

# INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

## *Analytical Chemistry Division*

### *SUBCOMMITTEE ON SOLUBILITY AND EQUILIBRIUM DATA*

#### **Minutes**

**7<sup>th</sup> Annual Meeting of SSED (34<sup>th</sup> overall)**  
held in conjunction  
with the 13<sup>th</sup> ISSP at Trinity College Dublin  
Dublin, Ireland  
26<sup>th</sup> and 27<sup>th</sup> July 2008

The "List of Attendees" with complete addresses, telephone and fax numbers together with e-mail addresses is attached to these minutes (Appendix 1)

#### **Note added following the Dublin Meeting.**

Professor Dana Knox passed away tragically on September 24, 2008.

Professor Knox was a long time member of the SSED and the IUPAC Solubility Data Commission before that. He will be greatly missed by members of the Subcommittee.

Professor Knox was Chairman of the SSED and organizer of the 14<sup>th</sup> ISSP, to be held at the New Jersey Institute of Technology.

Following discussion, officers of the SSED nominated Prof. Clara Magalhães, University of Aveiro, Department of Chemistry, Portugal, as Chairman of the SSED.

It was also decided to move the 14<sup>th</sup> ISSP to the University of Leoben, Austria. The proposed dates for the ISSP are July 25 - 30, 2010.

#### **Saturday, July 26, 2008**

##### **Morning Session, Day 1 (9:00 - 12:00)**

1. Introduction of participants

D. Knox

Dana Knox, Chairman of the SSED welcomed the participants. All of the

- participants were requested to introduce themselves together with their affiliation
2. Approval of Minutes of the 33<sup>rd</sup> Annual Meeting (6<sup>th</sup> of SSED) in conjunction with the 44<sup>th</sup> General Assembly of IUPAC at the Università di Torino, Torino, Italy held on 6 August 2007 R. Tomkins, D. Knox

The minutes had been distributed by e-mail prior to the meeting and also distributed as hard copies at the meeting. The minutes were approved as presented
  3. Other Items for Agenda E. Waghorne

Heinz Gamsjaeger explained that Monatshefte fur Chemie was prepared to publish articles based on presentations made at the 13<sup>th</sup> ISSP and that these could include colour illustrations.

Following the Chairman's report of the ADC meeting the issue of traceability was added to the agenda as item 9a
  4. Franzosini Award D. Knox

No award was made for 2008.

It was noted that details of any nominee for the award should be available to the SSED prior to the annual meeting.

Action Item

It was decided to explore having an article about the Franzosini award prepared for CI
  5. Chairman's Report for 2007 - 2008 D. Knox

The Chairman reported on the meeting of the ADC held 29 Feb. and 1 Mar. 2008 in Rome, Italy. These minutes are available

There was a discussion of the ADC decision to develop metrological traceability chains for some simple solubility measurements.
  6. SSED officers D. Knox

Subcommittee on Solubility and Equilibrium Data (2008-2009)

Prof. Dana E. Knox (Chairman g-liq Chair)  
 PD Dr. Wolfgang Hummel (link to environmental applications)  
 Prof. Earle W. Waghorne (Secretary)  
 Prof. Erich Königsberger (link to industrial applications)  
 Prof. John W. Lorimer (Terminology, Nomenclature and Symbols)  
 Dr. Mark Salomon (EIC)  
 Prof. David G. Shaw (liq-liq Chair)  
 Prof. Wolfgang Voigt (sol-liq Chair)

David Shaw raised the issue of SSED membership. Following discussion a restructuring was proposed wherein all members of task groups would be members of the SSED and there would then be an executive or advisory committee within the SSED
  7. Editor-in-Chief's Report for 2007 - 2008 M. Salomon

Mark Salomon presented the Editor-in-Chief's Report; this is attached as appendix 2. There was a discussion of several technical issues regarding publication.
  8. Publishing arrangements with JPCRD M. Salomon, A. Harvey

In the absence of Allan Harvey, Mark Salomon made a presentation regarding

JPCRD; this is attached as appendix 3

9. Preliminary discussion of how to encourage equilibrium-oriented projects and possible formation of an Equilibrium Subcommittee

E. Waghorne, H. Gamsjäger

Two possible projects were discussed:

Prof. Hefter described a project, in collaboration with Prof. Powell on the environmental impact of the "heavy metals" Hg, Cu and Pb complexes. The project is well advanced. A follow-up project on the Cd and Zn systems was suggested; this will require recruitment of contributors.

