENCES:

1. Smith, G.F. *J. Am. Chem. Soc.* 1923, *45*, 2072.

2. Willard, H.H.; Smith, G.F. *J. Am. Chem. Soc.* 1923, *45*, 286.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Lepeshkov, I.N.; Sal'nikova, L.N. |
| (2) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1973, *120*, 120-5. |
| (3) Hexamethylenetetramine; $C_6H_{12}N_4$ [100-97-0] | |
| (4) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 298 K | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$NH_4ClO_4$-$C_6H_{12}O_4$-water at 25 °C :

| Liquid phase composition | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| mass % | | | mol % [a] | | | molality[a]/ mol $kg^{-1}$ | | | Solid |
| (1) | (2) | (3) | (1) | (2) | (3) | (1) | (2) | (3) | phase[b] |
| - | 22.66 | 22.78 | - | 5.700 | 4.802 | - | 3.535 | 2.978 | A + B |
| - | 6.96 | 43.27 | - | 1.892 | 9.86 | - | 1.190 | 6.202 | B + C |
| - | 6.07 | 46.12 | - | 1.703 | 10.84 | - | 1.081 | 6.881 | C + D |
| 16.35 | - | 43.46 | 4.99 | - | 11.59 | 3.323 | - | 7.714 | D + E |
| 54.47 | - | 13.67 | 19.25 | - | 4.220 | 13.96 | - | 3.061 | E + F |
| 67.11 | - | 5.44 | 25.97 | - | 1.839 | 19.97 | - | 1.414 | F + G |
| 66.82 | 1.62 | - | 23.61 | 0.597 | - | 17.29 | 0.437 | - | G + H |
| 61.92 | 2.70 | - | 20.29 | 0.922 | - | 14.29 | 0.650 | - | A + H |
| 9.25 | 4.09 | 41.94 | 2.61 | 1.204 | 10.35 | 1.689 | 0.778 | 6.690 | B + C + D |
| 14.15 | 3.71 | 41.12 | 4.25 | 1.162 | 10.79 | 2.817 | 0.770 | 7.151 | B + D + E |
| 42.39 | 3.23 | 15.95 | 13.21 | 1.049 | 4.34 | 9.009 | 0.715 | 2.961 | A + B + E |
| 50.87 | 3.58 | 12.79 | 17.64 | 1.294 | 3.87 | 12.682 | 0.930 | 2.785 | A + E + F |
| 61.38 | 2.21 | 5.45 | 22.01 | 0.826 | 1.707 | 16.192 | 0.608 | 1.256 | A + F + G |
| 65.18 | 1.95 | 2.58 | 23.67 | 0.738 | 0.818 | 17.575 | 0.548 | 0.608 | A + G + H |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4$; B = $NH_4ClO_4.C_6H_{12}N_4$; C = $NH_4ClO_4.2C_6H_{12}N_4$;
D = $C_6H_{12}N_4$; E = $NaClO_4.C_6H_{12}N_4$; F = $5(NaClO_4).2(C_6H_{12}N_4).3H_2O$;
G = $NaClO_4.H_2O$; H = $n(NH_4ClO_4).m(NaClO_4)$ .

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Method of "invariant points" used; to the satd slns corresponding to the eutonic and transition points of the ternary systems, relevant salt or hexamethylenetetramine was added until a new solid phase appeared. Periods of equilibration varied from 2 weeks to 1 month. | Not stated. |
| | ESTIMATED ERROR: Not stated. |
| | REFERENCES: |

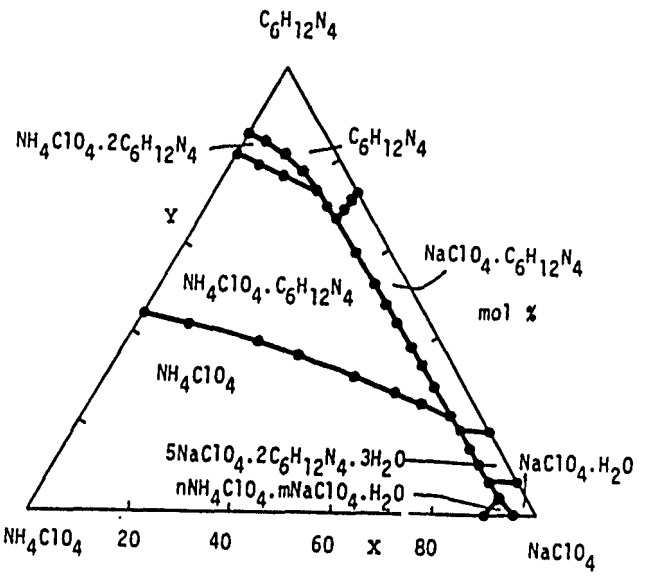
(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Lepeshkov, I.N.; Sal'nikova, L.N. |
| (2) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1973, *120*, 120-5. |
| (3) Hexamethylenetetramine; $C_6H_{12}N_4$ [100-97-0] | |
| (4) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

COMMENTS AND/OR ADDITIONAL DATA

The diagram given below, in which solute mol % values are plotted, shows eight crystallization fields: $NH_4ClO_4$; $NH_4ClO_4.C_6H_{12}N_4$; $NH_4ClO_4.2C_6H_{12}N_4$; $C_6H_{12}N_4$; $NaClO_4.C_6H_{12}N_4$; $5(NaClO_4).2(C_6H_{12}N_4).3H_2O$; $NaClO_4.H_2O$; and solid solutions represented by $n(NH_4ClO_4).m(NaClO_4)$ .




$X=100x_1/(x_1+x_2+x_3)$ $Y=100x_3/(x_1+x_2+x_3)$

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Bestuzheva, M.M.; Kinderov, A.P.; Karnaukhov, A.S. |
| (2) Copper perchlorate; $Cu(ClO_4)_2$; [13770-18-8] | *Sb. Tr. Yarosl. Gos. Ped. Inst.* 1978, *169*, 47-51. |
| (3) Benzamide; $C_7H_7NO$; [55-21-0] | |
| (4) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| Temperature: 298 K. | E.S. Gryzlova |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Cu(ClO_4)_2$-$C_6H_5CONH_2$-water at 25 °C :

| Liquid phase composition | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| mass % | | | mol % [a] | | | molality[a]/mol kg$^{-1}$ | | | Solid |
| (1) | (2) | (3) | (1) | (2) | (3) | (1) | (2) | (3) | phase[b] |
| 17.68 | 45.62 | - | 6.13 | 7.380 | - | 3.935 | 4.736 | - | A + B |
| 66.71 | - | 1.33 | 23.38 | - | 0.471 | 17.05 | - | 0.344 | A + C |
| - | 47.35 | 4.93 | - | 6.286 | 1.42 | - | 3.781 | 0.853 | C + B |
| 62.73 | 5.20 | 9.85 | 27.74 | 1.073 | 4.40 | 23.06 | 0.892 | 3.659 | A + C |
| 49.63 | 15.99 | 0.74 | 17.32 | 2.604 | 0.26 | 12.05 | 1.811 | 0.182 | A + C |
| 35.71 | 27.41 | 0.55 | 12.07 | 4.321 | 0.19 | 8.03 | 2.875 | 0.125 | A + C |
| 27.57 | 36.07 | 0.89 | 9.63 | 5.876 | 0.31 | 6.35 | 3.875 | 0.207 | A + C |
| 18.12 | 43.83 | 0.76 | 6.19 | 6.984 | 0.26 | 3.97 | 4.479 | 0.168 | A + B + C |
| 6.00 | 50.65 | 0.95 | 1.88 | 7.413 | 0.30 | 1.156 | 4.552 | 0.185 | B + C |
| 8.17 | 48.50 | 0.72 | 2.54 | 7.046 | 0.23 | 1.566 | 4.337 | 0.139 | B + C |
| 10.24 | 47.28 | 0.65 | 3.23 | 6.953 | 0.21 | 1.999 | 4.307 | 0.128 | B + C |
| 12.76 | 45.51 | 0.82 | 4.08 | 6.786 | 0.26 | 2.547 | 4.239 | 0.165 | B + C |
| 16.61 | 43.85 | 0.77 | 5.51 | 6.789 | 0.26 | 3.499 | 4.310 | 0.164 | B + C |

[a] Compiler's calculations.

[b] A = $NaClO_4 \cdot H_2O$; B = $Cu(ClO_4)2 \cdot 5H_2O$; C = $C_6H_5CONH_2$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Details of saturation method not given. Benzamide was determined by the Kjeldahl method; $Cu^{2+}$ iodimetrically; $ClO_4^-$ by nitron pptn.; $Na^+$ by difference.

SOURCE AND PURITY OF MATERIALS:

Not stated.

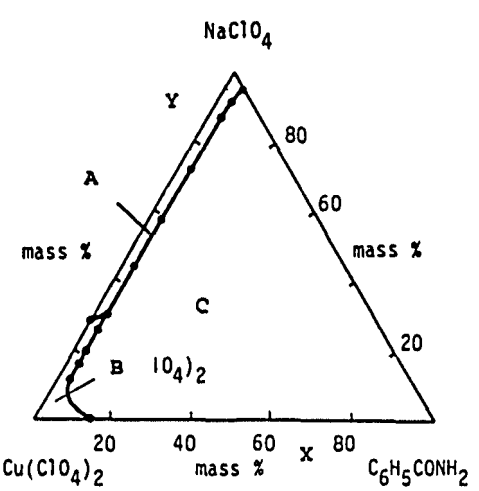
ESTIMATED ERROR:

Not stated.

The eutonic composition (mass %): 18.12 % $NaClO_4$, 43.83 % $Cu(ClO_4)_2$, 0.76 % $C_6H_5CONH_2$ and 37.29 % $H_2O$.

COMMENTS AND/OR ADDITIONAL DATA:

The diagram below shows three crystallization fields: $NaClO_4.H_2O$, $C_6H_5CONH_2$, and $Cu(ClO_4)_2.5H_2O$ .




$X=100x_1/(x_1+x_2+x_3)$ $Y=100x_3/(x_1+x_2+x_3)$

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Nickel perchlorate; $Ni(ClO_4)_2$; [13637-71-3]<br>(3) Carbamide (*urea*); $CH_4N_2O$; [57-13-6]<br>(4) Water; $H_2O$; [7732-18-5] | Tarakanov, V.F.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1976, *154*, 35-7. |
| **VARIABLES:**<br>Temperature: 298 K<br>Composition | **PREPARED BY:**<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$Ni(ClO_4)_2$-$CO(NH_2)_2$-water at 25 °C :

| | Liquid phase composition | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | mass % | | | mol % [a] | | | molality[a]/mol kg$^{-1}$ | | | Solid |
| | (1) | (2) | (3) | (1) | (2) | (3) | (1) | (2) | (3) | phase[b] |
| I | - | 51.12 | 7.58 | - | 7.582 | 4.82 | - | 4.805 | 3.06 | E + F |
| II | 22.66 | 34.18 | 6.55 | 7.526 | 5.396 | 4.44 | 5.055 | 3.624 | 2.98 | D + E + F |
| III | 62.41 | 2.37 | 6.20 | 22.83 | 0.412 | 4.62 | 17.56 | 0.317 | 3.56 | D + F + G |
| IV | 31.09 | 29.42 | - | 9.92 | 4.461 | - | 6.43 | 2.892 | - | D + E |
| V | - | 33.99 | 29.13 | - | 4.953 | 18.21 | - | 3.578 | 13.15 | F + G |
| VI | - | 20.30 | 55.63 | - | 3.366 | 39.57 | - | 3.274 | 38.48 | A + G |
| VII | 33.57 | 1.84 | 49.47 | 14.10 | 0.367 | 42.37 | 18.13 | 0.472 | 54.48 | A + C + G |
| VIII | 46.43 | 0.86 | 31.75 | 18.28 | 0.161 | 25.48 | 18.09 | 0.159 | 25.22 | B + C + G |
| IX | 59.42 | 0.99 | 20.18 | 25.51 | 0.202 | 17.66 | 25.00 | 0.198 | 17.31 | B + D + G |
| X | 34.85 | - | 50.06 | 14.55 | - | 42.62 | 18.86 | - | 55.24 | A + B |
| XI | 46.81 | - | 32.88 | 18.58 | - | 26.61 | 18.82 | - | 26.96 | B + C |
| XII | 59.69 | - | 19.71 | 24.88 | - | 16.75 | 23.67 | - | 15.93 | C + D |

[a] Compiler's calculations.

[b] A = $CO(NH_2)_2$; B = $NaClO_4.2CO(NH_2)_2$; C = $2NaClO_4.3CO(NH_2)_2$; D = $NaClO_4.H_2O$; E = $Ni(ClO_4)_2.6H_2O$; F = $Ni(ClO_4)_2.4CO(NH_2)_2.2H_2O$; G = $Ni(ClO_4)_2.6CO(NH_2)_2$ .

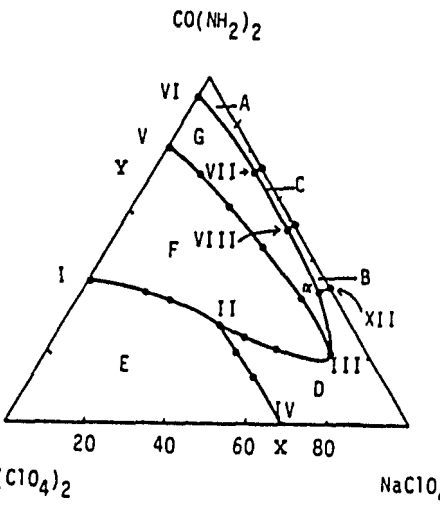
AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
No details, but reference given (ref. 1).

SOURCE AND PURITY OF MATERIALS:
Not stated.

ESTIMATED ERROR:
Not stated.

REFERENCES:
1. Karnaukhov, A.S.; Goryunov, Yu.A.; Tarakanov, V.F. *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 131.




$X=100x_1/(x_1+x_2+x_3)$ $Y=100x_3/(x_1+x_2+x_3)$

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(2) Sodium Chlorate; $NaClO_3$; [7775-09-9]<br>(3) Sodium chloride; NaCl; [7647-14-5]<br>(4) Water; $H_2O$; [7732-18-5] | 1. Il'in, K.G.; Loseva,G.K.; Semchenko, D.P. *Tr. Novoch. Politekhn. Inst.* 1969, *197*, 37-44.<br>2. Loseva, K.G.; Semchenko, D.P.; Il'in, K.G. *Tr. Novoch. Politekhn. Inst.* 1969, *197*, 45-49.<br>3. Loseva, G.K. *Tr. Novoch. Politekhn. Inst.* 1969, *197*, 78-81. |
| VARIABLES:<br>Temperature: 313.2 K and 333.2 K.<br>Composition. | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system $NaClO_4$-$NaClO_3$-NaCl-water at 40.0 °C :

| Liquid phase composition | | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| mass % | | | mol %[a] | | | molality[a]/mol kg$^{-1}$ | | | |
| (3) | (2) | (1) | (3) | (2) | (1) | (3) | (2) | (1) | |
| 11.80 | 34.70 | 0.00 | 5.77 | 9.32 | 0.00 | 3.77 | 6.09 | 0.00 | A + B |
| 10.60 | 33.00 | 4.60 | 5.33 | 9.11 | 1.10 | 3.50 | 5.99 | 0.73 | A + B |
| 9.90 | 31.80 | 7.20 | 5.04 | 8.88 | 1.75 | 3.31 | 5.85 | 1.15 | A + B |
| 9.20 | 30.80 | 10.90 | 4.83 | 8.87 | 2.73 | 3.21 | 5.89 | 1.81 | A + B |
| 8.80 | 29.80 | 12.40 | 4.63 | 8.61 | 3.11 | 3.07 | 5.71 | 2.07 | A + B |
| 8.00 | 29.70 | 13.90 | 4.26 | 8.68 | 3.53 | 2.83 | 5.77 | 2.35 | A + B |
| 7.00 | 27.40 | 18.90 | 3.83 | 8.24 | 4.94 | 2.56 | 5.51 | 3.31 | A + B |
| 6.20 | 26.30 | 22.30 | 3.48 | 8.12 | 5.98 | 2.35 | 5.47 | 4.03 | A + B |
| 5.90 | 20.20 | 23.70 | 3.09 | 5.80 | 5.92 | 2.01 | 3.78 | 3.86 | A + B |
| 5.70 | 25.00 | 27.60 | 3.40 | 8.18 | 7.85 | 2.34 | 5.63 | 5.41 | A + B |
| 4.80 | 23.50 | 30.80 | 2.91 | 7.82 | 8.91 | 2.01 | 5.40 | 6.15 | A + B |
| 3.90 | 18.80 | 32.70 | 2.23 | 5.91 | 8.94 | 1.50 | 3.96 | 5.99 | A + B |
| 3.10 | 18.00 | 40.70 | 1.98 | 6.32 | 12.43 | 1.39 | 4.43 | 8.70 | A + B |
| 2.50 | 17.10 | 43.90 | 1.65 | 6.21 | 13.85 | 1.17 | 4.40 | 9.82 | A + B |
| 2.10 | 15.30 | 48.60 | 1.46 | 5.83 | 16.11 | 1.06 | 4.23 | 11.67 | A + B |
| 1.70 | 14.20 | 51.20 | 1.21 | 5.54 | 17.37 | 0.88 | 4.05 | 12.71 | A + B |
| 1.60 | 13.70 | 51.20 | 1.12 | 5.29 | 17.18 | 0.82 | 3.84 | 12.48 | A + B |
| 1.70 | 13.20 | 51.40 | 1.19 | 5.08 | 17.18 | 0.86 | 3.68 | 12.46 | A + B |
| 1.50 | 14.10 | 53.30 | 1.11 | 5.71 | 18.77 | 0.83 | 4.26 | 14.00 | A + B |
| 1.30 | 12.80 | 55.40 | 0.97 | 5.26 | 19.78 | 0.73 | 3.94 | 14.83 | A + B |
| 1.20 | 12.20 | 55.50 | 0.89 | 4.95 | 19.58 | 0.66 | 3.69 | 14.57 | A + B |
| 1.00 | 11.70 | 58.40 | 0.77 | 4.98 | 21.60 | 0.59 | 3.80 | 16.50 | A + B + C |
| 2.00 | 0.00 | 63.70 | 1.39 | 0.00 | 21.16 | 1.00 | 0.00 | 15.17 | A + C |
| 2.00 | 3.10 | 63.40 | 1.47 | 1.25 | 22.23 | 1.09 | 0.92 | 16.44 | A + C |
| 1.40 | 3.50 | 62.40 | 1.01 | 1.38 | 21.40 | 0.73 | 1.01 | 15.59 | A + C |
| 1.30 | 4.70 | 61.60 | 0.94 | 1.86 | 21.25 | 0.69 | 1.36 | 15.53 | A + C |
| 1.20 | 7.30 | 61.00 | 0.90 | 3.01 | 21.85 | 0.67 | 2.25 | 16.33 | A + C |
| 1.10 | 11.40 | 58.80 | 0.86 | 4.87 | 21.84 | 0.66 | 3.73 | 16.73 | A + B + C |
| 0.00 | 11.40 | 59.80 | 0.00 | 4.88 | 22.26 | 0.00 | 3.72 | 16.96 | B + C |
| 0.90 | 11.10 | 59.20 | 0.70 | 4.74 | 21.96 | 0.53 | 3.62 | 16.79 | B + C |
| 1.00 | 11.50 | 59.00 | 0.78 | 4.94 | 22.01 | 0.60 | 3.79 | 16.91 | A + B + C |
| 1.00 | 11.20 | 58.30 | 0.77 | 4.71 | 21.29 | 0.58 | 3.57 | 16.14 | A + B + C |

[a] Compiler's calculations.

[b] A = NaCl; B = $NaClO_3$; C = $NaClO_4.H_2O$ .

(continued next page)

COMPONENTS:

(1) Sodium perchlorate; $NaClO_4$; [7601-89-0]

(2) Sodium Chlorate; $NaClO_3$; [7775-09-9]

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

(4) Water; H2O; [7732-18-5]

ORIGINAL MEASUREMENTS:

1. Il'in, K.G.; Loseva,G.K.; Semchenko, D.P.
   *Tr. Novoch. Politekhn. Inst.* 1969, *197*, 37-44.
2. Loseva, K.G.; Semchenko, D.P.; Il'in, K.G.
   *Tr. Novoch. Politekhn. Inst.* 1969, *197*, 45-49.
3. Loseva, G.K.
   *Tr. Novoch. Politekhn. Inst.* 1969, *197*, 78-81.