Prof. Lorimer expressed concern about the use of the term "heavy metals"

Prof. Hefter passed on greetings to the SSED from Prof. Marie Therese Cohen-Adad.

James Sangster proposed an ongoing project to maintain the database of octanol/water partition coefficients ( $K_{ow}$ ) that he has developed. The web-site for access to the database is: <http://logkow.cisti.nrc.ca/logkow/>.

Action Item

Dr. Sangster agreed to prepare a project proposal and to circulate it within the SSED.

- 9a Traceability in Solubility measurements

The ACD has requested input to its project on traceability (see:

[http://www.iupac.org/publications/ci/2003/2502/pp3\\_2001-010-3-500.html](http://www.iupac.org/publications/ci/2003/2502/pp3_2001-010-3-500.html)

Action Item

It was agreed to provide a suitable example to ACD.

J. Lorimer?

**Sunday, July 27, 2008**

**Morning Session, Day 2 9:00 - 1:00**

11. Reports of Subcommittees and Discussions

Subcommittee: Gas Solubilities

D. Knox (Chair)

Due to Prof. Knox's untimely death, no written report was received

Subcommittee: Liquid Solubilities

D. Shaw (Chair)

Dr. Shaw provided a written report which is attached as appendix 4

Subcommittee: Solid Solubilities

W. Voigt (Chair)

Prof. Voigt provided a written report which is attached as appendix 5

Subcommittee: Equilibrium (?)

??? (Chair)

No written report was received.

12. Volumes for next year's SDS proposals

M. Salomon

The current state of volumes, including those for publication in 2008 - 9 is included in the reports of the subcommittee chairs.

In addition the Dr. Salomon listed suggested volumes on:

(i) Benzoates - to be discussed further with Prof. Voigt.

(ii) Beryllium compounds - suggested by Prof. Hala

(iii) An update volume on gas solubilities - suggested by Prof. Clever

13. Status of NIST funding

H. Gamsjäger, D. Knox

It was reported that the status of the NIST funding had not changed and that this covers two volumes per year over the next three years. Renewal of this funding beyond that has not been agreed.

Funding is available from IUPAC, through ACD, for individual projects that have targeted audiences.

Prof. Gamsjaeger presented a summary of project funding which is attached as appendix 6

14. Report of Glossary Task Group

J.W. Lorimer

Prof. Lorimer reported that the "Glossary of Terms Related to Solubility" was published in PAC 80[2], 233-276 (2008), and is available free on the IUPAC web site under Publications - Pure & Applied Chemistry; the URL is <http://media.iupac.org/publications/pac/2008/pdf/8002x0233.pdf>.

Copies were distributed to members of the SSED.

15. Report on the 13<sup>th</sup> ISSP - Dublin, Ireland, 2008

E. Waghorne

Prof. Waghorne presented a brief overview of the meeting and thanked SSED members for their assistance.

16. Future International Symposia on Solubility Phenomena

Two proposals to hold the 14<sup>th</sup> ISSP in 2010 were received:

- (i) St. Petersburg State University with Prof. Toikka as organizer. The preferred location for the meeting was conference hotel at Repino although, at extra cost, it could be organized in or nearer the city.
- (ii) New Jersey Institute of Technology with Prof. Knox as organizer. The meeting would be held on the NJIT campus and make use of the Institute's conference facilities

Following presentations of the two proposals and discussion the subcommittee voted 13 to 7 to accept the NJIT proposal.