EXPERIMENTAL VALUES: (continued)

Solubility system $NaClO_4$-$NaClO_3$-NaCl-water at 60.0 °C :

| Liquid phase composition | | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| mass % | | | mol %[a] | | | molality[a]/mol kg$^{-1}$ | | | |
| (3) | (2) | (1) | (3) | (2) | (1) | (3) | (2) | (1) | |
| 6.30 | 45.80 | 4.80 | 3.63 | 14.49 | 1.32 | 2.50 | 9.98 | 0.91 | A + B |
| 5.00 | 41.00 | 12.90 | 2.99 | 13.48 | 3.69 | 2.08 | 9.37 | 2.56 | A + B |
| 4.10 | 36.40 | 21.40 | 2.60 | 12.66 | 6.47 | 1.84 | 8.98 | 4.59 | A + B |
| 3.20 | 32.90 | 27.70 | 2.11 | 11.89 | 8.70 | 1.51 | 8.54 | 6.25 | A + B |
| 2.80 | 30.40 | 32.30 | 1.91 | 11.37 | 10.50 | 1.39 | 8.28 | 7.65 | A + B |
| 2.30 | 27.50 | 37.60 | 1.63 | 10.70 | 12.72 | 1.21 | 7.93 | 9.42 | A + B |
| 2.00 | 25.80 | 40.10 | 1.43 | 10.16 | 13.73 | 1.07 | 7.55 | 10.20 | A + B |
| 1.80 | 24.00 | 42.60 | 1.31 | 9.56 | 14.75 | 0.97 | 7.14 | 11.01 | A + B |
| 1.40 | 21.60 | 48.50 | 1.09 | 9.20 | 17.96 | 0.84 | 7.12 | 13.90 | A + B |
| 1.20 | 19.40 | 52.20 | 0.96 | 8.52 | 19.93 | 0.75 | 6.70 | 15.67 | A + B |
| 1.10 | 18.40 | 53.70 | 0.89 | 8.16 | 20.71 | 0.70 | 6.45 | 16.36 | A + B |
| 1.00 | 18.30 | 55.10 | 0.83 | 8.35 | 21.84 | 0.67 | 6.72 | 17.58 | A + B |
| 0.90 | 17.00 | 57.60 | 0.77 | 7.96 | 23.46 | 0.63 | 6.52 | 19.20 | A + B |
| 0.80 | 16.00 | 59.60 | 0.70 | 7.67 | 24.83 | 0.58 | 6.37 | 20.63 | A + B |
| 0.70 | 15.30 | 61.50 | 0.63 | 7.54 | 26.34 | 0.53 | 6.39 | 22.32 | A + B + D |
| 0.80 | 3.80 | 69.80 | 0.67 | 1.75 | 27.94 | 0.53 | 1.39 | 22.27 | A + D |
| 0.70 | 13.10 | 65.20 | 0.65 | 6.71 | 29.05 | 0.57 | 5.86 | 25.36 | A + D |

[a] Compiler's calculations.

[b] A = NaCl; B = $NaClO_3$; C = $NaClO_4.H_2O$; D = $NaClO_4$ .

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturation details not given. Satd. slns. reached equilibrium in 24 h at 40 °C. $ClO_3^-$ was determined by titration with $KMnO_4$ std. sln. in the presence of Mohr's salt; perchlorate determined chromatographically (no details). The composition of the solid phase was determined by Schreinemakers' method and optical crystallography and X-ray powder analysis were employed.

SOURCE AND PURITY OF MATERIALS:

Chemically pure and reagent grade salts were recrystallized twice; 99.6 - 99.9 % pure.

ESTIMATED ERROR:

± 0.1 °C in temperature.

COMMENTS: The eutonic composition at 40 °C (mass %): 1.05% NaCl, 11.68% $NaClO_3$ and 58.40% $NaClO_4$.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Potassium chloride; KCl; [7447-40-7]<br>(4) Potassium perchlorate; $KClO_4$; [7778-74-7]<br>(5) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Troitskii, E.N.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, *59*, 8-21. |
| **VARIABLES:**<br>Temperature: 363 K.<br>Composition. | **PREPARED BY:**<br>I.S. Bodnya |

EXPERIMENTAL VALUES:

Solubility system $Na^+$, $K^+$ || $ClO_4^-$, $Cl^-$ - water at 90 °C :

| Liquid phase composition | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| mass % | | | | molality[a] / mol $kg^{-1}$ | | | | |
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 0.84 | 72.75 | - | 2.71 | 0.606 | 25.07 | - | 0.825 | A + B + C |
| 0.83 | 72.71 | - | 2.70 | 0.598 | 24.99 | - | 0.820 | A + B + C |
| 0.79 | 72.70 | - | 2.69 | 0.567 | 24.93 | - | 0.815 | A + B + C |
| 0.84 | 72.75 | - | 1.56 | 0.578 | 23.91 | - | 0.483 | A + B |
| 6.13 | 47.83 | - | 2.32 | 2.399 | 8.94 | - | 0.383 | A + C |
| 10.20 | 36.38 | - | 2.55 | 3.431 | 5.841 | - | 0.362 | A + C |
| 12.52 | 30.41 | - | 2.74 | 3.943 | 4.571 | - | 0.364 | A + C |
| 17.04 | 19.28 | - | 3.01 | 4.806 | 2.595 | - | 0.358 | A + C |
| 19.73 | 14.91 | - | 3.53 | 5.460 | 1.969 | - | 0.412 | A + C |
| 23.22 | 5.70 | - | 4.48 | 5.966 | 0.699 | - | 0.485 | A + C |
| 23.32 | - | 5.55 | 4.47 | 5.986 | - | 1.117 | 0.484 | A + C |
| 22.02 | - | 7.82 | 3.91 | 5.687 | - | 1.583 | 0.426 | A + C |
| 20.96 | - | 10.55 | 1.96 | 5.391 | - | 2.127 | 0.213 | A + C |
| 16.89 | - | 13.47 | 0.84 | 4.201 | - | 2.626 | 0.088 | A + C |
| 15.71 | - | 19.96 | 1.19 | 4.257 | - | 4.240 | 0.136 | A + C + D |
| 15.97 | - | 20.05 | 1.21 | 4.353 | - | 4.284 | 0.139 | A + C + D |
| 15.82 | - | 20.04 | 1.20 | 4.301 | - | 4.271 | 0.138 | A + C + D |
| 12.23 | - | 23.27 | 1.53 | 3.323 | - | 4.957 | 0.175 | C + D |
| 8.42 | - | 26.39 | 1.67 | 2.268 | - | 5.573 | 0.190 | C + D |
| 4.57 | - | 30.15 | 1.72 | 1.230 | - | 6.362 | 0.195 | C + D |
| - | 73.83 | - | 2.36 | - | 25.33 | - | 0.715 | C + B |
| 0.88 | 67.17 | - | - | 0.471 | 17.17 | - | - | A + B |
| 16.90 | - | 20.80 | - | 4.642 | - | 4.478 | - | A + E |
| - | - | 34.11 | 2.42 | - | - | 7.208 | 0.275 | C + E |

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Method of invariant points used; to a satd eutonic sln of a ternary system a third salt was added until a new solid phase appeared. $K^+$ was determined by the tetraphenylborate gravimetric method; $Na^+$ gravimetric- | Not stated. |
| | ESTIMATED ERROR:<br>Temperature: ± 0.05 °C. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5] | Karnaukhov, A.S.; Troitskii, E.N. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, *59*, 8-21. |
| (3) Potassium chloride; KCl; [7447-40-7] | |
| (4) Potassium perchlorate; $KClO_4$; [7778-74-7] | |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

| mol % [a] | | | | ion mol % [a] | | | | Solid phase [b] |
|---|---|---|---|---|---|---|---|---|
| | | | | Cation | | Anion | | |
| (1) | (2) | (3) | (4) | $Na^+$ | $K^+$ | $Cl^-$ | $ClO_4^-$ | |
| 0.74 | 30.57 | - | 1.006 | 96.89 | 3.11 | 2.29 | 97.71 | A + B + C |
| 0.73 | 30.51 | - | 1.001 | 96.89 | 3.11 | 2.26 | 97.74 | A + B + C |
| 0.69 | 30.47 | - | 0.996 | 96.90 | 3.10 | 2.16 | 97.84 | A + B + C |
| 0.72 | 29.72 | - | 0.563 | 98.18 | 1.82 | 2.32 | 97.68 | A + B |
| 3.57 | 13.29 | - | 0.570 | 96.73 | 3.27 | 20.48 | 79.52 | A + C |
| 5.27 | 8.97 | - | 0.555 | 96.24 | 3.76 | 35.61 | 64.39 | A + C |
| 6.12 | 7.10 | - | 0.565 | 95.90 | 4.10 | 44.41 | 55.59 | A + C |
| 7.60 | 4.10 | - | 0.566 | 95.39 | 4.61 | 61.94 | 38.06 | A + C |
| 8.62 | 3.11 | - | 0.650 | 94.75 | 5.25 | 69.63 | 30.37 | A + C |
| 9.52 | 1.12 | - | 0.775 | 93.21 | 6.79 | 83.43 | 16.57 | A + C |
| 9.49 | - | 1.77 | 0.767 | 78.90 | 21.10 | 93.62 | 6.38 | A + C |
| 9.00 | - | 2.50 | 0.674 | 73.89 | 26.11 | 94.47 | 5.53 | A + C |
| 8.52 | - | 3.36 | 0.336 | 69.73 | 30.27 | 97.25 | 2.75 | A + C |
| 6.73 | - | 4.21 | 0.141 | 60.75 | 39.25 | 98.73 | 1.27 | A + C |
| 6.64 | - | 6.61 | 0.212 | 49.31 | 50.69 | 98.42 | 1.58 | A + C + D |
| 6.77 | - | 6.66 | 0.216 | 49.60 | 50.40 | 98.41 | 1.59 | A + C + D |
| 6.70 | - | 6.65 | 0.214 | 49.38 | 50.62 | 98.42 | 1.58 | A + C + D |
| 5.20 | - | 7.75 | 0.274 | 39.30 | 60.70 | 97.93 | 2.07 | C + D |
| 3.57 | - | 8.77 | 0.299 | 28.24 | 71.76 | 97.64 | 2.36 | C + D |
| 1.94 | - | 10.1 | 0.309 | 15.80 | 84.20 | 97.49 | 2.51 | C + D |
| - | 31.06 | - | 0.877 | 97.25 | 2.75 | - | 100.00 | B + C |
| 0.64 | 23.47 | - | - | 100.00 | - | 2.67 | 97.33 | A + B |
| 7.18 | - | 6.93 | - | 50.90 | 49.10 | 100.00 | - | A + E |
| - | - | 11.44 | 0.437 | - | 100.00 | 96.32 | 3.68 | C + E |

[a] Compiler's calculations.

[b] A = NaCl; B = $NaClO_4$; C = $KClO_4$; D = m(KCl).n($KClO_4$)
E = KCl

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | REFERENCES: |
|---|---|
| ally as sodium uranyl acetate; perclorate gravimetrically with nitron and chloride mercurimetrically. Optical crystallographic and differential thermal analyses of the solid phases were carried out. | |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chromate; $Na_2CrO_4$; [7775-11-3]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Potassium chromate; $K_2CrO_4$; [7789-00-6]<br>(4) Potassium perchlorate; $KClO_4$; [7778-74-7]<br>(5) Water; $H_2O$; [7732-18-5] | Druzhinina, G.V.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* <u>1966</u>, *59*, 73-82. |
| VARIABLES:<br>Temperature: 323 K.<br>Composition. | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system $2Na^+$, $2K^+$ || $2ClO_4^-$, $CrO_4^{2-}$ - water at 50 °C :

| Point | Liquid phase composition, mass % | | | | Liquid phase composition, molality[a] / mol kg$^{-1}$ | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| I | - | - | 40.95 | 0.55 | - | - | 3.605 | 0.068 | A + B |
| | 2.59 | - | 37.66 | 0.46 | 0.270 | - | 3.271 | 0.056 | A + B |
| II | 6.04 | - | 37.32 | - | 0.658 | - | 3.393 | - | B + C |
| III | 5.78 | - | 35.28 | 0.49 | 0.611 | - | 3.108 | 0.061 | A + B + C |
| | 7.64 | - | 34.63 | 0.44 | 0.823 | - | 3.113 | 0.055 | A + C |
| | 15.56 | - | 28.35 | 0.58 | 1.731 | - | 2.630 | 0.075 | A + C |
| | 20.86 | - | 20.36 | 0.49 | 2.209 | - | 1.799 | 0.061 | A + C |
| | 27.62 | - | 13.40 | 0.52 | 2.917 | - | 1.180 | 0.064 | A + C |
| | 37.73 | - | 8.52 | 0.53 | 4.377 | - | 0.824 | 0.072 | A + C |
| IV | 44.35 | - | 5.87 | - | 5.500 | - | 0.607 | - | C + D |
| V | 49.92 | - | 2.61 | 1.06 | 6.641 | - | 0.290 | 0.165 | A + C + D |

(continued next page)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

This solubility study involved isothermal recrystallization and crystallization from supersaturated solutions. Details of saturation were not given. Sodium was determined gravimetrically as sodium zinc uranyl acetate and by flame photometry; potassium as potassium tetraphenylborate; chromate iodimetrically and perchlorate by difference.

SOURCE AND PURITY OF MATERIALS:

The chemically "pure" salts were purified by recrystallization. The hydrates of sodium salts were dehydrated (no details given).

ESTIMATED ERROR:

Not stated.

REFERENCES:

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chromate; $Na_2CrO_4$; [7775-11-3] | Druzhinina, G.V. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, *59*, 73-82. |
| (3) Potassium chromate; $K_2CrO_4$; [7789-00-6] | |
| (4) Potassium perchlorate; $KClO_4$ [7778-74-7] | |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system $2Na^+$, $2K^+$ || $2ClO_4^-$, $CrO_4^{2-}$ - water at 50 °C :

| Point | Liquid phase composition, mass % (1) | (2) | (3) | (4) | molality[a] / mol kg$^{-1}$ (1) | (2) | (3) | (4) | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| | 48.06 | 2.61 | - | 0.49 | 6.075 | 0.436 | - | 0.072 | A + D |
| | 48.15 | 5.14 | - | 0.33 | 6.409 | 0.905 | - | 0.051 | A + D |
| | 42.59 | 13.42 | - | 0.33 | 6.023 | 2.510 | - | 0.055 | A + D |
| | 39.58 | 18.21 | - | 0.25 | 5.824 | 3.544 | - | 0.043 | A + D |
| | 29.42 | 31.06 | - | 0.36 | 4.638 | 6.478 | - | 0.066 | A + D |
| | 24.82 | 39.03 | - | 0.29 | 4.273 | 8.889 | - | 0.058 | A + D |
| VI | 19.69 | 47.82 | - | 0.27 | 3.773 | 12.12 | - | 0.060 | A + D + E |
| VII | 19.47 | 47.02 | - | - | 3.587 | 11.46 | - | - | D + E |
| | 15.58 | 51.64 | - | 0.31 | 2.962 | 12.99 | - | 0.069 | A + E |
| | 10.93 | 58.54 | - | 0.32 | 2.234 | 15.83 | - | 0.076 | A + E |
| | 7.87 | 64.06 | - | 0.40 | 1.756 | 18.91 | - | 0.104 | A + E |
| VIII | 6.70 | 65.60 | - | 0.49 | 1.520 | 19.69 | - | 0.130 | A + E + F |
| IX | 6.41 | 67.19 | - | - | 1.499 | 20.79 | - | - | E + F |
| | 3.28 | 69.14 | - | 0.51 | 0.748 | 20.86 | - | 0.136 | A + F |
| X | - | 72.65 | - | 0.61 | - | 22.19 | - | 0.165 | A + F |

| Point | Liquid phase composition, mol % [a] (1) | (2) | (3) | (4) | ion mol % [a] Cation $2Na^+$ | $2K^+$ | Anion $CrO_4^{2-}$ | $2ClO_4^-$ | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| I | - | - | 6.09 | 0.115 | - | 100.00 | 99.07 | 0.93 | A + B |
| | 0.46 | - | 5.53 | 0.095 | 7.56 | 92.44 | 99.22 | 0.78 | A + B |
| II | 1.11 | - | 5.70 | - | 16.25 | 83.75 | 100.00 | - | B + C |
| III | 1.03 | - | 5.24 | 0.102 | 16.29 | 83.71 | 99.19 | 0.81 | A + B + C |
| | 1.38 | - | 5.23 | 0.093 | 20.77 | 79.23 | 99.30 | 0.70 | A + C |
| | 2.89 | - | 4.39 | 0.126 | 39.35 | 60.65 | 99.14 | 0.86 | A + C |
| | 3.71 | - | 3.02 | 0.102 | 54.71 | 45.29 | 99.25 | 0.75 | A + C |
| | 4.89 | - | 1.98 | 0.108 | 70.64 | 29.36 | 99.22 | 0.78 | A + C |
| | 7.20 | - | 1.36 | 0.118 | 83.57 | 16.43 | 99.31 | 0.69 | A + C |
| IV | 8.93 | - | 0.99 | - | 90.06 | 9.94 | 100.00 | - | C + D |
| V | 10.61 | - | 0.46 | 0.263 | 94.70 | 5.30 | 98.82 | 1.18 | A + C + D |
| | 9.78 | 0.70 | - | 0.117 | 99.43 | 0.57 | 95.98 | 4.02 | A + D |
| | 10.19 | 1.44 | - | 0.082 | 99.63 | 0.37 | 93.06 | 6.94 | A + D |
| | 9.40 | 3.92 | - | 0.085 | 99.63 | 0.37 | 82.44 | 17.56 | A + D |
| | 8.97 | 5.46 | - | 0.066 | 99.72 | 0.28 | 76.45 | 23.55 | A + D |
| | 6.95 | 9.71 | - | 0.099 | 99.58 | 0.42 | 58.64 | 41.36 | A + D |
| | 6.22 | 12.93 | - | 0.085 | 99.67 | 0.33 | 48.85 | 51.15 | A + D |

(continued next page)

COMPONENTS:

(1) Sodium chromate; $Na_2CrO_4$; [7775-11-3]

(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]

(3) Potassium chromate; $K_2CrO_4$; [7789-00-6]

(4) Potassium perchlorate; $KClO_4$ [7778-74-7]

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

ORIGINAL MEASUREMENTS:

Druzhinina, G.V.

*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1966, *59*, 73-82.

EXPERIMENTAL VALUES: (continued)

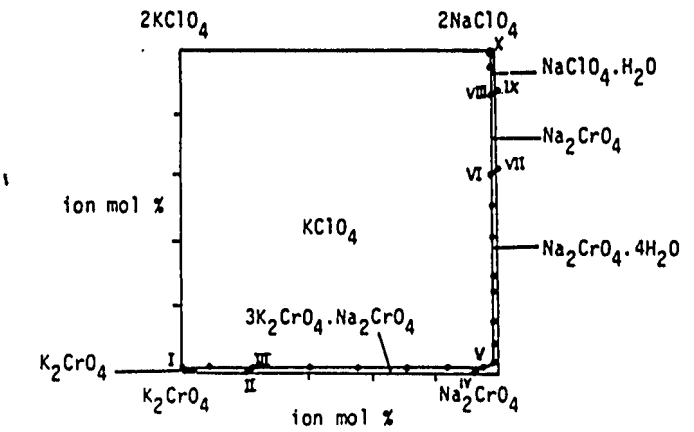
| | mol % [a] | | | | ion mol % [a] Cation | | ion mol % [a] Anion | | Solid phase [b] |
|---|---|---|---|---|---|---|---|---|---|
| Point | (1) | (2) | (3) | (4) | $2Na^+$ | $2K^+$ | $CrO_4^{2-}$ | $2ClO_4^-$ | |
| VI | 5.28 | 16.96 | - | 0.085 | 99.69 | 0.31 | 38.25 | 61.75 | A + D + E |
| VII | 5.08 | 16.24 | - | - | 100.00 | - | 38.50 | 61.50 | D + E |
| | 4.14 | 18.16 | - | 0.096 | 99.64 | 0.36 | 31.21 | 68.79 | A + E |
| | 3.03 | 21.49 | - | 0.104 | 99.62 | 0.38 | 21.93 | 78.07 | A + E |
| | 2.30 | 24.79 | - | 0.137 | 99.54 | 0.46 | 15.59 | 84.41 | A + E |
| VIII | 1.98 | 25.62 | - | 0.169 | 99.43 | 0.57 | 13.30 | 86.70 | A + E + F |
| IX | 1.93 | 26.72 | - | - | 100.00 | - | 12.61 | 87.39 | E + F |
| | 0.97 | 27.00 | - | 0.176 | 99.40 | 0.60 | 6.65 | 93.35 | A + F |
| X | - | 28.50 | - | 0.211 | 99.26 | 0.74 | - | 100.00 | A + F |

[a] Compiler's calculations

[b] A = $KClO_4$; B = $K_2CrO_4$; C = $Na_2CrO_4.3K_2CrO_4$; D = $Na_2CrO_4.4H_2O$; E = $Na_2CrO_4$; F = $NaClO_4.H_2O$ .

COMMENTS / ADDITIONAL DATA

The Janecke diagram given below shows six crystallization fields : $KClO_4$; $NaClO_4.H_2O$; $Na_2CrO_4$; $Na_2CrO_4.4H_2O$, $Na_2CrO_4.3K_2CrO_4$ and $K_2CrO_4$. Points I, II, VII, IX and X correspond to the average compositions of the liquid phases of the isothermal double saturation points of the ternary systems, e.g. point II in the system $K_2CrO_4$-$Na_2CrO_4$-$H_2O$ and point VII in the system $NaClO_4$-$Na_2CrO_4$-$H_2O$. Compositions of triple saturation points III, V, VI, VIII are given in the preceding tables. Potassium perchlorate was nearly completely salted out from solution by all the other salts of the system. The equilibrium of the exchange reaction $2NaClO_4 + K_2CrO_4 = 2KClO_4 + Na_2CrO_4$ was shifted to the right.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Cesium chloride; CsCl; [7647-17-8]<br>(4) Cesium perchlorate; $CsClO_4$; [13454-84-7]<br>(5) Water; $H_2O$; [7732-18-5] | Shklovskaya, R.M.; Arkhipov, S.M.; Kuzina, V.A.<br><br>*Zh. Neorg. Khim.* 1974, *19*, 3128-33; **Russ. J. Inorg. Chem.* Engl. Transl.) 1974, *19*, 1711-4. |
| VARIABLES:<br>Temperature: 298.2 K and 348.2 K.<br>Composition. | PREPARED BY:<br>W.L. Ng |

EXPERIMENTAL VALUES:

Solubility system $Na^+,Cs^+ \,||\, Cl^-,ClO_4^-$, water at 25.0 °C :

| Liquid phase composition, mass % | | | | Liquid phase composition, mol %[a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| - | - | 64.794 | 0.494 | - | - | 16.633 | 0.0919 | A + B |
| 0.316 | - | 62.528 | 0.321 | 0.2232 | - | 15.329 | 0.0570 | B + C |
| 1.373 | - | 60.770 | 0.267 | 0.9503 | - | 14.601 | 0.0465 | B + C |
| 3.838 | - | 56.644 | 0.260 | 2.5430 | - | 13.029 | 0.0433 | B + C |
| 4.042 | - | 56.311 | 0.258 | 2.6691 | - | 12.908 | 0.0429 | B + C |
| 5.823 | - | 51.593 | 0.274 | 3.6155 | - | 11.120 | 0.0428 | B + C |
| 8.160 | - | 46.492 | 0.342 | 4.7891 | - | 9.472 | 0.0505 | B + C |
| 10.753 | - | 44.893 | - | 6.3170 | - | 9.155 | - | B + C |
| 10.034 | - | 41.502 | 0.220 | 5.5436 | - | 7.959 | 0.0306 | C + D |
| 12.652 | - | 34.262 | 0.272 | 6.4569 | - | 6.070 | 0.0349 | B + C + D |

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| No details of saturation method were given. A thermostat maintained the temperature constant to within ± 0.1°C. Equilibrium was attained after 2-3 weeks at 25°C and 7-15 days at 75°C. $Cs^+$ was analysed by precipitation with sodium tetraphenylborate (ref. 1); $Cl^-$ by argentometric method (ref. 2); $ClO_4^-$ by precipitation with nitron ( ref.3 ) for concentrations higher than 1.5%, otherwise by photometry involving the use of | The salts were at least 99.5 % pure.<br><br>ESTIMATED ERROR:<br>Temperature: ± 0.1°C.<br>Solubility : not stated. |

REFERENCES:

1. Yanson, E.Yu.; Ievin'sh, A.F. *Uspekhi Khim.* 1953, *28*, 980.
2. Hillebrand, W. F.; Lundell G.E.F.; Bright, H.A. ; Hoffman, J.I. *Applied Inorganic Analysis*, Goskhimizdat, Moscow, 1964, 745

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5] | Shklovskaya, R.M.; Arkhipov, S.M.; Kuzina, V.A. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Zh. Neorg. Khim.* 1974, *19*, 3128-33; **Russ. J. Inorg. Chem.* Engl. Transl.) 1974, *19*, 1711-4. |
| (3) Cesium chloride; CsCl; [7647-17-8] | |
| (4) Cesium perchlorate; $CsClO_4$; [13454-84-7] | |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system $Na^+,Cs^+ \,||\, Cl^-,ClO_4^-$, water at 25.0 °C : (continued)

| Liquid phase composition | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| molality[a] / mol $kg^{-1}$ | | | | ion mol %[a] | | | | |
| | | | | cation | | anion | | |
| (1) | (2) | (3) | (4) | $Na^+$ | $Cs^+$ | $Cl^-$ | $ClO_4^-$ | |
| - | - | 11.087 | 0.0612 | - | 100.00 | 99.45 | 0.55 | A + B |
| 0.147 | - | 10.083 | 0.0375 | 1.43 | 98.57 | 99.63 | 0.37 | B + C |
| 0.625 | - | 9.602 | 0.0306 | 6.09 | 93.91 | 99.70 | 0.30 | B + C |
| 1.673 | - | 8.570 | 0.0285 | 16.29 | 83.71 | 99.72 | 0.28 | B + C |
| 1.756 | - | 8.492 | 0.0282 | 17.09 | 82.91 | 99.73 | 0.27 | B + C |
| 2.355 | - | 7.243 | 0.0279 | 24.46 | 75.54 | 99.71 | 0.29 | B + C |
| 3.102 | - | 6.136 | 0.0327 | 33.46 | 66.54 | 99.65 | 0.35 | B + C |
| 4.148 | - | 6.012 | - | 40.83 | 59.17 | 100.00 | - | B + C |
| 3.559 | - | 5.110 | 0.0196 | 40.96 | 59.04 | 99.77 | 0.23 | C + D |
| 4.099 | - | 3.853 | 0.0222 | 51.40 | 48.60 | 99.72 | 0.28 | B + C + D |
| 4.845 | - | 1.990 | 0.0203 | 70.67 | 29.33 | 99.70 | 0.30 | B + D |
| 5.042 | - | 1.495 | 0.0191 | 76.91 | 23.09 | 99.71 | 0.29 | B + D |
| 5.333 | - | 0.781 | 0.0238 | 86.89 | 13.11 | 99.61 | 0.39 | B + D |
| 5.553 | - | 0.307 | 0.0424 | 94.08 | 5.92 | 99.28 | 0.72 | B + D |
| 5.672 | - | - | 0.1396 | 97.60 | 2.40 | 97.60 | 2.40 | B + D |
| 5.968 | 0.495 | - | 0.0449 | 99.31 | 0.69 | 91.71 | 8.29 | B + D |
| 5.491 | 1.073 | - | 0.0283 | 99.57 | 0.43 | 83.30 | 16.70 | B + D |
| 4.710 | 1.776 | - | 0.0204 | 99.69 | 0.31 | 72.39 | 27.61 | B + D |
| 3.927 | 3.245 | - | 0.0343 | 99.52 | 0.48 | 54.49 | 45.51 | B + D + E |
| 1.898 | 4.985 | - | 0.0380 | 99.45 | 0.55 | 27.42 | 72.58 | B + E |
| 1.051 | 5.516 | - | 0.0419 | 99.37 | 0.63 | 15.91 | 84.09 | B + E |
| - | 6.362 | - | 0.0433 | 99.32 | 0.68 | - | 100.00 | B + E |
| - | 7.360 | - | 0.0454 | 99.39 | 0.61 | - | 100.00 | E + F |
| 0.243 | 6.109 | - | 0.0372 | 99.42 | 0.58 | 3.80 | 96.20 | E + F |
| 1.113 | 4.813 | - | 0.0261 | 99.56 | 0.44 | 18.69 | 81.31 | E + F |
| 1.478 | 4.758 | - | 0.0223 | 99.64 | 0.36 | 23.61 | 76.39 | E + F |
| 3.500 | 4.964 | - | 0.0280 | 99.67 | 0.33 | 41.22 | 58.78 | D + E + F |
| 2.797 | 6.847 | - | 0.0261 | 99.73 | 0.27 | 28.93 | 71.07 | D + F |
| 2.555 | 7.405 | - | 0.0210 | 99.79 | 0.21 | 25.60 | 74.40 | D + F |
| 1.745 | 9.544 | - | 0.0214 | 99.81 | 0.19 | 15.43 | 84.57 | D + F |
| 1.195 | 12.445 | - | 0.0233 | 99.83 | 0.17 | 8.75 | 91.25 | D + F |
| 0.678 | 16.725 | - | 0.0352 | 99.80 | 0.20 | 3.89 | 96.11 | D + F + G |
| 0.278 | 17.720 | - | 0.0379 | 99.79 | 0.21 | 1.54 | 98.46 | F + G |
| - | 18.019 | - | 0.0180 | 99.90 | 0.10 | - | 100.00 | F + G |
| 0.675 | 15.187 | - | - | 100.00 | - | 4.25 | 95.75 | D + G |

(continued next page)

COMPONENTS:

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

(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]

(3) Cesium chloride; CsCl; [7647-17-8]

(4) Cesium perchlorate; $CsClO_4$; [13454-84-7]

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

ORIGINAL MEASUREMENTS:

Shklovskaya, R.M.; Arkhipov, S.M.; Kuzina, V.A.

*Zh. Neorg. Khim.* 1974, *19*, 3128-33; **Russ. J. Inorg. Chem.* Engl. Transl.) 1974, *19*, 1711-4.

EXPERIMENTAL VALUES: (continued)

Solubility system $Na^+,Cs^+ \,||\, Cl^-,ClO_4^-$, water at 25.0 °C : (continued)

| Liquid phase composition, mass % | | | | Liquid phase composition, mol %[a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 17.448 | - | 20.646 | 0.290 | 7.7694 | - | 3.191 | 0.0325 | B + D |
| 19.001 | - | 16.230 | 0.286 | 8.1238 | - | 2.409 | 0.0308 | B + D |
| 21.515 | - | 9.075 | 0.381 | 8.6512 | - | 1.267 | 0.0385 | B + D |
| 23.412 | - | 3.731 | 0.710 | 9.0417 | - | 0.500 | 0.0690 | B + D |
| 24.305 | - | - | 2.378 | 9.2503 | - | - | 0.2276 | B + D |
| 24.567 | 4.266 | - | 0.734 | 9.6237 | 0.798 | - | 0.0723 | B + D |
| 21.997 | 9.003 | - | 0.451 | 8.8418 | 1.727 | - | 0.0456 | B + D |
| 18.381 | 14.522 | - | 0.316 | 7.5944 | 2.864 | - | 0.0328 | B + D |
| 14.037 | 24.306 | - | 0.488 | 6.2610 | 5.175 | - | 0.0547 | B + D + E |
| 6.411 | 35.277 | - | 0.511 | 3.040 | 7.984 | - | 0.0609 | B + E |
| 3.518 | 38.667 | - | 0.558 | 1.693 | 8.879 | - | 0.0675 | B + E |
| - | 43.540 | - | 0.563 | - | 10.275 | - | 0.0700 | B + E |
| - | 47.138 | - | 0.552 | - | 11.698 | - | 0.0722 | E + F |
| 0.801 | 42.240 | - | 0.488 | 0.392 | 9.870 | - | 0.0601 | E + F |
| 3.916 | 35.492 | - | 0.365 | 1.810 | 7.831 | - | 0.0424 | E + F |
| 5.158 | 34.801 | - | 0.309 | 2.392 | 7.704 | - | 0.0360 | E + F |
| 11.247 | 33.415 | - | 0.358 | 5.469 | 7.756 | - | 0.0438 | D + E + F |
| 8.142 | 41.754 | - | 0.302 | 4.292 | 10.506 | - | 0.0400 | D + F |
| 7.246 | 43.994 | - | 0.237 | 3.902 | 11.307 | - | 0.0321 | D + F |
| 4.483 | 51.352 | - | 0.219 | 2.612 | 14.283 | - | 0.0321 | D + F |
| 2.688 | 58.628 | - | 0.208 | 1.728 | 17.991 | - | 0.0336 | D + F |
| 1.280 | 66.152 | - | 0.264 | 0.929 | 22.928 | - | 0.0482 | D + F + G |
| 0.508 | 67.914 | - | 0.276 | 0.378 | 24.094 | - | 0.0516 | F + G |
| - | 68.722 | - | 0.130 | - | 24.501 | - | 0.0244 | F + G |
| 1.360 | 64.144 | - | - | 0.945 | 21.279 | - | - | D + G |

[a] Compiler's calculations.

[b] A = CsCl; B = $CsClO_4$; C = $Cs_{1-x}Na_xCl$; D = NaCl;
E = $3CsClO_4.NaClO_4$; F = $CsClO_4.NaClO_4$; G = $NaClO_4.H_2O$; H = $NaClO_4$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Brilliant Green and benzene (refs. 4,5); $Na^+$ by difference. Solid phases were identified by Schreinmakers' method and by x-ray diffraction.

REFERENCES:

3. Loebich, O. *Z. Analyt. Chem.* 1926, *68*, 34.

4. Golonitskaia, V.A. ; Petrashen, V.I. *Zh. Anal.Khim.* 1962, *17*, 848

5. Golonitskaia, V.A. ; Petrashen, V.I. *Zh. Tr. Novoch. Politekhn. Inst.* 1964, *141*, 65. (continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Cesium chloride; CsCl; [7647-17-8]<br>(4) Cesium perchlorate; $CsClO_4$; [13454-84-7]<br>(5) Water; $H_2O$; [7732-18-5] | Shklovskaya, R.M.; Arkhipov, S.M.; Kuzina, V.A.<br><br>*Zh. Neorg. Khim.* 1974, *19*, 3128-33; **Russ. J. Inorg. Chem.* Engl. Transl.) 1974, *19*, 1711-4. |

EXPERIMENTAL VALUES: (continued)

Solubility system $Na^+,Cs^+ \,||\, Cl^-,ClO_4^-$, water at 75.0 °C :

| Liquid phase composition, mass % | | | | Liquid phase composition, mol %[a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| - | - | 67.717 | 2.518 | - | - | 19.476 | 0.5247 | A + B |
| 6.157 | - | 59.744 | - | 4.477 | - | 15.081 | - | C + D |
| 6.787 | - | 57.734 | 1.402 | 4.928 | - | 14.551 | 0.2560 | B + C + D |
| 24.021 | - | - | 8.446 | 9.795 | - | - | 0.8663 | B + D |
| 5.706 | 44.779 | - | 3.346 | 3.211 | 12.028 | - | 0.4736 | B + D + F |
| - | 55.976 | - | 3.397 | - | 16.765 | - | 0.5361 | B + F |
| - | 74.185 | - | 0.539 | - | 30.125 | - | 0.1153 | F + H |
| 0.889 | 73.798 | - | 0.56 | 0.763 | 30.219 | - | 0.1200 | D + H + F |
| 0.881 | 75.116 | - | - | 0.769 | 31.286 | - | - | D + H |

| Liquid phase composition, molality[a] / mol $kg^{-1}$ | | | | Liquid phase composition, ion mol %[a] cation | | anion | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | $Na^+$ | $Cs^+$ | $Cl^-$ | $ClO_4^-$ | |
| - | - | 13.513 | 0.364 | - | 100.00 | 97.38 | 2.62 | A + B |
| 3.090 | - | 10.407 | - | 22.89 | 77.11 | 100.00 | 0.00 | C + D |
| 3.408 | - | 10.063 | 0.177 | 24.97 | 75.03 | 98.70 | 1.30 | B + C + D |
| 6.086 | - | - | 0.538 | 91.87 | 8.13 | 91.87 | 8.13 | B + D |
| 2.115 | 7.921 | - | 0.312 | 96.99 | 3.01 | 20.44 | 79.56 | B + D + F |
| - | 11.253 | - | 0.360 | 96.90 | 3.10 | - | 100.00 | B + F |
| - | 23.971 | - | 0.092 | 99.62 | 0.38 | - | 100.00 | H + F |
| 0.614 | 24.346 | - | 0.097 | 99.61 | 0.39 | 2.45 | 97.55 | D + H + F |
| 0.628 | 25.559 | - | - | 100.00 | - | 2.40 | 97.60 | D + H |

[a] Compiler's calculations.

[b] A = CsCl; B = $CsClO_4$; C = $Cs_{1-x}Na_xCl$; D = NaCl;
E = $3CsClO_4.NaClO_4$; F = $CsClO_4.NaClO_4$; G = $NaClO_4.H_2O$; H = $NaClO_4$

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5] | Karnaukhov, A.S.; Kudryakova, S.A. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 43-52. |
| (3) Ammonium chloride; $NH_4Cl$; [12125-02-9] | |
| (4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system $Na^+$, $NH_4^+$ || $ClO_4^-$, $Cl^-$ - water at 25 °C :

| Liquid phase composition | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| mol %[a] | | | | ion mol %[a] | | | | |
| (1) | (2) | (3) | (4) | $Na^+$ | $NH_4^+$ | $Cl^-$ | $ClO_4^-$ | |
| 0.808 | - | 10.74 | 1.123 | 6.38 | 93.62 | 91.13 | 8.87 | A + B |
| 1.609 | - | 10.09 | 0.859 | 12.81 | 87.19 | 93.16 | 6.84 | A + B |
| 2.205 | - | 9.75 | 0.813 | 17.27 | 82.73 | 93.64 | 6.36 | A + B |
| 2.415 | - | 10.02 | 0.749 | 18.32 | 81.68 | 94.32 | 5.68 | A + B |
| 4.052 | - | 8.39 | 0.658 | 30.94 | 69.06 | 94.98 | 5.02 | A + B |
| 6.066 | - | 7.35 | 0.919 | 42.33 | 57.67 | 93.59 | 6.41 | A + B |
| 6.561 | - | 7.02 | 0.965 | 45.12 | 54.88 | 93.37 | 6.63 | A + B |
| 6.759 | - | 6.64 | 1.018 | 46.89 | 53.11 | 92.94 | 7.06 | A + B + C |
| 6.740 | - | 6.63 | 0.975 | 46.98 | 53.02 | 93.20 | 6.80 | A + B + C |
| 6.725 | - | 6.62 | 0.962 | 47.01 | 52.99 | 93.28 | 6.72 | A + B + C |
| 7.524 | - | 5.02 | 1.315 | 54.29 | 45.71 | 90.51 | 9.49 | B + C |
| 8.111 | - | 3.68 | 1.302 | 61.97 | 38.03 | 90.06 | 9.94 | B + C |
| 9.511 | - | 0.207 | 2.664 | 76.82 | 23.18 | 78.49 | 21.51 | B + C |
| 7.832 | 2.60 | - | 1.783 | 85.41 | 14.59 | 64.11 | 35.89 | B + C |
| 6.856 | 4.42 | - | 1.495 | 88.30 | 11.70 | 53.68 | 46.32 | B + C |
| 5.938 | 5.49 | - | 1.247 | 90.16 | 9.84 | 46.84 | 53.16 | B + C |
| 5.767 | 5.82 | - | 1.228 | 90.42 | 9.58 | 45.00 | 55.00 | B + C |
| 4.295 | 9.92 | - | 1.310 | 91.56 | 8.44 | 27.66 | 72.34 | B + C |
| 3.049 | 12.35 | - | 1.109 | 93.28 | 6.72 | 18.47 | 81.53 | B + C |
| 2.137 | 15.09 | - | 0.948 | 94.78 | 5.22 | 11.76 | 88.24 | B + C |
| 1.983 | 17.05 | - | 1.002 | 95.00 | 5.00 | 9.89 | 90.11 | B + C + D |
| 1.649 | 17.79 | - | 1.000 | 95.11 | 4.89 | 8.07 | 91.93 | B + D + E |
| 1.625 | 18.25 | - | 1.032 | 95.06 | 4.94 | 7.77 | 92.23 | B + D + E |
| 1.593 | 18.72 | - | 1.070 | 95.00 | 5.00 | 7.45 | 92.55 | B + D + E |
| 0.270 | 18.72 | - | 0.879 | 95.57 | 4.43 | 1.36 | 98.64 | B + E |
| 0.906 | 19.63 | - | 0.952 | 95.57 | 4.43 | 4.22 | 95.78 | B + E |
| 6.37 | - | 7.061 | 0.337 | 46.27 | 53.73 | 97.55 | 2.45 | A + C |
| 6.75 | - | 6.673 | 0.948 | 46.97 | 53.03 | 93.40 | 6.60 | A + C |

[a] Compiler's calculations.