17. Adjournment

Appendix 1  
List of Attendees

## Attendees at the Meeting

### **BALAREW, Prof. Christo**

Institute of General and Inorganic Chemistry, Bulgarian Academy of Sciences, Acad. G. Bonchevstr Bl 11, 1113 Sofia Bulgaria  
Tel: +359 (2) 979-3925  
Fax: +359 (2) 870-5024  
Email: [balarew@svr.igic.bas.bg](mailto:balarew@svr.igic.bas.bg)

### **De VISSER, Dr. Alex**

University of Calgary  
Dept. Of Chemistry and Petroleum Engineering  
2500 University Drive NW  
Calgary, Alberta, Canada T2N 1N4  
Tel: +1 (403) 220-4739  
Email: [adevisse@ucalgary.ca](mailto:adevisse@ucalgary.ca)

### **EYSSELTOVA, Dr. Jitka**

Department of Inorganic Chemistry, Charles University, Faculty of Science, Albertov 2030  
Prague 12840, Czech Republic  
Tel: 420 (2) 8198-2637  
Email: [jeys@seznam.cz](mailto:jeys@seznam.cz)

### **FOGG, Dr. Peter**

Silver Birches, High Molewood, Hartford Hertford, SG14 2PL  
United Kingdom TEL: +[44] (1992) 581092  
FAX: + 44 (1992) 504985  
E-MAIL: [pandhfogg@tiscali.co.uk](mailto:pandhfogg@tiscali.co.uk)

### **GAMSJÄGER, Prof. Heinz**

Lehrstuhl für Physikalische Chemie, Montanuniversität, Leoben, Franz-Josef-Strasse 18, Leoben  
8700, Austria  
Tel: +43 (3842) 402-4804  
Fax: +43 (3842) 402-4802  
Email: [gamsjaeg@unileoben.ac.at](mailto:gamsjaeg@unileoben.ac.at)

### **GOTO, Dr. Ayako**

4-16-13 Koishikawa, Bunkyo-ku, 112-0002, Tokyo-to, Japan  
Email: [Aya510@aol.com](mailto:Aya510@aol.com)

### **GOTO, Prof. Emeritus Rensuke**

Bunkyo-Ku, Kaishikawa, Koisluka Tower 1005, Tokyo, Japan  
Email: [Rengoto@aol.com](mailto:Rengoto@aol.com)

### **GUMINSKI, Dr. Cezary**

Department of Chemistry, University of Warsaw, Pasteura 1, 02093 Warszawa, Poland  
Tel: +48 (22) 8220211, ext. 331  
Fax: +48 (22) 8225996  
Email: [cegie@chem.uw.edu.pl](mailto:cegie@chem.uw.edu.pl)

### **HERMANN, Ms Meg**

Tel: +1 (303) 998-1406

Email: [Megzpilgrim@yahoo.com](mailto:Megzpilgrim@yahoo.com)

**HEFTER, Prof. Glenn**

School of Chemical and Mathematical Sciences, Murdoch University, Murdoch, WA 6150, Australia

Email: [g.hefter@murdoch.edu.au](mailto:g.hefter@murdoch.edu.au)

**HUMMEL, Dr. Wolfgang**

Paul Scherrer Institut, Waste Management Laboratory, CH-5232 Villigen PSI, Switzerland

Email: [Wolfgang.Hummel@psi.ch](mailto:Wolfgang.Hummel@psi.ch)

**KNOX, Prof. Dana,**

Office of the Provost, New Jersey Institute of Technology, Newark NJ 07102, USA

Tel: +1 (973) 596-5287

Email: [knoxd@njit.edu](mailto:knoxd@njit.edu)

**KOENIGSBERGER, Prof. Erich**

School of Chemical and Mathematical Sciences, Murdoch University, Murdoch, WA 6150, Australia

Email: [E.Koenigsberger@murdoch.edu.au](mailto:E.Koenigsberger@murdoch.edu.au)

**KOENIGSBERGER, Dr Lan Chi**

School of Chemical and Mathematical Sciences, Murdoch University, Murdoch, WA 6150, Australia

Email: [L.Koenigsberger@murdoch.edu.au](mailto:L.Koenigsberger@murdoch.edu.au)

**LORIMER, Prof. Jack**

University of Western Ontario, Department of Chemistry, London, ON N6A 5B7

Canada TEL: +519-434-1944

FAX: +519-661-3022

Email: [lorimer@uwo.ca](mailto:lorimer@uwo.ca)