[b] A = $NH_4Cl$; B = $NH_4ClO_4$; C = NaCl; D = $NaClO_4.H_2O$;
E = $n(NH_4ClO_4).m(NaClO_4.H_2O)$

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5] | Karnaukhov, A.S.; Kudryakova, S.A. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 43-52. |
| (3) Ammonium chloride; $NH_4Cl$; [12125-02-9] | |
| (4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 298 K and 308 K. | N.A. Kozyreva |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $Na^+$, $NH_4^+$ || $ClO_4^-$, $Cl^-$ - water at 25 °C :

| Liquid phase composition | | | | | | | | Solid |
|---|---|---|---|---|---|---|---|---|
| mass % | | | | molality[a] / mol $kg^{-1}$ | | | | phase[b] |
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 2.03 | - | 24.68 | 5.67 | 0.514 | - | 6.823 | 0.714 | A + B |
| 4.07 | - | 23.37 | 4.37 | 1.021 | - | 6.407 | 0.545 | A + B |
| 5.56 | - | 22.51 | 4.12 | 1.403 | - | 6.206 | 0.517 | A + B |
| 6.06 | - | 23.01 | 3.78 | 1.544 | - | 6.406 | 0.479 | A + B |
| 10.17 | - | 19.27 | 3.32 | 2.588 | - | 5.358 | 0.420 | A + B |
| 14.78 | - | 16.38 | 4.50 | 3.931 | - | 4.759 | 0.595 | A + B |
| 15.90 | - | 15.56 | 4.70 | 4.262 | - | 4.556 | 0.627 | A + B |
| 16.38 | - | 14.72 | 4.96 | 4.383 | - | 4.304 | 0.660 | A + B + C |
| 16.37 | - | 14.74 | 4.76 | 4.368 | - | 4.297 | 0.632 | A + B + C |
| 16.35 | - | 14.73 | 4.70 | 4.356 | - | 4.288 | 0.623 | A + B + C |
| 18.21 | - | 11.12 | 6.40 | 4.848 | - | 3.235 | 0.848 | B + C |
| 19.84 | - | 8.23 | 6.40 | 5.180 | - | 2.348 | 0.831 | B + C |
| 22.61 | - | 0.45 | 12.73 | 6.025 | - | 0.131 | 1.687 | B + C |
| 17.83 | 12.41 | - | 8.16 | 4.953 | 1.645 | - | 1.127 | B + C |
| 14.90 | 20.13 | - | 6.53 | 4.363 | 2.813 | - | 0.951 | B + C |
| 12.67 | 24.55 | - | 5.35 | 3.775 | 3.491 | - | 0.793 | B + C |
| 12.19 | 25.78 | - | 5.22 | 3.672 | 3.706 | - | 0.782 | B + C |
| 7.99 | 38.67 | - | 4.90 | 2.822 | 6.520 | - | 0.861 | B + C |
| 5.36 | 45.48 | - | 3.92 | 2.027 | 8.211 | - | 0.738 | B + C |
| 3.51 | 51.93 | - | 3.13 | 1.450 | 10.237 | - | 0.643 | B + C |
| 3.08 | 55.50 | - | 3.13 | 1.376 | 11.838 | - | 0.696 | B + C + D |
| 2.52 | 56.94 | - | 3.07 | 1.151 | 12.411 | - | 0.697 | B + D + E |
| 2.45 | 57.65 | - | 3.13 | 1.140 | 12.805 | - | 0.725 | B + D + E |
| 2.37 | 58.37 | - | 3.20 | 1.125 | 13.220 | - | 0.755 | B + D + E |
| 0.41 | 59.46 | - | 2.68 | 0.187 | 12.967 | - | 0.609 | B + E |
| 1.33 | 60.34 | - | 2.81 | 0.641 | 13.874 | - | 0.673 | B + E |
| 15.89 | - | 16.12 | 1.69 | 4.101 | - | 4.545 | 0.217 | A + C |
| 16.40 | - | 14.84 | 4.63 | 4.376 | - | 4.326 | 0.614 | A + C |

(continued next page)

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS. |
|---|---|
| No details of method given. | Not stated. |
| | ESTIMATED ERROR: Not stated. |

(continued next page)

COMPONENTS:

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

(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]

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

(4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]

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

ORIGINAL MEASUREMENTS:

Karnaukhov, A.S.; Kudryakova, S.A.

*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 43-52.

EXPERIMENTAL VALUES: (continued)

Solubility system $Na^+$, $NH_4^+$ || $ClO_4^-$, $Cl^-$ - water at 35 °C :

| Liquid phase composition, mass % | | | | Liquid phase composition, molality[a] / mol kg$^{-1}$ | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 1.10 | 68.64 | - | - | 0.622 | 18.53 | - | - | C + D |
| 1.30 | 64.55 | - | 1.95 | 0.691 | 16.373 | - | 0.515 | C + D |
| 2.83 | 55.66 | - | 3.98 | 1.290 | 12.113 | - | 0.903 | B + C + D |
| - | 60.75 | - | 4.50 | - | 14.278 | - | 1.102 | B + E |
| - | 67.66 | - | 2.29 | - | 18.389 | - | 0.649 | D + E |
| 1.17 | 59.35 | - | 3.98 | 0.564 | 13.654 | - | 0.954 | B + D + E |
| 5.60 | 45.80 | - | 5.22 | 2.209 | 8.623 | - | 1.024 | B + C |
| 8.50 | 37.44 | - | 6.46 | 3.055 | 6.424 | - | 1.155 | B + C |
| 14.06 | 22.17 | - | 8.36 | 4.342 | 3.268 | - | 1.284 | B + C |
| 20.99 | 1.74 | - | 13.51 | 5.633 | 0.223 | - | 1.803 | B + C |
| 20.86 | - | 3.33 | 10.90 | 5.499 | - | 0.959 | 1.429 | B + C |
| 19.13 | - | 6.93 | 8.75 | 5.021 | - | 1.987 | 1.142 | B + C |
| 17.63 | - | 11.12 | 6.92 | 4.689 | - | 3.232 | 0.916 | B + C |
| 14.88 | - | 17.26 | 5.35 | 4.073 | - | 5.162 | 0.728 | A + B + C |
| 15.24 | - | 17.75 | 2.87 | 4.066 | - | 5.173 | 0.381 | A + C |
| 15.20 | - | 18.15 | - | 3.902 | - | 5.091 | - | A + C |
| 12.77 | - | 19.50 | 6.61 | 3.575 | - | 5.964 | 0.920 | A + B |
| 10.88 | - | 20.49 | 6.95 | 3.018 | - | 6.210 | 0.959 | A + B |
| 8.42 | - | 21.58 | 6.46 | 2.267 | - | 6.349 | 0.865 | A + B |
| 4.24 | - | 24.48 | 8.02 | 1.147 | - | 7.234 | 1.079 | A + B |
| - | - | 28.11 | 7.12 | - | - | 8.113 | 0.936 | A + B |

| Liquid phase composition, mol %[a] | | | | Liquid phase composition, ion mol %[a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | $Na^+$ | $NH_4^+$ | $Cl^-$ | $ClO_4^-$ | |
| 0.833 | 24.82 | - | - | 100.0 | - | 3.3 | 96.8 | C + D |
| 0.945 | 22.40 | - | 0.705 | 97.1 | 2.9 | 3.9 | 96.1 | C + D |
| 1.85 | 17.35 | - | 1.293 | 93.7 | 6.3 | 9.0 | 91.0 | B + C + D |
| - | 20.14 | - | 1.555 | 92.8 | 7.2 | - | 100.0 | B + E |
| - | 24.67 | - | 0.870 | 96.6 | 3.4 | - | 100.0 | D + E |
| 0.798 | 19.32 | - | 1.350 | 93.7 | 6.3 | 3.7 | 96.3 | B + D + E |
| 3.279 | 12.80 | - | 1.520 | 91.4 | 8.6 | 18.6 | 81.4 | B + C |
| 4.620 | 9.71 | - | 1.746 | 89.1 | 10.9 | 28.7 | 71.3 | B + C |
| 6.742 | 5.07 | - | 1.994 | 85.6 | 14.4 | 48.8 | 51.2 | B + C |
| 8.917 | 0.353 | - | 2.855 | 76.5 | 23.6 | 73.5 | 26.5 | B + C |
| 8.674 | - | 1.51 | 2.255 | 69.7 | 30.3 | 81.9 | 18.1 | B + C |
| 7.888 | - | 3.12 | 1.795 | 61.6 | 38.4 | 86.0 | 14.0 | B + C |
| 7.288 | - | 5.02 | 1.423 | 53.1 | 46.9 | 89.6 | 10.4 | B + C |
| 6.221 | - | 7.88 | 1.113 | 40.9 | 59.1 | 92.7 | 7.3 | A + B + C |
| 6.242 | - | 7.94 | 0.585 | 42.3 | 57.7 | 96.0 | 4.0 | A + C |
| 6.050 | - | 7.89 | - | 43.4 | 56.6 | 100.0 | - | A + C |
| 5.419 | - | 9.04 | 1.395 | 34.2 | 65.8 | 91.2 | 8.8 | A + B |
| 4.594 | - | 9.45 | 1.460 | 29.6 | 70.4 | 90.6 | 9.4 | A + B |
| 3.489 | - | 9.77 | 1.331 | 23.9 | 76.1 | 90.9 | 9.1 | A + B |
| 1.765 | - | 11.15 | 1.661 | 12.1 | 87.9 | 88.6 | 11.4 | A + B |
| - | - | 12.57 | 1.449 | - | 100.0 | 89.7 | 10.3 | A + B |

(continued next page)

COMPONENTS:

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

(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]

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

(4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]

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

ORIGINAL MEASUREMENTS:

Karnaukhov, A.S.; Kudryakova, S.A.

*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 43-52.

EXPERIMENTAL VALUES: (continued)

[a] Compiler's calculations.

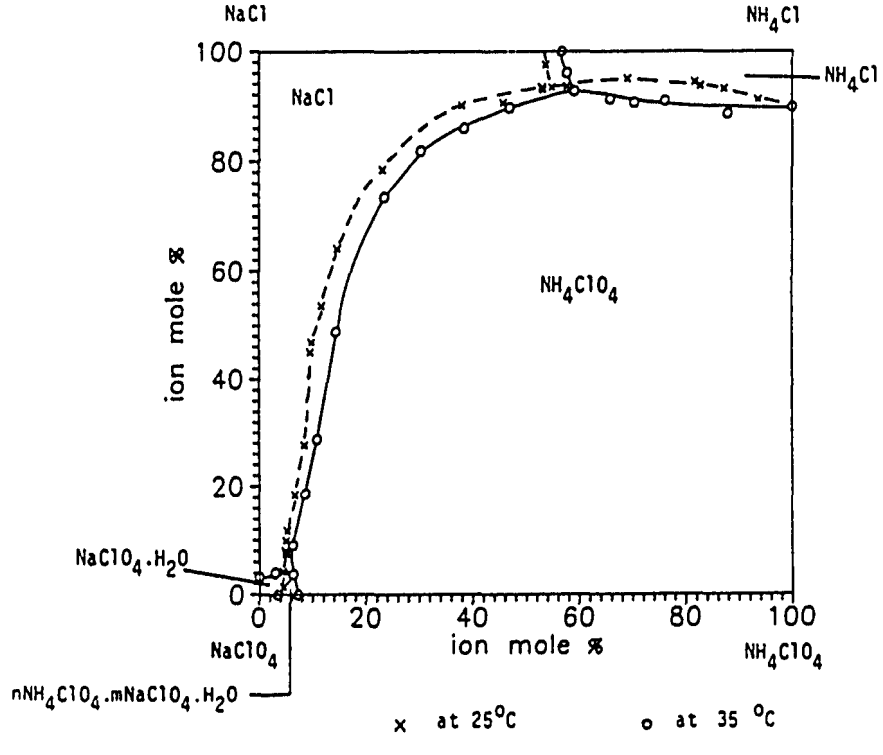
[b] A = $NH_4Cl$; B = $NH_4ClO_4$; C = NaCl; D = $NaClO_4.H_2O$; E = $n(NH_4ClO_4).m(NaClO_4.H_2O)$

COMMENTS AND/OR ADDITIONAL DATA

The phase diagrams shown below for both temperatures for this system show five crystallization fields: $NaClO_4.H_2O$; NaCl; $NH_4Cl$; $NH_4ClO_4$; and solid solutions of the type represented by $n(NH_4ClO_4).m(NaClO_4.H_2O)$. Increase in temperature causes a slight increase in the NaCl and $NH_4Cl$ crystallization fields and a decrease in the $NaClO_4.H_2O$ and $NH_4ClO_4$ crystallization fields. The equilibrium of the reaction

$$NaClO_4 + NH_4Cl = NaCl + NH_4ClO_4$$

is shifted in the direction of formation of ammonium perchlorate.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5] | Karnaukhov, A.S.; Kudryakova, S.A. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1971, *95*, 3-7. |
| (3) Ammonium chloride; $NH_4Cl$; [12125-02-9] | |
| (4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 363 K | N.A. Kozyreva |
| Composition | |

EXPERIMENTAL VALUES:

Solubility system $Na^+$, $NH_4^+$ || $ClO_4^-$, $Cl^-$ - water at 90 °C :

| Point | Liquid phase composition, mass % | | | | Liquid phase composition, molality[a] / mol kg$^{-1}$ | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 1 | 0.56 | - | 33.54 | 17.36 | 0.197 | - | 12.92 | 3.044 | A + B |
| 2 | 2.14 | - | 31.93 | 17.50 | 0.756 | - | 12.33 | 3.076 | A + B |
| 3 | 5.24 | - | 28.89 | 17.48 | 1.85 | - | 11.16 | 3.075 | A + B |
| 4 | 7.50 | - | 27.46 | 17.36 | 2.69 | - | 10.77 | 3.099 | A + B |
| 5 | 8.67 | - | 26.63 | 16.45 | 3.07 | - | 10.32 | 2.902 | A + B + C |
| 6 | 8.83 | - | 26.75 | 15.60 | 3.09 | - | 10.24 | 2.720 | A + B + C |
| 7 | 9.33 | - | 26.43 | 15.80 | 3.30 | - | 10.20 | 2.776 | A + B + C |
| 8 | 9.33 | - | 25.77 | 17.82 | 3.39 | - | 10.23 | 3.222 | A + B + C |
| 9 | 10.58 | - | 23.66 | 13.21 | 3.44 | - | 8.42 | 2.140 | A + C |
| 10 | 12.23 | - | 17.10 | 19.98 | 4.13 | - | 6.31 | 3.355 | A + C |
| 11 | 16.61 | - | 6.72 | 23.30 | 5.33 | - | 2.35 | 3.716 | A + C |
| 12 | 17.75 | - | 4.48 | 24.54 | 5.71 | - | 1.57 | 3.924 | A + C |
| 13 | 23.47 | 4.05 | - | 34.92 | 10.69 | 0.881 | - | 7.913 | A + C |
| 14 | 17.45 | 11.40 | - | 26.89 | 6.75 | 2.104 | - | 5.171 | A + C |
| 15 | 9.10 | 28.60 | - | 18.80 | 3.58 | 5.370 | - | 3.678 | A + C |
| 16 | 6.03 | 38.93 | - | 15.04 | 2.58 | 7.949 | - | 3.200 | A + C |
| 17 | 4.61 | 43.99 | - | 13.06 | 2.06 | 9.371 | - | 2.899 | A + C |
| 18 | 1.20 | 69.03 | - | 3.72 | 0.79 | 21.64 | - | 1.215 | A + C + D |
| 19 | 0.97 | 71.48 | - | 4.44 | 0.72 | 25.26 | - | 1.635 | A + E |
| 20 | 1.11 | 70.34 | - | 4.10 | 0.78 | 23.50 | - | 1.427 | A + D + E |
| 21 | 1.17 | 70.52 | - | 4.44 | 0.84 | 24.13 | - | 1.583 | A + D + E |
| 22 | 11.62 | - | 30.35 | 4.27 | 3.70 | - | 10.55 | 0.676 | B + C |
| I | - | - | 34.12 | 17.46 | - | - | 13.17 | 3.069 | A + B |
| II | 12.70 | - | 31.50 | - | 3.89 | - | 10.55 | - | B + C |
| III | 0.87 | 67.31 | - | - | 0.47 | 17.28 | - | - | C + D |
| IV | - | 78.28 | - | 3.63 | - | 35.34 | - | 1.708 | D + E |
| V | - | 68.13 | - | 6.28 | - | 21.74 | - | 2.089 | A + E |
| VI | 9.16 | - | 24.16 | 16.42 | 3.12 | - | 8.99 | 2.781 | A + B + C |
| VII | 1.20 | 69.03 | - | 3.72 | 0.79 | 21.64 | - | 1.215 | A + C + D |
| VIII | 1.11 | 70.34 | - | 4.20 | 0.78 | 23.60 | - | 1.468 | A + D + E |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4$; B = $NH_4Cl$; C = NaCl;
D = $NaClO_4$; E = $n(NH_4ClO_4).m(NaClO_4)$ .
Points I-VIII are nodal points of this quaternary aqueous reciprocal system.

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Ammonium chloride; $NH_4Cl$; [12125-02-9]<br>(4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]<br>(5) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Kudryakova, S.A.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1971, *95*, 3-7. |

EXPERIMENTAL VALUES: (continued)

Solubility system $Na^+$, $NH_4^+$ || $ClO_4^-$, $Cl^-$ - water at 90 °C :

| Point | Liquid phase composition mol %[a] | | | | ion mol %[a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | $Na^+$ | $NH_4^+$ | $Cl^-$ | $ClO_4^-$ | |
| 1 | 0.275 | - | 18.02 | 4.247 | 1.22 | 98.78 | 81.16 | 18.84 | A + B |
| 2 | 1.055 | - | 17.20 | 4.292 | 4.68 | 95.32 | 80.96 | 19.04 | A + B |
| 3 | 2.588 | - | 15.59 | 4.294 | 11.52 | 88.48 | 80.89 | 19.11 | A + B |
| 4 | 3.735 | - | 14.94 | 4.300 | 16.26 | 83.74 | 81.28 | 18.72 | A + B |
| 5 | 4.282 | - | 14.37 | 4.041 | 18.87 | 81.13 | 82.19 | 17.81 | A + B + C |
| 6 | 4.324 | - | 14.31 | 3.800 | 19.27 | 80.73 | 83.06 | 16.94 | A + B + C |
| 7 | 4.591 | - | 14.21 | 3.868 | 20.25 | 79.75 | 82.94 | 17.06 | A + B + C |
| 8 | 4.687 | - | 14.14 | 4.453 | 20.13 | 79.87 | 80.88 | 19.12 | A + B + C |
| 9 | 4.956 | - | 12.11 | 3.078 | 24.60 | 75.40 | 84.72 | 15.28 | A + C |
| 10 | 5.957 | - | 9.10 | 4.841 | 29.94 | 70.06 | 75.67 | 24.33 | A + C |
| 11 | 7.960 | - | 3.52 | 5.554 | 46.73 | 53.27 | 67.39 | 32.61 | A + C |
| 12 | 8.553 | - | 2.36 | 5.882 | 50.93 | 49.07 | 64.97 | 35.03 | A + C |
| 13 | 14.257 | 1.174 | - | 10.552 | 59.39 | 40.61 | 54.87 | 45.13 | A + C |
| 14 | 9.703 | 3.026 | - | 7.437 | 63.12 | 36.88 | 48.11 | 51.89 | A + C |
| 15 | 5.253 | 7.881 | - | 5.399 | 70.87 | 29.13 | 28.35 | 71.65 | A + C |
| 16 | 3.726 | 11.481 | - | 4.622 | 76.69 | 23.31 | 18.79 | 81.21 | A + C |
| 17 | 2.946 | 13.418 | - | 4.152 | 79.76 | 20.24 | 14.36 | 85.64 | A + C |
| 18 | 0.996 | 27.342 | - | 1.54 | 94.86 | 5.14 | 3.33 | 96.67 | A + C + D |
| 19 | 0.864 | 30.390 | - | 1.97 | 94.08 | 5.92 | 2.60 | 97.40 | A + E |
| 20 | 0.957 | 28.933 | - | 1.76 | 94.45 | 5.55 | 3.02 | 96.98 | A + D + E |
| 21 | 1.022 | 29.404 | - | 1.93 | 94.04 | 5.96 | 3.16 | 96.84 | A + D + E |
| 22 | 5.251 | - | 14.98 | 0.960 | 24.77 | 75.23 | 95.47 | 4.53 | B + C |
| I | - | - | 18.36 | 4.278 | - | 100.00 | 81.10 | 18.90 | A + B |
| II | 5.567 | - | 15.09 | - | 26.95 | 73.05 | 100.00 | - | B + C |
| III | 0.639 | 23.585 | - | - | 100.00 | - | 2.64 | 97.36 | C + D |
| IV | - | 38.183 | - | 1.845 | 95.39 | 4.61 | - | 100.00 | D + E |
| V | - | 27.406 | - | 2.633 | 91.24 | 8.76 | - | 100.00 | A + E |
| VI | 4.430 | - | 12.77 | 3.950 | 20.95 | 79.05 | 81.32 | 18.68 | A + B + C |
| VII | 0.996 | 27.342 | - | 1.536 | 94.86 | 5.14 | 3.33 | 96.67 | A + C + D |
| VIII | 0.959 | 29.002 | - | 1.805 | 94.32 | 5.68 | 3.02 | 96.98 | A + D + E |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4$; B = $NH_4Cl$; C = NaCl;
D = $NaClO_4$; E = n($NH_4ClO_4$).m($NaClO_4$) .

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

No details of method given. $Na^+$ was determined by precipitating as sodium uranyl acetate; $Cl^-$ was determined mercurimetrically; $ClO_4^-$ gravimetrically by precipitation with nitron; and $NH_4^+$ by the volumetric formalin method (ref. 1).

(continued next page)

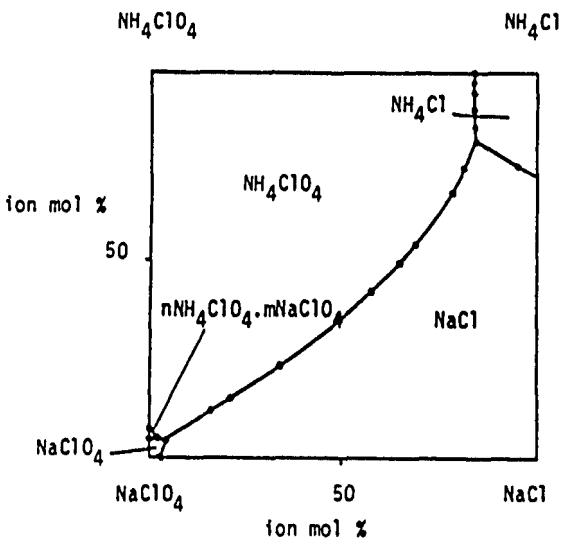
| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Ammonium chloride; $NH_4Cl$; [12125-02-9]<br>(4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]<br>(5) Water; $H_2O$; [7732-18-5] | Karnaukhov, A.S.; Kudryakova, S.A.<br><br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1971, *95*, 3-7. |

EXPERIMENTAL VALUES: (continued)

COMMENTS AND/OR ADDITIONAL DATA

The solubility diagram given below shows five crystallization fields: NaCl, $NaClO_4$, $NH_4Cl$, $NH_4ClO_4$ and solid solutions represented by $n(NH_4ClO_4).m(NaClO_4)$ .