**MACZYŃSKI, D. Andrzej**

Institute of Physical Chemistry, Polish Academy of Sciences, 44/52 Kspreczaka, 01-224 Warsaw, Poland

Tel: +48 (608) 033360

Email: [macz@ichf.edu.pl](mailto:macz@ichf.edu.pl)

**MAGALHÃES, Prof. Clara**

University of Aveiro, Department of Chemistry, P-3810 Aveiro, Portugal

Tel: +351 (234) 370200

Fax: +351 (234) 370084

Email: [mclara@ua.pt](mailto:mclara@ua.pt)

**MAKINO, Dr. Masakazu**

Institute for Environmental Sciences, University of Shizuoka, 52-1 Yada Suruga, Shizuoka 422-8526, Japan

Tel: +81 (54) 264 5785

Fax: + 81 (54) 264-5785

Email: [makinom@u-shizuoka-ken.ac.jp](mailto:makinom@u-shizuoka-ken.ac.jp)

**SALOMON, Dr. Mark**

MaxPower, Inc., 141 Christopher Lane, Harleysville, PA 19438, USA

Tel (MaxPower): +1 215 256 4575, extension 103

Email (MaxPower): [mark.salomon@maxpowerinc.com](mailto:mark.salomon@maxpowerinc.com)

Email: [marksalomon@comcast.net](mailto:marksalomon@comcast.net)

**SANGSTER, Dr. James**

Sangster Research Laboratories

P.O. Box 49562

5122 Cote des Neiges

Montreal, Quebec, Canada H3T 2A5

Tel: +1 (514) 340-4711

Fax: +1 (514) 340-5840

Email: [James.Sangster@polymtl.ca](mailto:James.Sangster@polymtl.ca)

**SAWADA, Prof. Kiyoshi**

Graduate School of Science and Technology

Niigata University, Japan

Email: [sawada@sc.niigata-u.ac.jp](mailto:sawada@sc.niigata-u.ac.jp)

**SHAW, Prof. David**

University of Alaska at Fairbanks, 85 E. India Row, No. 26 A

Boston, MA 02110

Email: [DavidShaw@Post.harvard.edu](mailto:DavidShaw@Post.harvard.edu)

**TOIKKA, Prof. Alexander**

Saint-Petersburg State University, Universitetskij pr. 26, Petrodvoretz, St. Petersburg, Russia

Email: [toikka@yandex.ru](mailto:toikka@yandex.ru)

**TOMKINS, Prof. Reginald**

Department of Chemical Engineering, New Jersey Institute of Technology, University Heights,  
Newark, NJ 07102

Tel: 973 596 5656

Fax: 973 596 8436

Email: [tomkinsr@njit.edu](mailto:tomkinsr@njit.edu)

**VALYASHKO, Prof. Vladimir**

Kurnakov Institute of General and Inorganic Chem. Russian Academy of Sciences, Leninsky Ave.  
31, Moscow

Email: [valyashko@igic.ras.ru](mailto:valyashko@igic.ras.ru)

**VOIGT, Prof. Wolfgang**

Institut für Anorganische Chemie, TU Bergakademie Freiberg, Leipziger Strasse 29, D-09596  
Freiberg, Federal Republic of Germany

Tel: +49 (3731) 394338

Fax: + 49 (3731) 394058

Email: [wolfgang.voigt@chemie.tu-freiberg.de](mailto:wolfgang.voigt@chemie.tu-freiberg.de)

**WAGHORNE, Prof. Earle W.**

UCD School of Chemistry and Chemical Biology, University College Dublin, Belfield, Dublin 4,  
Ireland

Tel: +353 (1) 706-2132



Fax: +353 (1) 706-2415  
Email: [earle.waghorne@ucd.ie](mailto:earle.waghorne@ucd.ie)

**WILLIAMS, Prof. Peter**  
School of Natural Sciences, University of Western Sydney, Australia  
Email: [P.Williams@uws.edu.au](mailto:P.Williams@uws.edu.au)

Appendix 2  
Agenda Item 7  
Editor in Chief's Report

**Editor-in-Chief's Report**  
**Dublin, July, 2008**

1. IUPAC-NIST Published Volumes In *J. Phys. Chem. Ref. Data*.

Vol. 82. Alcohols with Water – Revised and Updated, A. Maczynski, D.G. Shaw, M. Goral and B. Wisniewska-Gocłowska.

- Part 1. C<sub>4</sub> Alcohols with Water, *JPCRD*, **36(1)**, 59 (2007).
- Part 2. C<sub>5</sub> Alcohols with Water, *JPCRD*, **36(1)**, 133 (2007).
- Part 3. C<sub>6</sub> Alcohols with Water, *JPCRD*, **36(2)**, 399 (2007).
- Part 4. C<sub>7</sub> Alcohols with Water, *JPCRD*, **36(2)**, 445 (2007).
- Part 5. C<sub>8</sub> – C<sub>17</sub> Alcohols with Water, *JPCRD*, **36(3)**, 685 (2007).

Vol. 83. Acetonitrile: Ternary & Quaternary Systems. V.P. Sazonov, D.G. Shaw, A. Skrzecz, N.I. Lisov and N.V. Sazonov, *JPCRD* **36 (3)**, 733, 2007

Vol. 84. Solubility of Inorganic Actinide Compounds. J. Hala and H. Miyamoto, *JPCRD* **36(4)**, 1417 (2007).

Vol. 85. Transition and 12-14 Main Group Metals, Lanthanide, Actinide and Ammonium Halates. H. Miyamoto, R. Miyamoto, C. Guminski, M. Salomon, C. Balarew, E.V. Zagnit'ko, J. Eysseleva and J-J. Counieux, *JPCRD*, **37(2)**, 933 (2008).

Vol. 86. Ethers and Ketones with Water. A. Maczynski, D.G. Shaw, M. Goral and B. Wisniewska-Gocłowska.

- Part 1. C<sub>2</sub>–C<sub>5</sub> Ethers with Water, *JPCRD*, **37(2)**, 1119 (2008)
- Part 2. C<sub>6</sub> Ethers with Water, *JPCRD*, **37(2)**, 1147 (2008)
- Part 3. C<sub>7</sub>–C<sub>14</sub> Ethers with Water, *JPCRD* **37(2)**, 1169 (2008)
- Part 4. C<sub>4</sub> and C<sub>5</sub> Ketones with Water, in press
- Part 5. C<sub>6</sub> Ketones with Water, in press
- Part 6. C<sub>7</sub>–C<sub>12</sub> Ketones with Water, in press

**Vol. 87. Rare Earth Metal Chlorides in Water and Aqueous Systems. T. Mioduski, C. Guminski and D. Zeng.**

- Part 1. Scandium Group (Sc, Y, La), in press

2. Notes on New Formats for Manuscripts Submitted to JPCRD

A number of changes in formatting have been requested by the AIP (publishers of JPCRD). Manuscripts should be submitted in editable format; Microsoft Word is a preferred word processor. The AIP and NIST have allotted a total of around 1000 pages for the SDS per year (an exact number will be forthcoming).

Details on submitting manuscripts with the updated formatting requests are given below.

1) From the standpoint of JPCRD, having the whole paper in one file is not essential (that can be unwieldy), but the number of files should be small. Having every data (compilation, evaluation) sheet a separate file is unacceptable. From the standpoint of JPCRD, it would not be unreasonable to have a set of files in this case like:

- a) Introductory material, including title page, Table of Contents, key words and lists of tables and figures and so forth.
  - b) Evaluation and compilations of system No. 1 (no continuation heading on a succeeding page).
  - c) Evaluation and compilations of system No. 2.
  - d) Evaluation and compilations of system No. n.
  - e) Each of the above sections should be numbered consistent with the Table of Contents.
  - f) Cumulative References (see summary below).
- 2). Figures need to be separate files and in .pdf format
  - 3). JPCRD needs equations to be entered with the Microsoft Equation Editor in Word (alternatively, MathType is also acceptable).
  - 4). The "References" sections at the end of individual compilation sheets must be removed. JPCRD cannot have references 1, 2, etc. in individual compilations when those numbers are for different references in the context of the whole paper, i.e. in the Cumulative References (see 1f) above). If a data sheet refers to other papers in addition to the data paper being compiled, the reference must either use a superscript number corresponding to the reference number in the context of the whole paper (most useful if that paper is being referred to multiple times or is the data reference on another data sheet so would have a reference number anyway), or preferably, the reference can be inserted in brackets on the compilation pages.
  - 5). Please do not use Word's automatic Table of Contents feature, as this makes it difficult to edit. There is no need to put manuscript page numbers in the Contents; the only page numbers that matter are in the printed version and this will be done by the publisher.
  - 6). The indexes (author, system CAS Registry number) are no longer needed. The American Institute of Physics (the AIP) publishes JPCRD electronically in .pdf format which has its own efficient search options.
  - 7). The following is a summary of 4) above on placing references in manuscripts.
 