Three triple points were found that corresponded to equilibria with the following combination of solid phases: (point VI) NaCl + $NH_4Cl$ + $NH_4ClO_4$, (point VII) $NaClO_4$ + NaCl + $NH_4ClO_4$, and (point VIII) $NaClO_4$ + $NH_4ClO_4$ + $n(NH_4ClO_4).m(NaClO_4)$ .




AUXILIARY INFORMATION

| SOURCE AND PURITY OF MATERIALS: | ESTIMATED ERROR: |
|---|---|
| Not stated. | Not stated. |

REFERENCES:

1. Karnaukhov, A.S.; Kudryakova, S.A. *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *79*, 32.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Freeth, F.A. |
| (2) Sodium sulfate; $Na_2SO_4$; [7757-82-6] | *Rec. Trav. Chim. Pays-Bas* 1924, 43, 475-507 |
| (3) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (4) Ammonium sulfate; $(NH_4)_2SO_4$; [7783-20-2] | |
| (5) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| Two temperatures: 298 K and 333 K. | C.Y. Chan |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $2NH_4^+$, $2Na^+$ || $2ClO_4^-$, $SO_4^{2-}$ - $H_2O$ at 25 °C :

| Liquid phase composition | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| mass % | | | | mol %[a] | | | | |
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| - | - | 3.00 | 41.70 | - | - | 0.749 | 9.253 | A + B |
| - | 3.47 | 2.71 | 39.55 | - | 0.727 | 0.687 | 8.910 | A + B |
| - | 6.87 | 2.66 | 37.38 | - | 1.465 | 0.686 | 8.570 | A + B + C |
| - | 8.20 | - | 38.70 | - | 1.750 | - | 8.880 | B + C |
| - | 6.83 | 2.28 | 37.69 | - | 1.455 | 0.587 | 8.628 | A + C |
| - | 9.82 | 2.84 | 30.70 | - | 1.993 | 0.697 | 6.696 | A + C |
| - | 14.69 | 3.48 | 22.47 | - | 2.874 | 0.823 | 4.726 | A + C |
| - | 20.39 | 4.30 | 15.08 | - | 3.946 | 1.006 | 3.137 | A + C |
| - | 21.81 | 4.07 | 14.10 | - | 4.234 | 0.955 | 2.942 | A + C |
| - | 24.38 | 4.36 | 11.59 | - | 4.756 | 1.028 | 2.431 | A + D + C |
| - | 25.76 | - | 14.10 | - | 5.001 | - | 2.943 | D + C |
| - | 24.97 | 1.60 | 13.15 | - | 4.836 | 0.375 | 2.738 | D + C |
| - | 9.84 | 3.85 | 28.90 | - | 1.975 | 0.934 | 6.236 | A |
| - | 23.22 | 7.80 | 6.56 | - | 4.366 | 1.773 | 1.326 | A + D |
| - | 22.45 | 13.47 | - | - | 4.127 | 2.994 | - | A + D |
| 12.71 | 15.79 | 9.41 | - | 2.774 | 2.971 | 2.141 | - | A + D |
| 24.38 | 10.79 | 6.73 | - | 5.597 | 2.135 | 1.610 | - | A + D |
| 31.21 | 9.07 | - | - | 7.015 | 1.757 | - | - | D + E |
| 31.62 | 6.91 | 5.74 | - | 7.487 | 1.410 | 1.416 | - | A + E |
| - | 25.94 | 6.09 | 7.83 | - | 5.028 | 1.427 | 1.632 | D + E |
| 67.67 | 0.26 | - | - | 23.673 | 0.078 | - | - | E + F |
| 67.42 | - | 1.51 | - | 24.065 | - | 0.562 | - | A + F |
| 66.79 | 0.21 | 1.50 | - | 23.632 | 0.064 | 0.553 | - | A + E + F |

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The saturation apparatus was similar to that used by Van't Hoff (ref.1) and samples of clear satd sln were taken using a weight-pipette. $Na^+$ was determined as $Na_2SO_4$ by addition of pure sulphuric acid to the sln in | $NaClO_4$ was prepared from very pure ammonium perchlorate (% purity not stated) and an aqueous sln of pure NaOH. Source and other details not given. $Na_2SO_4$ was recrystallized from the reagent grade salt. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Freeth, F.A. |
| (2) Sodium sulfate; $Na_2SO_4$; [7757-82-6] | *Rec. Trav. Chim. Pays-Bas* 1924, *43*, 475-507 |
| (3) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (4) Ammonium sulfate; $(NH_4)_2SO_4$; [7783-20-2] | |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

Solubility system $2NH_4^+$, $2Na^+$ || $2ClO_4^-$, $SO_4^{2-}$ - $H_2O$ at 25 °C :

| Liquid phase composition | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| molality[a]/ mol $kg^{-1}$ | | | | ion mol %[a] | | | | |
| (1) | (2) | (3) | (4) | $2Na^+$ | $2NH_4^+$ | $2ClO_4^-$ | $SO_4^{2-}$ | |
| - | - | 0.462 | 5.707 | - | 100.00 | 3.89 | 96.11 | A + B |
| - | 0.450 | 0.425 | 5.515 | 7.29 | 92.71 | 3.44 | 96.56 | A + B |
| - | 0.911 | 0.426 | 5.328 | 14.12 | 85.88 | 3.30 | 96.70 | A + B + C |
| - | 1.087 | - | 5.516 | 16.47 | 83.53 | - | 100.00 | B + C |
| - | 0.904 | 0.365 | 5.361 | 14.02 | 85.98 | 2.83 | 97.17 | A + C |
| - | 1.221 | 0.427 | 4.102 | 22.05 | 77.95 | 3.85 | 96.15 | A + C |
| - | 1.742 | 0.499 | 2.865 | 35.88 | 64.12 | 5.14 | 94.86 | A + C |
| - | 2.383 | 0.608 | 1.895 | 52.02 | 47.98 | 6.63 | 93.37 | A + C |
| - | 2.558 | 0.577 | 1.778 | 55.32 | 44.68 | 6.24 | 93.76 | A + C |
| - | 2.876 | 0.622 | 1.470 | 61.76 | 38.24 | 6.68 | 93.32 | A + D + C |
| - | 3.016 | - | 1.774 | 62.96 | 37.04 | - | 100.00 | D + C |
| - | 2.916 | 0.226 | 1.651 | 62.31 | 37.69 | 2.41 | 97.59 | D + C |
| - | 1.207 | 0.571 | 3.810 | 22.76 | 77.24 | 5.38 | 94.62 | A |
| - | 2.619 | 1.064 | 0.795 | 66.37 | 33.63 | 13.48 | 86.52 | A + D |
| - | 2.466 | 1.789 | - | 73.38 | 26.62 | 26.62 | 73.38 | A + D |
| 1.672 | 1.790 | 1.290 | - | 80.28 | 19.72 | 45.27 | 54.73 | A + D |
| 3.427 | 1.307 | 0.986 | - | 85.97 | 14.03 | 62.79 | 37.21 | A + D |
| 4.268 | 1.069 | - | - | 100.00 | - | 66.62 | 33.38 | D + E |
| 4.634 | 0.873 | 0.877 | - | 87.92 | 12.08 | 75.94 | 24.06 | A + E |
| - | 3.037 | 0.862 | 0.985 | 68.19 | 31.81 | 9.68 | 90.32 | D + E |
| 17.23 | 0.057 | - | - | 100.00 | - | 99.34 | 0.66 | E + F |
| 17.72 | - | 0.414 | - | 97.72 | 2.28 | 100.00 | - | A + F |
| 17.32 | 0.047 | 0.405 | - | 97.73 | 2.27 | 99.47 | 0.53 | A + E + F |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4$ B = $(NH_4)_2SO_4$ C = $Na_2SO_4.(NH_4)_2SO_4.4H_2O$
D = $Na_2SO_4.10H_2O$ E = $Na_2SO_4$ F = $NaClO_4.H_2O$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: (continued)

silica basins and evaporating at a low red heat. Sulfate was determined gravimetrically as $BaSO_4$. To determine ammonia content, the slns were distilled with excess NaOH and the ammonia distillate absorbed in excess of standard acid solution, followed by back-titration of the acid. All analyses were duplicated. Solid phase compositions were determined using Schreinemakers' method. Gas-heated thermostats were used and thermometers were checked against N.P.L. Standards.

(continued next page)

COMPONENTS:

(1) Sodium perchlorate; $NaClO_4$; [7601-89-0]

(2) Sodium sulfate; $Na_2SO_4$; [7757-82-6]

(3) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]

(4) Ammonium sulfate; $(NH_4)_2SO_4$; [7783-20-2]

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

ORIGINAL MEASUREMENTS:

Freeth, F.A.

*Rec. Trav. Chim. Pays-Bas* 1924, 43, 475-507

EXPERIMENTAL VALUES: (continued)

Solubility system $2NH_4^+$, $2Na^+$ || $2ClO_4^-$, $SO_4^{2-}$ - $H_2O$ at 60 °C :

| Liquid phase composition, mass % | | | | Liquid phase composition, mol %[a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| - | - | 8.21 | 40.92 | - | - | 2.181 | 9.667 | A + B |
| - | 5.06 | 6.79 | 38.68 | - | 1.137 | 1.845 | 9.346 | A + B |
| - | 8.62 | 6.27 | 37.07 | - | 1.982 | 1.743 | 9.164 | A + B |
| - | 14.51 | 5.12 | 34.66 | - | 3.468 | 1.480 | 8.906 | A + B + E |
| - | 14.66 | 4.95 | 34.65 | - | 3.503 | 1.430 | 8.899 | A + B + E |
| - | 15.27 | 2.77 | 35.26 | - | 3.595 | 0.788 | 8.924 | B + E |
| - | 16.33 | - | 36.91 | - | 3.845 | - | 9.342 | B + E |
| - | 17.27 | 9.86 | 21.33 | - | 3.767 | 2.600 | 5.001 | A + E |
| - | 18.35 | 16.25 | 10.51 | - | 3.806 | 4.075 | 2.344 | A + E |
| - | 18.60 | 25.30 | - | - | 3.784 | 6.223 | - | A + E |
| 10.42 | 12.52 | 21.42 | - | 2.47 | 2.559 | 5.294 | - | A + E |
| 26.77 | 4.61 | 15.36 | - | 6.55 | 0.972 | 3.916 | - | A + E |
| 34.87 | 2.79 | 12.88 | - | 9.01 | 0.622 | 3.470 | - | A + E |
| 44.02 | 1.44 | 9.04 | - | 12.10 | 0.341 | 2.589 | - | A + E |
| 56.69 | 0.730 | 7.11 | - | 18.54 | 0.206 | 2.423 | - | A + E |
| 61.24 | 0.370 | 5.00 | - | 20.85 | 0.109 | 1.774 | - | A + E |
| 72.98 | 0.240 | 1.86 | - | 29.85 | 0.085 | 0.793 | - | A + E + G |
| 72.86 | - | 1.87 | - | 29.55 | - | 0.790 | - | A + G |
| 74.40 | 0.290 | - | - | 30.16 | 0.101 | - | - | E + G |

| Liquid phase composition, molality[a]/ mol $kg^{-1}$ | | | | Liquid phase composition, ion mol %[a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | $2Na^+$ | $2NH_4^+$ | $2ClO_4^-$ | $SO_4^{2-}$ | |
| - | - | 1.374 | 6.088 | - | 100.00 | 10.14 | 89.86 | A + B |
| - | 0.720 | 1.168 | 5.917 | 9.97 | 90.03 | 8.09 | 91.91 | A + B |
| - | 1.263 | 1.111 | 5.840 | 16.50 | 83.50 | 7.25 | 92.75 | A + B |
| - | 2.235 | 0.953 | 5.738 | 26.45 | 73.55 | 5.64 | 94.36 | A + B + E |
| - | 2.256 | 0.921 | 5.733 | 26.70 | 73.30 | 5.45 | 94.55 | A + B + E |
| - | 2.302 | 0.505 | 5.714 | 27.84 | 72.16 | 3.05 | 96.95 | B + E |
| - | 2.459 | - | 5.974 | 29.16 | 70.84 | - | 100.00 | B + E |
| - | 2.359 | 1.628 | 3.132 | 37.41 | 62.59 | 12.91 | 87.09 | A + E |
| - | 2.354 | 2.520 | 1.449 | 46.49 | 53.51 | 24.89 | 75.11 | A + E |
| - | 2.334 | 3.838 | - | 54.88 | 45.12 | 45.12 | 54.88 | A + E |
| 1.530 | 1.584 | 3.277 | - | 58.91 | 41.09 | 60.27 | 39.73 | A + E |
| 4.105 | 0.609 | 2.455 | - | 68.44 | 31.56 | 84.33 | 15.67 | A + E |
| 5.758 | 0.397 | 2.216 | - | 74.72 | 25.28 | 90.94 | 9.06 | A + E |
| 7.902 | 0.223 | 1.691 | - | 83.15 | 16.85 | 95.56 | 4.44 | A + E |
| 13.05 | 0.145 | 1.706 | - | 88.66 | 11.34 | 98.07 | 1.93 | A + E |
| 14.98 | 0.078 | 1.275 | - | 92.23 | 7.77 | 99.05 | 0.95 | A + E |
| 23.92 | 0.068 | 0.635 | - | 97.43 | 2.57 | 99.45 | 0.55 | A + E + G |
| 23.55 | - | 0.630 | - | 97.39 | 2.61 | 100.00 | - | A + G |
| 24.01 | 0.081 | - | - | 100.00 | - | 99.33 | 0.67 | E + G |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium perchlorate; $NaClO_4$; [7601-89-0] | Freeth, F.A. |
| (2) Sodium sulfate; $Na_2SO_4$; [7757-82-6] | *Rec. Trav. Chim. Pays-Bas* 1924, *43*, 475-507 |
| (3) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (4) Ammonium sulfate; $(NH_4)_2SO_4$; [7783-20-2] | |
| (5) Water; $H_2O$; [7732-18-5] | |

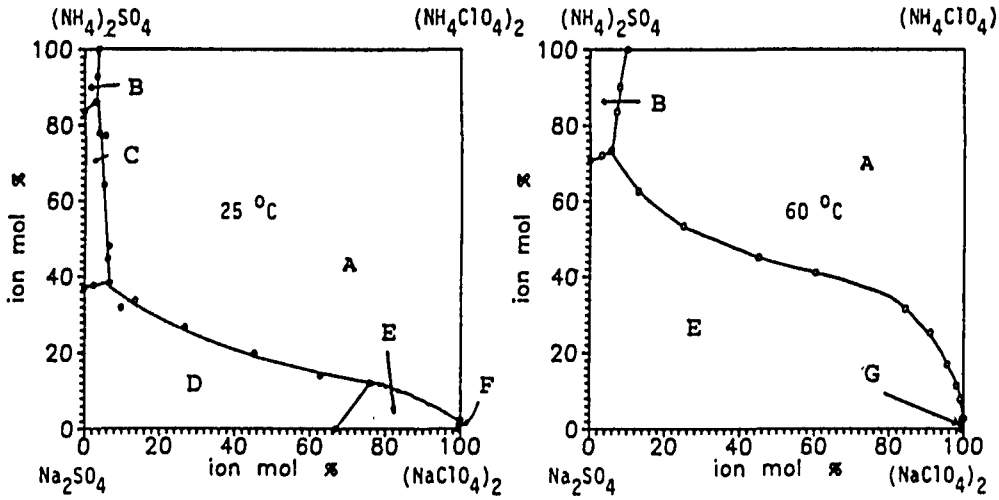
EXPERIMENTAL VALUES: (continued)

[a] Compiler's calculations.

[b] A = $NH_4ClO_4$ B = $(NH_4)_2SO_4$ C = $Na_2SO_4.(NH_4)_2SO_4.4H_2O$
D = $Na_2SO_4.10H_2O$ E = $Na_2SO_4$ F = $NaClO_4.H_2O$ .
G = $NaClO_4$

COMMENTS AND/OR ADDITIONAL DATA:

The Janecke diagrams for the system $2NH_4^+, 2Na^+ \,||\, 2ClO_4^-, SO_4^{2-} - H_2O$ at 25 °C and 60 °C are shown below.




AUXILLIARY INFORMATION

| ESTIMATED ERROR: | REFERENCES: |
|---|---|
| Not stated. | 1. Van't Hoff, J.H. *Zur Bildung der Ozeanischen Salzablagerungen Wieweg, Braunschweig* 1905, *1*; 1902, *2*. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chromate; $Na_2CrO_4$; [7775-11-3]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Ammonium chromate; $(NH_4)_2CrO_4$; [7788-98-9]<br>(4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]<br>(5) Water; $H_2O$; [7732-18-5] | Lepeshkov, I.N.; Karnaukhov, A.S.; Molchanov, S.M.<br><br>*Tr. po Khimii i Khim. Tekhnologii* <u>1966</u>, *2*, 215-18. |
| VARIABLES:<br>Temperature: 298 K.<br>Composition. | PREPARED BY:<br>N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system $2Na^+$, $2NH_4^+$ || $2ClO_4^-$, $CrO_4^{2-}$ - water at 25 °C :

| Point | Liquid phase composition, mass % | | | | molality[a] / mol kg$^{-1}$ | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| I | - | - | 3.85 | 17.56 | - | - | 0.322 | 1.902 | A + B |
| II | - | - | 25.50 | 1.08 | - | - | 2.284 | 0.125 | B + C |
| III | 16.22 | 0.39 | - | 13.16 | 1.426 | 0.045 | - | 1.593 | A + B + C |
| IV | 16.19 | - | 21.61 | - | 1.607 | - | 2.285 | - | C + D |
| V | 44.95 | - | 1.50 | - | 5.182 | - | 0.184 | - | D + E |
| VI | 31.23 | 2.13 | - | 8.14 | 3.296 | 0.297 | - | 1.184 | A + C + D |
| VII | 38.71 | 8.15 | - | 2.93 | 4.760 | 1.326 | - | 0.497 | A + D + E |
| VIII | 7.82 | 53.91 | - | - | 1.262 | 11.505 | - | - | E + F |
| IX | 6.18 | 60.70 | - | - | 1.152 | 14.968 | - | - | F + G |
| X | 9.94 | 52.72 | - | 2.08 | 1.740 | 12.211 | - | 0.502 | A + E + F |
| XI | 6.99 | 58.68 | - | 1.63 | 1.320 | 14.656 | - | 0.424 | A + G + H |
| XII | 3.77 | 61.88 | - | 1.63 | 0.711 | 15.446 | - | 0.424 | F + G + H |
| XIII | - | 66.35 | - | 1.92 | - | 17.078 | - | 0.515 | G + H |
| XIV | - | 61.92 | - | 2.56 | - | 14.237 | - | 0.613 | A + H |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4$; B = $NH_4ClO_4.(NH_4)_2CrO_4$; C = $(NH_4)_2CrO_4$;
D = $NaNH_4CrO_4.2H_2O$; E = $Na_2CrO_4.4H_2O$; F = $Na_2CrO_4$;
G = $NaClO_4.H_2O$; H = $nNH_4ClO_4.mNaClO_4$.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Method of "invariant points" used. Periods of equilibration varied from 3 to 10 days. $Na^+$ was determined gravimetrically as $NaZn(UO_2)_3(C_2H_3O_2)_9$; $NH_4^+$ by the "formalin method"; chromate iodimetrically and perchlorate by difference. | Not stated.<br><br>ESTIMATED ERROR:<br>Not stated.<br><br>REFERENCES:<br>(continued next page) |

COMPONENTS:

(1) Sodium chromate; $Na_2CrO_4$; [7775-11-3]

(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]

(3) Ammonium chromate; $(NH_4)_2CrO_4$; [7788-98-9]

(4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]

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

ORIGINAL MEASUREMENTS:

Lepeshkov, I.N.; Karnaukhov, A.S.; Molchanov, S.M.

*Tr. po Khimii i Khim. Tekhnologii* 1966, *2*, 215-18.

EXPERIMENTAL VALUES: (continued)

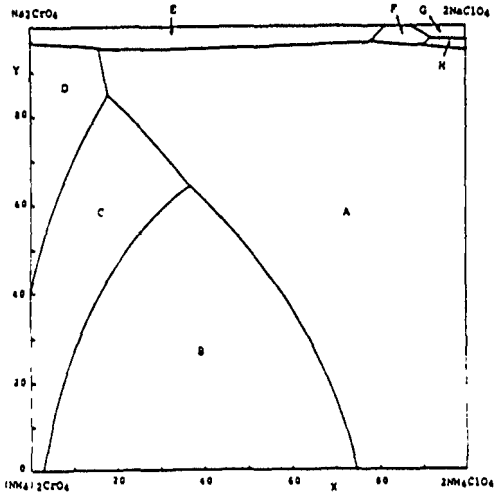
| Point | Liquid phase composition mol %[a] (1) | (2) | (3) | (4) | ion mol %[a] $2Na^+$ | $2NH_4^+$ | $CrO_4^{2-}$ | $2ClO_4^-$ | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| I | - | - | 0.56 | 3.294 | - | 100.00 | 25.31 | 74.69 | A + B |
| II | - | - | 3.94 | 0.216 | - | 100.00 | 97.33 | 2.67 | B + C |
| III | 2.43 | 0.08 | - | 2.720 | 64.51 | 35.49 | 63.50 | 36.50 | A + B + C |
| IV | 2.71 | - | 3.85 | - | 41.29 | 58.71 | 100.00 | - | C + D |
| V | 8.51 | - | 0.30 | - | 96.57 | 3.43 | 100.00 | - | D + E |
| VI | 5.47 | 0.49 | - | 1.965 | 85.33 | 14.67 | 81.65 | 18.35 | A + C + D |
| VII | 7.67 | 2.14 | - | 0.800 | 95.62 | 4.38 | 83.93 | 16.07 | A + D + E |
| VIII | 1.85 | 16.85 | - | - | 100.00 | - | 17.99 | 82.01 | E + F |
| IX | 1.61 | 20.90 | - | - | 100.00 | - | 13.34 | 86.66 | F + G |
| X | 2.49 | 17.45 | - | 0.718 | 96.90 | 3.10 | 21.49 | 78.51 | A + E + F |
| XI | 1.84 | 20.38 | - | 0.590 | 97.61 | 2.39 | 14.90 | 85.10 | A + F + H |
| XII | 0.99 | 21.43 | - | 0.588 | 97.55 | 2.45 | 8.23 | 91.77 | F + G + H |
| XIII | - | 23.36 | - | 0.705 | 97.07 | 2.93 | - | 100.00 | G + H |
| XIV | - | 20.24 | - | 0.872 | 95.87 | 4.13 | - | 100.00 | A + H |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4$; B = $NH_4ClO_4.(NH_4)_2CrO_4$; C = $(NH_4)_2CrO_4$;
D = $NaNH_4CrO_4.2H_2O$; E = $Na_2CrO_4.4H_2O$; F = $Na_2CrO_4$;
G = $NaClO_4.H_2O$; H = $nNH_4ClO_4.mNaClO_4$.

COMMENTS AND/OR ADDITIONAL DATA

The phase diagram given below shows eight crystallization fields:
(a) $NH_4ClO_4$; (b) $NH_4ClO_4.(NH_4)_2CrO_4$; (c) $(NH_4)_2CrO_4$;
(d) $NaNH_4CrO_4.2H_2O$; (e) $Na_2CrO_4.4H_2O$; (f) $Na_2CrO_4$; (g) $NaClO_4.H_2O$;
(h) solid solutions represented by $nNH_4ClO_4.mNaClO_4$




$X = 100X_{ClO_4}/(X_{ClO_4}+2X_{CrO_4})$

$Y = 100X_{NH_4}/(X_{NH_4}+X_{Na})$

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chromate; $Na_2CrO_4$; [7775-11-3] | Guseva, A.D.; Druzhinina, G.V. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *78*, 32-8. |
| (3) Ammonium chromate; $(NH_4)_2CrO_4$; [7788-98-9] | |
| (4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 308 K. | I.S. Bodnya |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $2Na^+$, $2NH_4^+$ || $2ClO_4^-$, $CrO_4^{2-}$ - water at 35 °C :

| Point | Liquid phase composition, mass % | | | | molality[a] / mol kg$^{-1}$ | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| I | - | - | 3.62 | 20.45 | - | - | 0.314 | 2.292 | A + B |
| | 3.84 | - | 1.11 | 18.04 | 0.308 | - | 0.095 | 1.994 | A + B |
| | 10.53 | - | 0.71 | 22.40 | 0.980 | - | 0.070 | 2.873 | A + B |
| II | 17.05 | - | 0.83 | 14.42 | 1.555 | - | 0.081 | 1.813 | A + B + C |
| | 24.10 | - | 1.76 | 12.11 | 2.399 | - | 0.187 | 1.662 | B + C |
| III | 31.24 | - | 2.88 | 7.30 | 3.292 | - | 0.323 | 1.061 | B + C + D |
| | - | - | 28.00 | 1.19 | - | - | 2.600 | 0.143 | A + C |
| | 2.61 | - | 25.64 | 1.11 | 0.228 | - | 2.387 | 0.134 | A + C |
| | 4.79 | - | 17.71 | 2.40 | 0.394 | - | 1.551 | 0.272 | A + C |
| | 11.27 | - | 4.82 | 13.87 | 0.993 | - | 0.453 | 1.686 | A + C |
| | 15.36 | - | 3.83 | 14.59 | 1.432 | - | 0.380 | 1.875 | A + C |
| V | 20.36 | - | 20.45 | - | 2.124 | - | 2.272 | - | C + D |
| | 26.35 | - | 11.37 | 2.93 | 2.741 | - | 1.260 | 0.420 | C + D |
| VI | 45.92 | - | 2.18 | - | 5.462 | - | 0.276 | - | D + E |
| | 44.66 | 3.25 | - | 4.04 | 5.738 | 0.552 | - | 0.716 | D + E |
| VII | 36.66 | 12.67 | - | 2.57 | 4.705 | 2.151 | - | 0.455 | B + D + E |
| | 25.43 | 26.79 | - | 4.82 | 3.655 | 5.093 | - | 0.955 | B + E |
| | 16.16 | 36.32 | - | 4.95 | 2.344 | 6.968 | - | 0.990 | B + E |
| VIII | 14.19 | 40.90 | - | 5.67 | 2.233 | 8.513 | - | 1.230 | B + E + F |
| | 8.38 | 47.67 | - | 4.04 | 1.296 | 9.755 | - | 0.862 | B + F |
| IX | - | 60.75 | - | 4.50 | - | 14.278 | - | 1.102 | B + F |
| X | 11.52 | 47.24 | - | 3.91 | 1.905 | 10.335 | - | 0.891 | E + F + G |
| XI | 8.63 | 61.62 | - | - | 1.791 | 16.916 | - | - | E + G |
| XII | - | 67.64 | - | 2.33 | - | 18.396 | - | 0.660 | F + G |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4.(NH_4)_2CrO_4$; B = $NH_4ClO_4$; C = $(NH_4)_2CrO_4$;
D = $NaNH_4CrO_4.2H_2O$; E = $Na_2CrO_4.4H_2O$;
F = $n(NH_4ClO_4).m(NaClO_4.H_2O)$; G = $NaClO_4.H_2O$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: Method of "invariant points" used. Details of method not given. $Na^+$ was analysed gravimetrically as sodium zinc uranyl acetate; $NH_4^+$ by distillation and titrimetry; $CrO_4^{2-}$ iodimetrically and $ClO_4^-$ by difference.

(continued next page)

COMPONENTS:

(1) Sodium chromate; $Na_2CrO_4$; [7775-11-3]

(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]

(3) Ammonium chromate; $(NH_4)_2CrO_4$; [7788-98-9]

(4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]

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

ORIGINAL MEASUREMENTS:

Guseva, A.D.; Druzhinina, G.V.

*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *78*, 32-8.

EXPERIMENTAL VALUES: (continued)

| Point | Liquid phase composition, mol % [a] | | | | Ion mol % [a] | | | | Solid phase [b] |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | $2Na^+$ | $2NH_4^+$ | $CrO_4^{2-}$ | $2ClO_4^-$ | |
| I | - | - | 0.539 | 3.945 | - | 100.00 | 21.48 | 78.52 | A + B |
| | 0.532 | - | 0.164 | 3.443 | 22.00 | 78.00 | 28.77 | 71.23 | A + B |
| | 1.648 | - | 0.118 | 4.834 | 39.40 | 60.60 | 42.23 | 57.77 | A + B |
| II | 2.637 | - | 0.137 | 3.075 | 61.17 | 38.83 | 64.34 | 35.66 | A + B + C |
| | 4.014 | - | 0.312 | 2.781 | 70.22 | 29.78 | 75.68 | 24.32 | B + C |
| III | 5.471 | - | 0.537 | 1.762 | 79.41 | 20.59 | 87.21 | 12.79 | B + C + D |
| | - | - | 4.464 | 0.246 | - | 100.00 | 97.32 | 2.68 | A + C |
| | 0.392 | - | 4.097 | 0.230 | 8.51 | 91.49 | 97.51 | 2.49 | A + C |
| | 0.682 | - | 2.686 | 0.471 | 18.93 | 81.07 | 93.46 | 6.54 | A + C |
| | 1.694 | - | 0.772 | 2.874 | 43.41 | 56.59 | 63.18 | 36.82 | A + C |
| | 2.419 | - | 0.642 | 3.168 | 52.07 | 47.93 | 65.90 | 34.10 | A + C |
| V | 3.545 | - | 3.793 | - | 48.31 | 51.69 | 100.00 | - | C + D |
| | 4.574 | - | 2.102 | 0.701 | 65.09 | 34.91 | 95.01 | 4.99 | C + D |
| VI | 8.92 | - | 0.451 | - | 95.19 | 4.81 | 100.00 | - | D + E |
| | 9.18 | 0.884 | - | 1.145 | 94.38 | 5.62 | 90.05 | 9.95 | D + E |
| VII | 7.49 | 3.425 | - | 0.724 | 96.22 | 3.78 | 78.31 | 21.69 | B + D + E |
| | 5.60 | 7.81 | - | 1.464 | 92.85 | 7.15 | 54.72 | 45.28 | B + E |
| | 3.56 | 10.59 | - | 1.504 | 92.17 | 7.83 | 37.07 | 62.93 | B + E |
| VIII | 3.31 | 12.62 | - | 1.822 | 91.34 | 8.66 | 31.43 | 68.57 | B + E + F |
| | 1.92 | 14.47 | - | 1.278 | 93.48 | 6.52 | 19.63 | 80.37 | B + F |
| IX | - | 20.14 | - | 1.555 | 92.83 | 7.17 | - | 100.00 | B + F |
| X | 2.78 | 15.06 | - | 1.299 | 94.07 | 5.93 | 25.34 | 74.66 | E + F + G |
| XI | 2.41 | 22.79 | - | - | 100.00 | - | 17.47 | 82.53 | E + G |
| XII | - | 24.67 | - | 0.886 | 96.53 | 3.47 | - | 100.00 | F + G |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4.(NH_4)_2CrO_4$; B = $NH_4ClO_4$; C = $(NH_4)_2CrO_4$; D = $NaNH_4CrO_4.2H_2O$; E = $Na_2CrO_4.4H_2O$; F = $n(NH_4ClO_4).m(NaClO_4.H_2O)$; G = $NaClO_4.H_2O$ .

AUXILIARY INFORMATION

SOURCE AND PURITY OF MATERIALS:

Not stated.

ESTIMATED ERROR:

Not stated.

REFERENCES:

(continued next page)

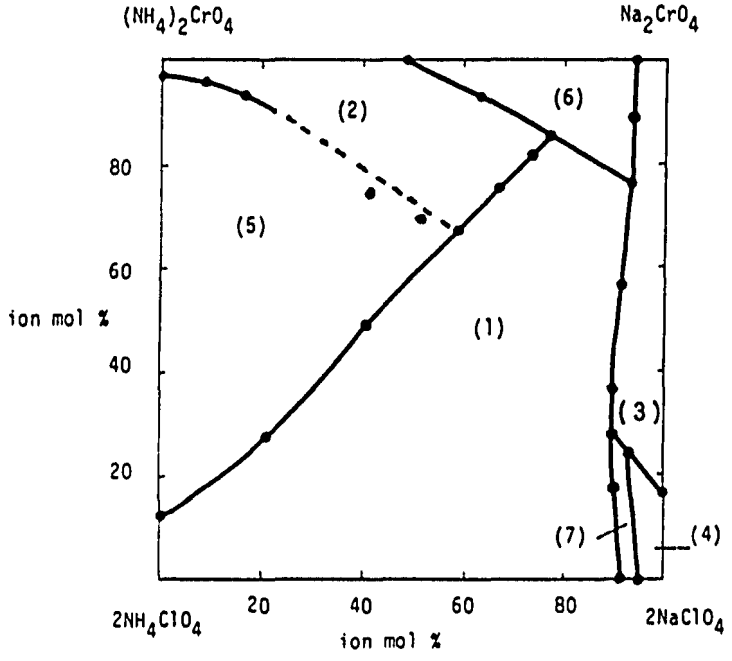
| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chromate; $Na_2CrO_4$; [7775-11-3] | Guseva, A.D.; Druzhinina, G.V. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *78*, 32-8. |
| (3) Ammonium chromate; $(NH_4)_2CrO_4$; [7788-98-9] | |
| (4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

COMMENTS AND/OR ADDITIONAL DATA

The phase diagram given below for this aqueous reciprocal salts solubility system at 308 K shows seven crystallization fields:

(1) $NH_4ClO_4$
(2) $(NH_4)_2CrO_4$
(3) $Na_2CrO_4 \cdot 4H_2O$
(4) $NaClO_4 \cdot H_2O$
(5) $NH_4ClO_4 \cdot (NH_4)_2CrO_4$
(6) $NaNH_4CrO_4 \cdot 2H_2O$
(7) solid solutions formed by sodium and ammonium perchlorates.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium dichromate; $Na_2Cr_2O_7$; [10588-01-9] | Lepeshkov, I.N.; Sal'nikova, L.N. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped.* 1970, *79*, 11-5. |
| (3) Ammonium dichromate; $(NH_4)_2Cr_2O_7$; [7789-09-5] | |
| (4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: Temperature: 298 K. Composition. | PREPARED BY: N.A. Kozyreva |

EXPERIMENTAL VALUES:

Solubility system $2Na^+$, $2NH_4^+$ || $2ClO_4^-$, $Cr_2O_7^{2-}$ - water at 25 °C :

| Point | Liquid phase composition, mass % (1) | (2) | (3) | (4) | molality[a] / mol kg$^{-1}$ (1) | (2) | (3) | (4) | Solid phase[b,c] |
|---|---|---|---|---|---|---|---|---|---|
| P | 3.26 | 64.57 | - | 2.59 | 0.421 | 17.83 | - | 0.745 | A + B + C |
| | 5.08 | 61.58 | - | 2.39 | 0.627 | 16.25 | - | 0.657 | A + B |
| | 7.06 | 61.10 | - | 2.57 | 0.921 | 17.05 | - | 0.747 | A + B |
| | 10.47 | 53.22 | - | 2.83 | 1.194 | 12.98 | - | 0.719 | A + B |
| | 17.62 | 48.43 | - | 3.08 | 2.179 | 12.81 | - | 0.849 | A + B |
| | 22.44 | 37.16 | - | 3.46 | 2.319 | 8.22 | - | 0.797 | A + B |
| | 24.87 | 44.26 | - | 3.26 | 3.438 | 13.09 | - | 1.005 | A + B |
| Q | 32.29 | 38.50 | - | 2.72 | 4.653 | 11.87 | - | 0.874 | A + B + D |
| | 29.66 | 39.82 | - | 3.51 | 4.192 | 12.04 | - | 1.106 | A + D |
| | 39.06 | 28.47 | - | 4.15 | 5.265 | 8.21 | - | 1.247 | A + D |
| R | 44.27 | 24.07 | - | 5.08 | 6.358 | 7.40 | - | 1.627 | A + D + F |
| | 45.17 | 21.52 | - | 5.41 | 6.180 | 6.30 | - | 1.650 | D + E |
| | 52.98 | 14.22 | - | 7.20 | 7.900 | 4.54 | - | 2.394 | D + E |
| | 55.30 | 11.77 | - | 9.31 | 8.937 | 4.07 | - | 3.355 | D + E |
| | 56.77 | 8.81 | - | 7.10 | 7.932 | 2.63 | - | 2.212 | D + E |
| | 62.29 | 3.25 | - | 7.29 | 8.751 | 0.98 | - | 2.284 | D + E |
| | 64.70 | - | 3.42 | 3.51 | 8.706 | - | 0.478 | 1.053 | D + F |
| | 2.10 | - | 20.78 | 12.96 | 0.125 | - | 1.285 | 1.719 | A + E |
| | 5.00 | - | 18.70 | 12.75 | 0.300 | - | 1.167 | 1.708 | A + E |
| | 17.07 | - | 10.88 | 15.05 | 1.143 | - | 0.757 | 2.247 | A + E |
| | 35.23 | - | 0.89 | 16.61 | 2.845 | - | 0.075 | 2.991 | A + E |
| | 39.13 | 7.08 | - | 11.07 | 3.496 | 1.35 | - | 2.206 | A + E |
| | 38.08 | 7.83 | - | 9.96 | 3.294 | 1.45 | - | 1.921 | A + E |
| | 38.67 | 12.41 | - | 8.08 | 3.614 | 2.48 | - | 1.684 | A + E |
| | 39.17 | 14.11 | - | 7.68 | 3.830 | 2.95 | - | 1.674 | A + E |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4$; B = $NaClO_4.H_2O$; C = $nNH_4ClO_4.mNaClO_4.H_2O$; D = $Na_2Cr_2O_7.2H_2O$; E = $n(NH_4)_2Cr_2O_7.mNH_4ClO_4$; F = $(NH_4)_2Cr_2O_7$ .

[c] See critical Evaluation

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Method of "invariant points" used; the third salt is added to the saturated sln. corresponding to the eutonic composition of the ternary | Not stated. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium dichromate; $Na_2Cr_2O_7$; [10588-01-9] | Lepeshkov, I.N.; Sal'nikova, L.N. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped.* 1970, *79*, 11-5. |
| (3) Ammonium dichromate; $(NH_4)_2Cr_2O_7$; [7789-09-5] | |
| (4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

| Point | Liquid phase composition mol %[a] (1) | (2) | (3) | (4) | ion mol %[a] $2Na^+$ | $2NH_4^+$ | $Cr_2O_7^{2-}$ | $2ClO_4^-$ | Solid phase[b,c] |
|---|---|---|---|---|---|---|---|---|---|
| P | 0.565 | 23.93 | - | 1.00 | 96.2 | 3.8 | 4.3 | 95.7 | A + B + C |
| | 0.858 | 22.25 | - | 0.90 | 96.4 | 3.6 | 6.9 | 93.1 | A + B |
| | 1.240 | 22.97 | - | 1.01 | 96.2 | 3.8 | 9.4 | 90.6 | A + B |
| | 1.696 | 18.44 | - | 1.02 | 95.5 | 4.5 | 14.8 | 85.2 | A + B |
| | 3.054 | 17.96 | - | 1.19 | 95.3 | 4.7 | 24.2 | 75.8 | A + B |
| | 3.469 | 12.29 | - | 1.19 | 94.2 | 5.8 | 34.0 | 66.0 | A + B |
| | 4.707 | 17.92 | - | 1.38 | 95.2 | 4.8 | 32.8 | 67.2 | A + B |
| Q | 6.382 | 16.28 | - | 1.20 | 96.0 | 4.0 | 42.2 | 57.8 | A + B + D |
| | 5.754 | 16.53 | - | 1.52 | 94.9 | 5.1 | 38.9 | 61.1 | A + D |
| | 7.497 | 11.69 | - | 1.78 | 93.8 | 6.2 | 52.7 | 47.3 | A + D |
| R | 8.969 | 10.43 | - | 2.29 | 92.5 | 7.5 | 58.5 | 41.5 | A + D + F |
| | 8.875 | 9.05 | - | 2.37 | 91.9 | 8.1 | 60.9 | 39.1 | D + E |
| | 11.231 | 6.45 | - | 3.40 | 89.5 | 10.5 | 69.5 | 30.5 | D + E |
| | 12.435 | 5.66 | - | 4.67 | 86.7 | 13.3 | 70.7 | 29.3 | D + E |
| | 11.616 | 3.86 | - | 3.24 | 89.3 | 10.7 | 76.6 | 23.4 | D + E |
| | 12.961 | 1.45 | - | 3.38 | 89.0 | 11.0 | 84.3 | 15.7 | D + E |
| | 13.241 | 0.00 | 0.727 | 1.60 | 89.7 | 10.3 | 94.6 | 5.4 | D + F |
| | 0.213 | - | 2.19 | 2.93 | 5.5 | 94.5 | 62.1 | 37.9 | A + E |
| | 0.512 | - | 1.99 | 2.91 | 12.9 | 87.1 | 63.2 | 36.8 | A + E |
| | 1.916 | - | 1.27 | 3.77 | 37.8 | 62.2 | 62.8 | 37.2 | A + E |
| | 4.632 | - | 0.122 | 4.87 | 64.4 | 35.6 | 66.1 | 33.9 | A + E |
| | 5.589 | 2.16 | - | 3.53 | 79.1 | 20.9 | 66.3 | 33.7 | A + E |
| | 5.298 | 2.33 | - | 3.09 | 80.7 | 19.3 | 66.2 | 33.8 | A + E |
| | 5.711 | 3.92 | - | 2.66 | 85.2 | 14.8 | 63.4 | 36.6 | A + E |
| | 5.988 | 4.61 | - | 2.62 | 86.4 | 13.6 | 62.3 | 37.7 | A + E |

[a] Compiler's calculations.

[b] A = $NH_4ClO_4$; B = $NaClO_4.H_2O$; C = $nNH_4ClO_4.mNaClO_4.H_2O$;
D = $Na_2Cr_2O_7.2H_2O$; E = $n(NH_4)_2Cr_2O_7.mNH_4ClO_4$; F = $(NH_4)_2Cr_2O_7$ .