The Cumulative Set of References" contains all the references from the introductory material and the critical evaluations – this cumulative set is in alphabetical order by lead author and chronologically within the same author. Note that there are two issues of which to be aware. *Issue one* is that with each critical evaluation, the reference numbers will not be continuous, but they will be sequential.

*Issue two* is that in each compilation, the references which occur in the "Auxiliary Data" section must be handled differently; they are buried within the text (in parentheses).
  - 8). For less common journals, the AIP often requests that we provide full journal titles, ISSN and CODEN numbers. If you can't find them, it's OK.

Examples of the most recent submission is attached (portions of Volume 86 by Cezary and Dwen are attached).

IUPAC-NIST Solubility Data Series. 87.  
Rare Earth Metal Chlorides in Water and Aqueous Systems.  
**Part 1. Scandium Group (Sc, Y, La)**

**Tomasz Mioduski<sup>a)</sup>**

*Institute of Nuclear Chemistry and Technology, Warsaw, Poland*

**Cezary Gumiński**

*Department of Chemistry, University of Warsaw, Warsaw, Poland*

*Email: cegie@chem.uw.edu.pl*

**Dewen Zeng**

*College of Chemistry and Chemical Engineering, Hunan University, Hunan, P.R. China*

*Email: dewen\_zeng@hotmail.com*

This volume presents solubility data for rare earth metal chlorides in water and in ternary and quaternary aqueous systems. The material is divided into 3 parts: scandium group (Sc, Y, La), light lanthanide (Ce–Eu) and heavy lanthanide (Gd–Lu) chlorides; this part covers the scandium group. Compilations of all available experimental data are introduced for each rare earth metal chloride with a corresponding critical evaluation. Every such evaluation contains a tabulated collection of all solubility results in water, a scheme of the water-rich part of the equilibrium  $\text{LnCl}_3\text{-H}_2\text{O}$  phase diagram, solubility equation(s), a selection of suggested solubility data and a discussion of the multicomponent systems. Because the ternary and quaternary systems were almost never studied more than once, no critical evaluations or systematic comparisons of such data were possible. Only simple chlorides (no complexes) are treated as the input substances in this work. The literature (including a through coverage of papers in Chinese and Russian) has been covered through the middle of 2007.

Key words: aqueous solution; lanthanum chloride; phase diagram, scandium chloride; solubility; yttrium chloride.

a) deceased.