[c] See critical Evaluation

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: (continued)

system until a new solid phase appeared. Sodium was determined by precipitation as sodium zinc uranyl acetate; ammonium ion by distillation of ammonia into satd. boric acid sln. and titrating with 0.05 mol $L^{-1}$ $H_2SO_4$; dichromate iodimetrically and perchlorate by difference. Solid phases were analysed chemically and examined under a microscope.

ESTIMATED ERROR:

Not stated.

REFERENCES:

(continued next page)

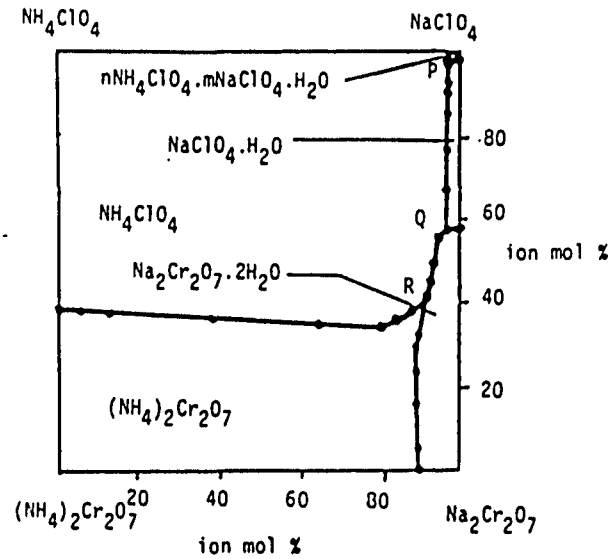
| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium dichromate; $Na_2Cr_2O_7$; [10588-01-9] | Lepeshkov, I.N.; Sal'nikova, L.N. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped.* 1970, *79*, 11-5. |
| (3) Ammonium dichromate; $(NH_4)_2Cr_2O_7$; [7789-09-5] | |
| (4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9] | |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

COMMENTS AND/OR ADDITIONAL DATA

The phase diagram given below shows five crystallization fields: $NH_4ClO_4$; $NaClO_4.H_2O$; $Na_2Cr_2O_7.2H_2O$; $(NH_4)_2Cr_2O_7$ and solid solutions represented by $nNH_4ClO_4.mNaClO_4$.

P, Q and R correspond to isothermal triple saturation points.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Magnesium chloride; $MgCl_2$; [7786-30-3]<br>(4) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8]<br>(5) Water; $H_2O$; [7732-18-5] | Kudryakova, S.A.; Lepeshkhov, I.N.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1969, *66*, 40-50. |
| VARIABLES:<br>One temperature: 363 K<br>Composition | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system : $NaClO_4$-NaCl-$Mg(ClO_4)_2$-$MgCl_2$-$H_2O$ at 90°C

| Liquid phase composition | | | | | | | | Solid Phase[b] |
|---|---|---|---|---|---|---|---|---|
| mass % | | | | mol %[a] | | | | |
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 0.63 | - | 28.93 | 16.17 | 0.317 | - | 8.938 | 2.131 | A + B |
| 0.69 | - | 26.79 | 19.51 | 0.355 | - | 8.467 | 2.630 | A + B |
| 0.69 | - | 25.29 | 22.76 | 0.366 | - | 8.237 | 3.162 | A + B + C |
| 1.14 | - | 19.35 | 31.32 | 0.642 | - | 6.690 | 4.619 | A + C |
| 1.60 | - | 12.98 | 39.38 | 0.945 | - | 4.708 | 6.093 | A + C |
| 3.65 | - | 5.54 | 48.69 | 2.333 | - | 2.174 | 8.149 | A + C |
| 0.54 | 5.62 | - | 52.82 | 0.360 | 1.787 | - | 9.212 | A + C |
| 3.34 | 19.51 | - | 38.19 | 2.241 | 6.248 | - | 6.709 | A + C |
| 2.39 | 23.59 | - | 32.89 | 1.535 | 7.232 | - | 5.531 | A + C + D |
| 2.24 | 25.45 | - | 27.68 | 1.346 | 7.300 | - | 4.355 | A + D |
| 1.85 | 39.20 | - | 23.23 | 1.298 | 13.13 | - | 4.268 | A + D |
| 1.44 | 47.72 | - | 18.77 | 1.081 | 17.10 | - | 3.691 | A + D |
| 1.33 | 52.62 | - | 13.94 | 0.991 | 18.71 | - | 2.719 | A + D |
| 1.22 | 57.46 | - | 9.19 | 0.902 | 20.27 | - | 1.779 | A + D |

[a] Editors' calculations.

[b] A = NaCl; B = $MgCl_2.6H_2O$ ; C = $Mg(ClO_4)_2.6H_2O$ ; D = $NaClO_4$.

AUXILIARY INFORMATION

METHOD/PROCEDURE/APPARATUS:

The isothermal method was used. The solubility was studied by the method of nonvariant points: to the saturated solution corresponding to the eutonic composition of a ternary system, a third salt is added until a new solid phase appears. $Mg^{2+}$ was determined with Trilon B; $Na^+$, by precipitation with zinc uranyl acetate, $ClO_4^-$ gravimetrically by nitron precipitation, and $Cl^-$ mercurimetrically.

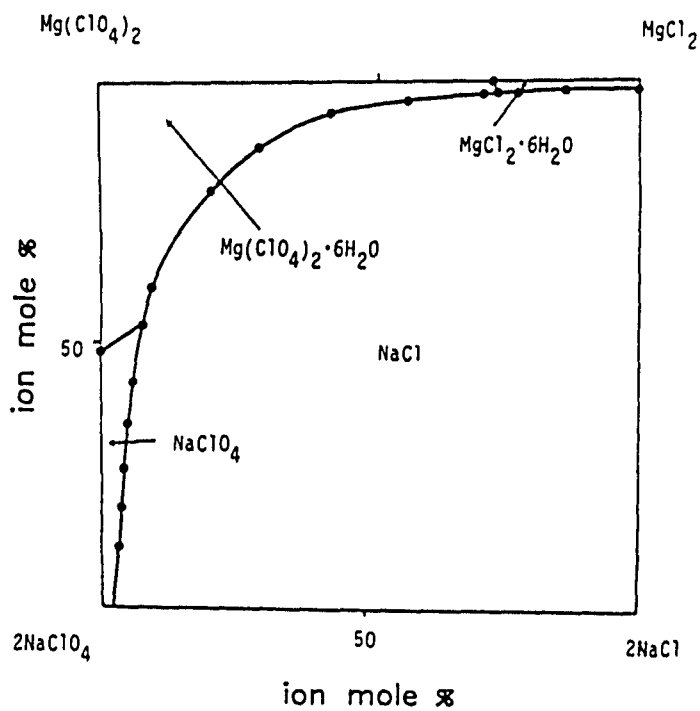
| SOURCE AND PURITY OF MATERIALS: | ESTIMATED ERROR: |
|---|---|
| Not stated. | Not stated. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Magnesium chloride; $MgCl_2$; [7786-30-3]<br>(4) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8]<br>(5) Water; $H_2O$; [7732-18-5] | Kudryakova, S.A.; Lepeshkhov, I.N.<br><br>*Sb. Tr. Yarosl. Gos. Ped. Inst.* 1969, *66*, 40-50. |

EXPERIMENTAL VALUES: (continued)

COMMENTS AND/OR ADDITIONAL DATA:
The Janecke diagram is given below.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5]<br>(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]<br>(3) Barium chloride; $BaCl_2$; [10361-37-2]<br>(4) Barium perchlorate; $Ba(ClO_4)_2$; [13465-95-7]<br>(5) Water; $H_2O$; [7732-18-5] | 1. Zaitseva, S.N.; Karnaukhov, A.S.<br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 107-12.<br>2. Zaitseva, S.N.; Karnaukhov, A.S.<br>*Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *78*, 86-91. |
| VARIABLES:<br>Temperature/K: 298.2 and 323.2<br>Composition | PREPARED BY:<br>E.S. Gryzlova |

EXPERIMENTAL VALUES:

Solubility system : $Na^+, Ba^{2+} // ClO_4^-, Cl^- - H_2O$ at 25.0°C

| Liquid phase composition | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| mass % | | | | mol %[a] | | | | |
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 24.72 | - | 3.12 | - | 9.519 | - | 0.337 | - | A + B |
| 23.38 | 3.16 | 3.04 | - | 9.198 | 0.593 | 0.336 | - | " |
| 19.38 | 12.83 | 2.97 | - | 8.417 | 2.599 | 0.354 | - | " |
| 17.77 | 16.61 | 3.18 | - | 7.755 | 3.460 | 0.389 | - | " |
| 9.00 | 38.06 | 2.62 | - | 4.709 | 9.504 | 0.385 | - | " |
| 6.07 | 43.02 | 2.43 | - | 3.289 | 11.13 | 0.370 | - | " |
| 4.96 | 49.25 | 1.66 | - | 2.882 | 13.66 | 0.271 | - | " |
| 2.26 | 53.08 | 1.24 | - | 1.339 | 15.01 | 0.206 | - | " |
| 1.30 | 61.76 | 1.62 | - | 0.892 | 20.22 | 0.312 | - | A + B + C |
| 1.35 | 66.18 | - | - | 0.976 | 22.85 | - | - | B + C |
| 1.80 | 61.39 | - | 4.14 | 1.306 | 21.26 | - | 0.522 | A + C |
| - | 47.17 | 2.58 | 1.74 | - | 14.45 | 0.400 | 0.167 | A + C + D |
| - | 45.84 | 4.23 | 1.93 | - | 12.22 | 0.663 | 0.187 | A + D |
| - | 38.07 | 5.90 | 1.49 | - | 9.223 | 0.840 | 0.131 | " |
| - | 32.37 | 7.31 | 1.84 | - | 7.444 | 0.989 | 0.154 | " |
| - | 8.63 | 22.11 | 0.25 | - | 1.759 | 2.649 | 0.019 | " |
| - | 1.54 | 23.87 | 1.18 | - | 0.299 | 2.726 | 0.083 | " |
| - | 0.48 | 24.78 | 1.70 | - | 0.094 | 2.845 | 0.121 | " |
| - | - | 26.18 | 1.74 | - | - | 3.043 | 0.125 | " |
| 1.37 | 59.15 | - | 10.11 | 1.082 | 22.29 | - | 1.388 | C + D |
| 1.43 | 44.35 | - | 21.76 | 1.086 | 16.08 | - | 2.872 | " |
| 1.57 | 40.84 | - | 25.75 | 1.219 | 15.13 | - | 3.474 | " |
| 1.52 | 35.67 | - | 34.84 | 1.318 | 14.76 | - | 5.250 | C + D + E |
| - | 41.32 | - | 32.90 | - | 18.08 | - | 5.243 | C + F |
| - | 32.53 | 2.61 | 27.74 | - | 10.97 | 0.518 | 3.407 | D + E |
| - | 28.01 | 1.91 | 32.68 | - | 9.488 | 0.380 | 4.031 | " |
| - | 23.05 | 4.08 | 33.25 | - | 7.512 | 0.782 | 3.946 | " |
| - | 18.42 | 7.84 | 29.84 | - | 5.544 | 1.387 | 3.270 | " |
| - | 8.84 | 6.84 | 33.71 | - | 2.395 | 1.090 | 3.326 | " |
| - | - | 6.78 | 50.82 | - | - | 1.283 | 5.957 | " |

[a] Editors' calculations.

[b] A = $BaCl_2.2H_2O$ ; B = NaCl ; C = $NaClO_4.H_2O$ ; D = n($BaCl_2$).m[$Ba(ClO_4)_2$] ; E = n[$Ba(ClO_4)_2$].m($BaCl_2$) ; F = $Ba(ClO_4)_2.H_2O$.

(continued next page)

COMPONENTS:

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

(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]

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

(4) Barium perchlorate; $Ba(ClO_4)_2$; [13465-95-7]

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

ORIGINAL MEASUREMENTS:

1. Zaitseva, S.N.; Karnaukhov, A.S.

*Uch. Zap. Yarosl. Gos. Ped. Inst.* **1969**, *66*, 107-12.

2. Zaitseva, S.N.; Karnaukhov, A.S.

*Uch. Zap. Yarosl. Gos. Ped. Inst.* **1970**, *78*, 86-91.

EXPERIMENTAL VALUES: (continued)

Solubility system : $Na^+, Ba^{2+}//ClO_4^-, Cl^- - H_2O$ at 50.0°C

| Liquid phase composition | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| mass % | | | | mol %[a] | | | | |
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 23.96 | - | 6.58 | - | 9.541 | - | 0.735 | - | A + B |
| 20.13 | 3.46 | 5.97 | - | 7.989 | 0.655 | 0.665 | - | " |
| 18.30 | 11.29 | 5.25 | - | 7.736 | 2.278 | 0.623 | - | " |
| 15.12 | 16.13 | 5.25 | - | 6.566 | 3.343 | 0.640 | - | " |
| 10.85 | 27.74 | 5.19 | - | 5.218 | 6.368 | 0.701 | - | " |
| 9.35 | 28.49 | 3.65 | - | 4.374 | 6.361 | 0.479 | - | " |
| 6.30 | 40.15 | 3.68 | - | 3.346 | 10.18 | 0.549 | - | " |
| 2.44 | 53.51 | 3.93 | - | 1.532 | 16.04 | 0.693 | - | " |
| 1.53 | 54.26 | 3.62 | - | 0.956 | 16.17 | 0.634 | - | " |
| 0.08 | 61.28 | 3.37 | - | 0.055 | 20.22 | 0.654 | - | " |
| 0.08 | 63.84 | 2.90 | - | 0.058 | 21.92 | 0.585 | - | A + B + C |
| 1.35 | 67.25 | - | 2.91 | 1.068 | 25.40 | - | 0.400 | B + C |
| 1.30 | 69.27 | - | 2.47 | 1.063 | 27.05 | - | 0.351 | " |
| 1.35 | 71.19 | - | 1.27 | 1.120 | 28.20 | - | 0.183 | " |
| 1.37 | 69.00 | - | - | 1.050 | 25.25 | - | - | B + D |
| - | 55.16 | 2.82 | 3.89 | - | 17.38 | 0.522 | 0.446 | C + E + F |
| - | 49.84 | 3.91 | 5.29 | - | 14.99 | 0.692 | 0.579 | A + F |
| - | 41.26 | 5.93 | 3.13 | - | 10.76 | 0.909 | 0.297 | " |
| - | 32.53 | 8.60 | 3.38 | - | 7.821 | 1.216 | 0.296 | " |
| - | 24.39 | 12.36 | 6.15 | - | 5.780 | 1.722 | 0.531 | " |
| - | 17.84 | 16.82 | 6.88 | - | 4.173 | 2.313 | 0.586 | " |
| - | 10.22 | 22.05 | 4.16 | - | 2.238 | 2.838 | 0.332 | " |
| - | 0.75 | 25.19 | 3.97 | - | 0.152 | 3.002 | 0.293 | " |
| - | - | 27.24 | 4.14 | - | - | 3.310 | 0.312 | " |
| - | 61.01 | - | 13.31 | - | 25.38 | - | 2.016 | C + D |
| 1.25 | 45.36 | - | 20.74 | 0.944 | 16.35 | - | 2.722 | C + D + F |
| 1.32 | 35.30 | - | 30.68 | 1.019 | 13.00 | - | 4.115 | D + F |
| - | 36.21 | 2.03 | 37.53 | - | 16.78 | 0.553 | 6.334 | D + F + G |
| - | 35.40 | - | 41.71 | - | 17.17 | - | 7.367 | D + E |
| - | 24.27 | 4.32 | 37.85 | - | 9.033 | 0.945 | 5.130 | F + G |
| - | 11.50 | 4.76 | 49.38 | - | 4.326 | 1.053 | 6.765 | " |
| - | 4.37 | 4.88 | 53.05 | - | 1.545 | 1.015 | 6.831 | " |
| - | 3.99 | 5.20 | 52.90 | - | 1.405 | 1.077 | 6.784 | " |
| - | 5.26 | - | 54.20 | - | 1.750 | - | 6.567 | " |

[a] Editors' calculations.

[b] A = $BaCl_2.2H_2O$ ; B = NaCl ; C = $NaClO_4.H_2O$ ; D = $NaClO_4$ ; E = $BaCl_2.H_2O$
F = $n(BaCl_2).m[Ba(ClO_4)_2]$ ; G = $n[Ba(ClO_4)_2].m(BaCl_2)$.

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5] | 1. Zaitseva, S.N.; Karnaukhov, A.S. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1969, *66*, 107-12. |
| (3) Barium chloride; $BaCl_2$; [10361-37-2] | 2. Zaitseva, S.N.; Karnaukhov, A.S. |
| (4) Barium perchlorate; $Ba(ClO_4)_2$; [13465-95-7] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1970, *78*, 86-91. |
| (5) Water; $H_2O$; [7732-18-5] | |

EXPERIMENTAL VALUES: (continued)

COMMENTS AND/OR ADDITIONAL DATA:

The Janecke diagrams are given below.

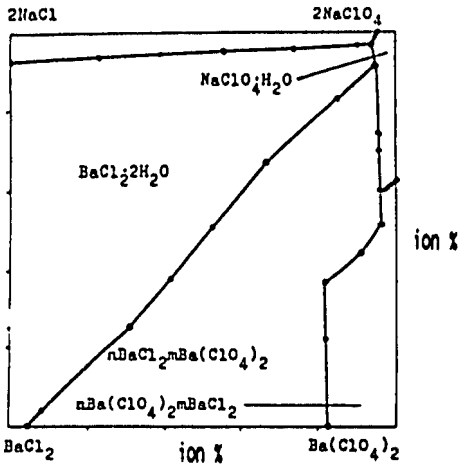



Fig. 1. Solubility isotherm at 25°C

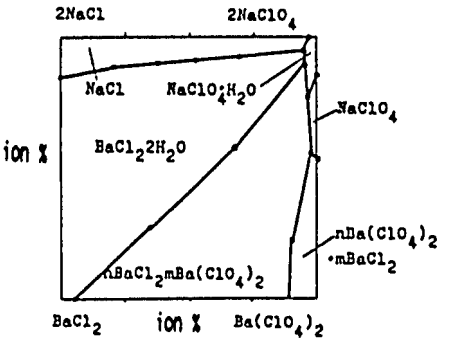



Fig. 2. Solubility isotherm at 50°C

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method of nonvariant points. To the solution corresponding to the transition point of the ternary system a third salt was added until a new solid phase appeared. Conditions of saturation not stated. $Ba^{2+}$ was determined gravimetrically in the presence of picric acid; $Na^+$ gravimetrically as sodium zinc uranyl acetate; $Cl^-$ mercurimetrically; $ClO_4^-$ by difference.

| SOURCE AND PURITY MATERIALS: | ESTIMATED ERROR: |
|---|---|
| The starting salts are of reagent and chemically pure grades were recrystallized twice. | Temp.: ±0.1°C. |
| | REFERENCES: None. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium nitrate; $NaNO_3$; [7631-99-4] | Karnaukhov, A.S.; Tarakanov, V.F. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1972, *103*, 3-6. |
| (3) Nickel nitrate; $Ni(NO_3)_2$; [14216-75-2] | |
| (4) Nickel perchlorate; $Ni(ClO_4)_2$ [13637-71-3] | |
| (5) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 298 K. | E.S. Gryzlova |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $2Na^+$, $Ni^{2+}$ || $2ClO_4^-$, $2NO_3^-$ - water at 25 °C :

| Liquid phase composition | | | | | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| mass % | | | | molality[a] / mol $kg^{-1}$ | | | | |
| (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 11.58 | 58.47 | - | - | 4.549 | 15.945 | - | - | A + B |
| 12.10 | 46.56 | - | 8.94 | 4.394 | 11.737 | - | 1.071 | A + B |
| 12.61 | 38.38 | - | 15.34 | 4.406 | 9.310 | - | 1.769 | A + B |
| 15.06 | 16.36 | - | 32.89 | 4.965 | 3.744 | - | 3.577 | A + B + C |
| 17.11 | 3.92 | - | 41.61 | 5.388 | 0.857 | - | 4.323 | A + C |
| 12.61 | - | 12.54 | 34.82 | 3.706 | - | 1.714 | 3.377 | A + C |
| 8.05 | - | 24.70 | 25.91 | 2.291 | - | 3.270 | 2.433 | A + C + D |
| 8.03 | - | 37.93 | 9.85 | 2.138 | - | 4.698 | 0.865 | A + D |
| 8.96 | - | 44.87 | - | 2.283 | - | 5.319 | - | A + D |
| 5.18 | - | 24.87 | 27.18 | 1.425 | - | 3.182 | 2.467 | C + D |
| - | - | 28.19 | 25.89 | - | - | 3.360 | 2.189 | C + D |
| 9.74 | 24.71 | - | 32.11 | 3.427 | 6.035 | - | 3.727 | B + C |
| - | 31.09 | - | 29.42 | - | 6.430 | - | 2.892 | B + C |

(continued next page)

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| To the satd. solutions corresponding to the compositions of the nodal points (in the presence of an excess of solid phases) a third salt was added until a new solid phase appeared. Nitrate was determined using Devarda's method; perchlorate by gravimetric method using nitron precipitation; nickel by complexometric titration with Trilon B. The solid phases were examined under a microscope. | Not stated. |
| | **ESTIMATED ERROR:** |
| | Not stated. |
| | **REFERENCES:** |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium nitrate; $NaNO_3$; [7631-99-4] | Karnaukhov, A.S.; Tarakanov, V.F. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Uch. Zap. Yarosl. Gos. Ped. Inst.* 1972, *103*, 3-6. |
| (3) Nickel nitrate; $Ni(NO_3)_2$; [14216-75-2] | |
| (4) Nickel perchlorate; $Ni(ClO_4)_2$ [13637-71-3] | |
| (5) Water; $H_2O$; [7732-18-5] | |

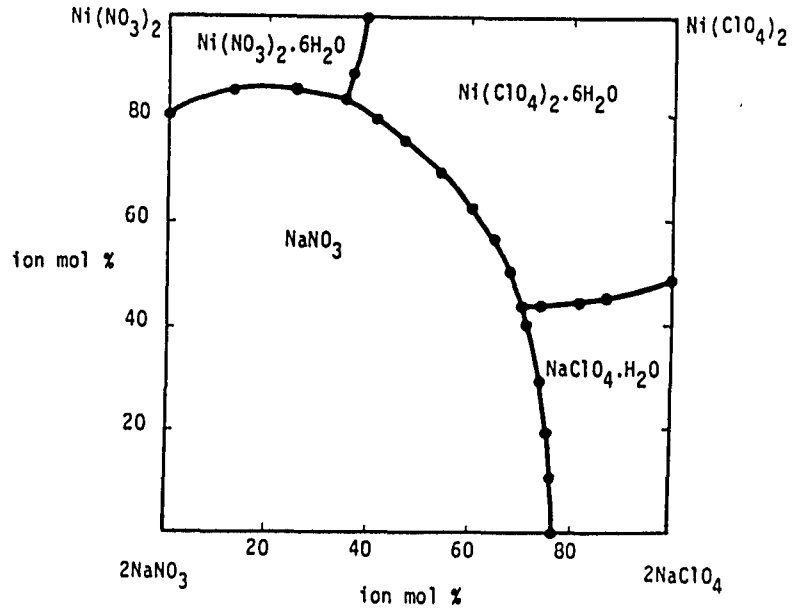
EXPERIMENTAL VALUES: (continued)

| mol % [a] | | | | ion mol % [a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|
| | | | | Cation | | Anion | | |
| (1) | (2) | (3) | (4) | $2Na^+$ | $Ni^{2+}$ | $2NO_3^-$ | $2ClO_4^-$ | |
| 5.99 | 20.98 | - | - | 100.0 | - | 22.2 | 77.8 | A + B |
| 6.04 | 16.14 | - | 1.473 | 88.3 | 11.7 | 24.0 | 76.0 | A + B |
| 6.21 | 13.11 | - | 2.491 | 79.5 | 20.5 | 25.5 | 74.5 | A + B |
| 7.32 | 5.52 | - | 5.277 | 54.9 | 45.1 | 31.3 | 68.7 | A + B + C |
| 8.15 | 1.30 | - | 6.543 | 41.9 | 58.1 | 36.2 | 63.8 | A + C |
| 5.76 | - | 2.666 | 5.251 | 26.7 | 73.3 | 51.4 | 48.6 | A + C |
| 3.61 | - | 5.149 | 3.831 | 16.7 | 83.3 | 64.5 | 35.5 | A + C + D |
| 3.38 | - | 7.432 | 1.369 | 16.1 | 83.9 | 87.0 | 13.0 | A + D |
| 3.62 | - | 8.428 | - | 17.7 | 82.3 | 100.0 | 0.0 | A + D |
| 2.28 | - | 5.085 | 3.942 | 11.2 | 88.8 | 61.2 | 38.8 | C + D |
| - | - | 5.503 | 3.585 | - | 100.0 | 60.6 | 39.4 | C + D |
| 4.99 | 8.78 | - | 5.426 | 55.9 | 44.1 | 20.3 | 79.7 | B + C |
| - | 9.92 | - | 4.461 | 52.6 | 47.4 | - | 100.0 | B + C |

[a] Compiler's calculations

[b] A = $NaNO_3$ B = $NaClO_4$ C = $Ni(ClO_4)_2.6H_2O$
D = $Ni(NO_3)_2.6H_2O$

COMMENTS AND/OR ADDITIONAL DATA The phase diagram given below shows four crystallization fields: anhydrous $NaNO_3$, $Ni(NO_3)_2.6H_2O$, $Ni(ClO_4)_2.H_2O$, and $NaClO_4.H_2O$.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium sulfate; $Na_2SO_4$; [7757-82-6] | Karnaukhov, A.S.; Leboschina, V.I. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Tr. Yarosl. Gos. Ped. Inst.* 1979, *178*, 24-7. |
| (3) Zinc sulfate; $ZnSO_4$; [7733-02-0] | |
| (4) Zinc perchlorate; $Zn(ClO_4)_2$ [13637-61-1] | |
| (5) Water; $H_2O$; [7732-18-5] | |

| VARIABLES: | PREPARED BY: |
|---|---|
| Temperature: 298 K. | I.S. Bodnya |
| Composition. | |

EXPERIMENTAL VALUES:

Solubility system $2Na^+, Zn^{2+} \,||\, 2ClO_4^-, SO_4^{2-}$ - water at 25 °C :

| Point | Liquid phase composition, mass % | | | | Liquid phase composition, molality[a] / mol kg$^{-1}$ | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | (1) | (2) | (3) | (4) | |
| 1 | 13.19 | 34.35 | - | - | 1.770 | 5.35 | - | - | A + B |
| I | 4.43 | 36.90 | - | 4.24 | 0.573 | 5.54 | - | 0.295 | A + B |
| VII | 0.82 | 41.02 | - | 6.34 | 0.111 | 6.47 | - | 0.463 | A + B + D |
| 2 | 0.58 | 49.87 | - | 2.58 | 0.087 | 8.67 | - | 0.208 | A + D |
| II | 0.79 | 67.42 | - | - | 0.175 | 17.32 | - | - | A + D |
| 3 | 0.59 | 42.70 | - | 10.38 | 0.090 | 7.53 | - | 0.848 | B + D |
| 4 | 0.60 | 31.73 | - | 14.34 | 0.079 | 4.86 | - | 1.017 | B + D |
| 5 | - | 28.29 | - | 23.47 | - | 4.79 | - | 1.841 | B + D |
| VIII | - | 26.62 | 0.60 | 32.96 | - | 5.46 | 0.093 | 3.132 | B + D + E |
| III | - | 24.92 | - | 35.39 | - | 5.13 | - | 3.374 | D + E |
| IX | 0.49 | - | 0.12 | 50.70 | 0.071 | - | 0.015 | 3.940 | B + E + F |
| IV | - | - | 0.55 | 53.43 | - | - | 0.074 | 4.393 | E + F |
| 6 | 2.50 | - | 0.22 | 34.45 | 0.280 | - | 0.022 | 2.075 | B + F |
| 7 | 6.14 | - | 2.71 | 27.33 | 0.677 | - | 0.263 | 1.620 | B + F |
| X | 7.42 | - | 8.24 | 21.00 | 0.825 | - | 0.806 | 1.255 | B + F + G |
| 8 | 11.75 | - | 11.61 | 11.88 | 1.277 | - | 1.111 | 0.694 | B + G |
| V | 15.63 | - | 17.54 | - | 1.647 | - | 1.626 | - | B + G |
| 9 | 0.16 | 4.93 | 32.25 | - | 0.018 | 0.643 | 3.188 | - | F + G |
| VI | 4.40 | - | 34.35 | - | 0.506 | - | 3.474 | - | F + G |

[a] Compiler's calculations

[b] A = $Na_2SO_4$; B = $Na_2SO_4.10H_2O$; C = $Na_2SO_4.H_2O$; D = $NaClO_4.H_2O$; E = $Zn(ClO_4)_2.6H_2O$; F = $ZnSO_4.7H_2O$; G = $Na_2SO_4.ZnSO_4.4H_2O$ .

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Method of "invariant points" used. $ClO_4^-$ was determined gravimetrically with nitron; $SO_4^{2-}$ by precipitation as barium sulfate; $Zn^{2+}$ by complexometric titration with Eriochrome Black as indicator; $Na^+$ by difference. | Not stated. |
| | ESTIMATED ERROR: Not stated. |

(continued next page)

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium sulfate; $Na_2SO_4$; [7757-82-6] | Karnaukhov, A.S.; Leboschina, V.I. |
| (2) Sodium perchlorate; $NaClO_4$; [7601-89-0] | *Tr. Yarosl. Gos. Ped. Inst.* 1979, *178*, 24-7. |
| (3) Zinc sulfate; $ZnSO_4$; [7733-02-0] | |
| (4) Zinc perchlorate; $Zn(ClO_4)_2$ [13637-61-1] | |
| (5) Water; $H_2O$; [7732-18-5] | |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 298 K. | I.S. Bodnya |
| Composition. | |

EXPERIMENTAL VALUES:

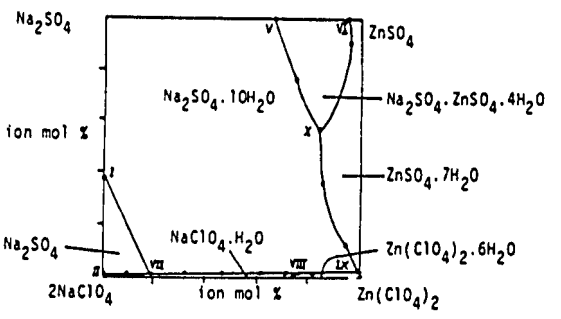
| Point | Liquid phase composition, mol %[a] | | | | ion mol %[a] | | | | Solid phase[b] |
|---|---|---|---|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) | $2Na^+$ | $Zn^{2+}$ | $2ClO_4^-$ | $SO_4^{2-}$ | |
| 1 | 2.826 | 8.54 | - | - | 100.0 | - | 60.2 | 39.8 | A + B |
| I | 0.925 | 8.94 | - | 0.476 | 91.9 | 8.1 | 84.2 | 15.8 | A + B |
| VII | 0.178 | 10.34 | - | 0.740 | 87.8 | 12.2 | 97.1 | 2.9 | A + B + D |
| 2 | 0.135 | 13.45 | - | 0.322 | 95.5 | 4.5 | 98.1 | 1.9 | A + D |
| II | 0.240 | 23.73 | - | - | 100.0 | - | 98.0 | 2.0 | A + D |
| 3 | 0.140 | 11.77 | - | 1.325 | 82.0 | 18.0 | 98.1 | 1.9 | B + D |
| 4 | 0.129 | 7.91 | - | 1.655 | 71.1 | 28.9 | 97.8 | 2.2 | B + D |
| 5 | - | 7.71 | - | 2.963 | 56.5 | 43.5 | 100.0 | - | B + D |
| VIII | - | 8.51 | 0.145 | 4.879 | 45.8 | 54.2 | 98.4 | 1.6 | B + D + E |
| III | - | 8.01 | - | 5.271 | 43.2 | 56.8 | 100.0 | 0.0 | D + E |
| IX | 0.119 | - | 0.026 | 6.618 | 1.8 | 98.2 | 97.9 | 2.1 | B + E + F |
| IV | - | - | 0.123 | 7.325 | - | 100.0 | 98.3 | 1.7 | E + F |
| 6 | 0.484 | - | 0.037 | 3.584 | 11.8 | 88.2 | 87.3 | 12.7 | B + F |
| 7 | 1.166 | - | 0.453 | 2.791 | 26.4 | 73.6 | 63.3 | 36.7 | B + F |
| X | 1.412 | - | 1.380 | 2.148 | 28.6 | 71.4 | 43.5 | 56.5 | B + F + G |
| 8 | 2.180 | - | 1.895 | 1.185 | 41.4 | 58.6 | 22.5 | 77.5 | B + G |
| V | 2.801 | - | 2.766 | - | 50.3 | 49.7 | - | 100.0 | B + G |
| 9 | 0.030 | 1.083 | 5.371 | - | 9.6 | 90.4 | 9.1 | 90.9 | F + G |
| VI | 0.850 | - | 5.840 | - | 12.7 | 87.3 | - | 100.0 | F + G |

[a] Compiler's calculations

[b] A = $Na_2SO_4$; B = $Na_2SO_4.10H_2O$; C = $Na_2SO_4.H_2O$; D = $NaClO_4.H_2O$; E = $Zn(ClO_4)_2.6H_2O$; F = $ZnSO_4.7H_2O$; G = $Na_2SO_4.ZnSO_4.4H_2O$ .

COMMENTS AND/OR ADDITIONAL DATA

The phase diagram given below shows six crystallization fields: $Na_2SO_4.10H_2O$ (77.06%); $ZnSO_4.7H_2O$ (9.45%); $Na_2SO_4$ (3.88%); $Na_2SO_4.ZnSO_4.4H_2O$ (7.33%); $NaClO_4.H_2O$ (1.59%); $Zn(ClO_4)_2.6H_2O$ (0.69%). VII, VIII, IX and X' are isothermal triple saturation points.



| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Sodium chloride; NaCl; [7647-14-5]<br>(2) Sodium percnlorate; $NaClO_4$; [7601-89-07]<br>(3) Ammonium chloride; $NH_4Cl$; [12125-02-9]<br>(4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]<br>(5) Ammonia; $NH_3$; [7664-41-7] | Marshall, P.R.; Hunt, H.<br><br>*J. Chem. Eng. Data* 1959, *4*, 217-22. |
| VARIABLES:<br>Temperature: 240 - 323 K.<br>Composition | PREPARED BY:<br>C.Y. Chan |

EXPERIMENTAL VALUES:

Solubility system $Na^+, NH_4^+ \,||\, Cl^-, ClO_4^-, -H_2O$ at various temperatures :

| | Liquid phase composition | | | | | | | | Solid phase[c] |
|---|---|---|---|---|---|---|---|---|---|
| t/ °C | molality/ mol $kg^{-1}$ | | | | 100X(cation)[a] | | 100X(anion)[b] | | |
| | $Na^+$ | $NH_4^+$ | $Cl^-$ | $ClO_4^-$ | $Na^+$ | $NH_4^+$ | $Cl^-$ | $ClO_4^-$ [d] | |
| -33 | 0.78 | 12.39 | 2.1 | 11.1 | 5.9 | 94.1[d] | 15.8 | 84.2 | (1)+(3)+(4) |
| 0 | 0.32 | 20.51 | 0.88 | 19.95 | 1.54 | 98.46[d] | 4.22 | 95.78 | " " " |
| 25 | 0.76 | 19.15 | 1.4 | 18.5 | 3.84 | 96.16[d] | 7.0 | 93.0 | " " " |
| 50 | 0.58 | 23.9 | 1.49 | 23.0 | 2.36 | 97.64[d] | 6.08 | 93.92 | " " " |

(table continued next page)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility determinations were carried out using a specially constructed apparatus (diagram given in original paper), involving a gas line connected to the saturation cell. The cell consisted of two compartments separated by a sintered glass partition, the larger one of which was connected to the gas line in such a way that the cell could be inverted, with either one of the compartments vertically above the other. Weighed amts of the salts were sealed in the smaller compartment of the cell which was then connected to the gas line via the larger compartment. Excess of dry ammonia was condensed in the cell until the salts had all dissolved at the set temperature. The coolants used were dry ice and $CCl_4$. The cell was thermostated in a liquid $NH_3$ bath for -33 °C determinations, in an ice + water bath for 0 °C, and in a water bath for the other temperatures. Ammonia was bled from the solution until salt

(continued next page)

COMPONENTS:

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

(2) Sodium perchlorate; $NaClO_4$; [7601-89-07]

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

(4) Ammonium perchlorate; $NH_4ClO_4$; [7790-98-9]

(5) Ammonia; $NH_3$; [7664-41-7]

ORIGINAL MEASUREMENTS:

Marshall, P.R.; Hunt, H.

*J. Chem. Eng. Data* 1959, *4*, 217-22.

EXPERIMENTAL VALUES: (continued)

Solubility system $Na^+, NH_4^+ \,||\, Cl^-, ClO_4^-, -H_2O$ at various temperatures :

| | Liquid phase composition | | | | | | | | Solid phase[c] |
|---|---|---|---|---|---|---|---|---|---|
| t/ °C | molality/ mol $kg^{-1}$ | | | | 100X(cation)[a] | | 100X(anion)[b] | | |
| | $Na^+$ | $NH_4^+$ | $Cl^-$ | $ClO_4^-$ | $Na^+$ | $NH_4^+$ | $Cl^-$ | $ClO_4^-$ | |
| -33 | 17.4 | 3.06 | - | 20.46 | 85.0[d] | 15.0 | - | 100.0 | (1)+(2)+(4) |
| 0 | 19.45 | 4 1 | 0.086 | 23.33 | 95.9[d] | 4.1 | 0.366 | 99.6 | " " " |
| 25 | 19.4 | 3.53 | - | 22.93 | 96.47[d] | 3.53 | - | 100.0 | " " " |
| 50 | 25.1 | 4.74 | - | 29.84 | 84.1[d] | 15.9 | - | 100.0 | " " " |

[a] $X(Na^+) = n(Na^+)/[n(Na^+) + n(NH_4^+)]$; $X(NH_4^+) = 1 - X(Na^+)$ ;

[b] $X(ClO_4^-) = n(ClO_4^-)/[n(ClO_4^-) + n(Cl^-)]$; $X(Cl^-) = 1 - X(ClO_4^-)$

$n(\ )$ = amount of substance.

[c] All components anhydrous.

[d] Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

crystals were formed, and the cell inverted so that the solution filtered through the partition into the larger compartment. After filtration the ammonia in the solution was all removed by condensation into a reservoir in the apparatus and determined quantitatively by absorption in std. HCl sln and back-titrated with std. base. The cell was then opened and the solids removed for analysis. Ammonium ion was determined by a standard Kjeldahl procedure. Chloride was determined by titration with $AgNO_3$, using dichlorofluorescein as indicator.

SOURCE AND PURITY OF MATERIALS:

Not stated. Ammonia was dried with sodium.

ESTIMATED ERROR:

Reproducibility (3 detn) is within ± 2 % of the mean value in most cases.

SYSTEM INDEX

Page numbers preceded by E refer to evaluation text whereas those not preceded by E refer to compiled tables.

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REGISTRY NUMBER INDEX

Page numbers preceded by E refer to evaluation text whereas those not preceded by E refer to compiled tables.

In the indexes of this volume, the names used in compilation sheets have been used, but it should be noted that Chemical Abstracts list salts as derivatives of the appropriate acid, for example, sodium dichromate is listed as chromic acid, disodium salt, $Cr_2H_2O_7.2Na$; manganese perchlorate is listed as perchloric acid, manganese(2+) salt, $ClHO_4.1/2Mn$.

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| [75-64-9] | $C_4H_{11}N$ | 2-methyl-2-propanamine | E7, 30, 31 |
| [75-65-0] | $C_4H_{10}O$ | 2-methyl-2-propanol | E7, 28, 29 |
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| [108-91-8] | $C_6H_7N$ | Cyclohexanamine | E8, 30, 31 |
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| [109-73-9] | $C_4H_{11}N$ | 1-butanamine | E7, 30, 31 |
| [109-99-9] | $C_4H_8O$ | Tetrahydrofurane | E7, 38, 40 |
| [110-40-7] | $C_{14}H_{26}O_4$ | Diethyl decanedioate | E8, 36, 37 |
| [110-80-5] | $C_4H_{10}O_2$ | 2-ethoxyethanol | E7, 33, 34 |
| [110-86-1] | $C_5H_5N$ | Pyridine | E7, 30, 31 |

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| [111-87-5] | $C_8H_{18}O$ | 1-octanol | E8, 28, 29 |
| [111-92-2] | $C_8H_{19}N$ | N-butyl-1-butanamine | E8, 30, 31 |
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| [7601-90-3] | $HClO_4$ | Perchloric acid | E8, 45,E111, E115, 143, 159-161 |
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| [7732-18-5] | $H_2O$ | Water | E2-E6, E10-E19, 23-25, 51-63, 67, 103, E104-E110, |

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| [7775-11-3] | $Na_2CrO_4$ | Sodium chromate | E114, E115, E117, 197-199, 217-219, 232-236 |
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| [7783-20-2] | $(NH_4)_2SO_4$ | Ammonium sulfate | E117, 228-231 |
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| [7789-00-6] | $K_2CrO_4$ | Potassium chromate | E18, 96, 97, E11, 214-216 |
| [7789-09-5] | $(NH_4)_2Cr_2O_7$ | Ammonium dichromate | E117, 237-239 |
| [7789-20-0] | $D_2O$ | water-$d_2$ | E111, 128 |
| [7790-69-4] | $LiNO_3$ | Lithium nitrate | E15, E16, 84-86 |
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| [7791-03-9] | $LiClO_4$ | Lithium perchlorate | E1-E22 |
| [10034-81-8] | $Mg(ClO_4)_2$ | Magnesium perchlorate | E17, E19, 90-92, 102, 103, E113, E118, 172-174, 240, 241 |
| [10361-37-2] | $BaCl_2$ | Barium chloride | E118, 242-244 |
| [10588-01-9] | $Na_2Cr_2O_7$ | Sodium dichromate | E115, E117, 200, 237-239 |
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| [13450-97-0] | $Sr(ClO_4)_2$ | Strontium perchlorate | E113, 178 |
| [13453-40-2] | $TlClO_4$ | Thallium perchlorate | E12, E13, 73, E113, 171 |

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