## CONTENTS

1. Preface.....	
1.1. Scope of the Volume .....	
1.2. Nature of the Equilibrium Solid Phases .....	
1.3. Solubility as a Function of Temperature.....	
1.4. Quality of the Solubility Results.....	
1.5. Solubility as a Function of <i>REM</i> Atomic Number.....	
1.6. Acknowledgements and History of the Project.....	
1.7. References for the Preface .....	
2. Solubility of Scandium Chloride .....	
2.1. Critical Evaluation of the Solubility of $\text{ScCl}_3$ in Aqueous Solutions .....	
2.2. Data for $\text{ScCl}_3$ in Aqueous Systems .....	
3. Solubility for Yttrium Chloride.....	
3.1. Critical Evaluation of the Solubility of $\text{YCl}_3$ in Aqueous Solutions .....	
3.2. Data for the $\text{YCl}_3\text{-H}_2\text{O}$ System .....	
3.3. $\text{YCl}_3\text{-Inorganic Salt-H}_2\text{O}$ Systems.....	
3.3.1. $\text{YCl}_3\text{-MCl-H}_2\text{O}$ (M=H, K) Systems .....	
3.3.2. $\text{YCl}_3\text{-MCl}_2\text{-H}_2\text{O}$ (M=Mg, Cd) Systems.....	
3.3.3. $\text{YCl}_3\text{-MCl}_3\text{-H}_2\text{O}$ (M=La, Ce, Pr, Nd, Eu, Gd, Ho) Systems.....	
3.3.4. $\text{YCl}_3\text{-YF}_3\text{-H}_2\text{O}$ System.....	
3.4. $\text{YCl}_3\text{-Organic Compound-H}_2\text{O}$ Systems.....	
3.5. Quaternary Systems.....	
4. Solubility of Lanthanum Chloride.....	
4.1. Critical Evaluation of the Solubility of $\text{LaCl}_3$ in Aqueous Solutions .....	
4.2. Data for the $\text{LaCl}_3\text{-H}_2\text{O}$ System .....	
4.3. $\text{LaCl}_3\text{-Inorganic Salt-H}_2\text{O}$ Systems.....	
4.3.1. $\text{LaCl}_3\text{-MCl-H}_2\text{O}$ (M=H, $\text{NH}_4$ , Li, Na, K, Rb, Cs) Systems .....	
4.3.2. $\text{LaCl}_3\text{-MCl}_2\text{-H}_2\text{O}$ (M=Mg, Ca, Sr, Ba, Cd) Systems.....	
4.3.3. $\text{LaCl}_3\text{-MCl}_3\text{-H}_2\text{O}$ (M=Ce, Eu, Yb, Sm, Nd) Systems .....	
4.3.4. $\text{LaCl}_3\text{-LaA}_3\text{-H}_2\text{O}$ (A=F, Br, $\text{NO}_3$ ) Systems .....	
4.4. $\text{LaCl}_3\text{-Organic Compound-H}_2\text{O}$ Systems.....	
4.4.1. $\text{LaCl}_3\text{-Amine Hydrochloride-H}_2\text{O}$ Systems .....	
4.4.2. $\text{LaCl}_3\text{-Hydrazine hydrochloride-H}_2\text{O}$ Systems.....	
4.4.3. $\text{LaCl}_3\text{-Ketone-H}_2\text{O}$ Systems.....	
4.4.4. $\text{LaCl}_3\text{-Acetic acid-H}_2\text{O}$ System.....	
4.4.5. $\text{LaCl}_3\text{-Amide-H}_2\text{O}$ Systems .....	
4.4.6. $\text{LaCl}_3\text{-Urea (Thiourea)-H}_2\text{O}$ Systems.....	
4.4.7. $\text{LaCl}_3\text{-N-heterocycle-H}_2\text{O}$ Systems .....	
4.5. Quaternary Systems.....	
5. Cumulative References .....	

### List of Tables

1. The experimental solubility data of  $\text{ScCl}_3$  in water at two temperatures
2. Experimental values of solubility of  $\text{YCl}_3$  in  $\text{H}_2\text{O}$  as a function of temperature
3. Recommended (R), tentative (T) and doubtful (D) solubilities of  $\text{YCl}_3$  in  $\text{H}_2\text{O}$  at selected temperatures
- 4 Experimental solubilities reported for the  $\text{LaCl}_3\text{-H}_2\text{O}$  system as a function of temperature
- 5 Recommended (R), tentative (T) and doubtful (D) solubilities of  $\text{LaCl}_3$  in  $\text{H}_2\text{O}$  at selected temperatures

### List of Figures

1. Selected solubility data for  $\text{REMCl}_3$ , ordered according to atomic number of  $\text{REM}$ , at 273, 298 and 333 K.
2. Water-rich part of the  $\text{YCl}_3\text{-H}_2\text{O}$  equilibrium phase diagram
3. Water-rich part of the  $\text{LaCl}_3\text{-H}_2\text{O}$  equilibrium phase diagram

# 1. Preface

## 1.1. Scope of the Volume

A rather traditional generic term rare earth metal (*REM*) is convenient for the title because it comprises the title elements: scandium, yttrium, lanthanum and all lanthanides. Their chlorides seem to be the most common salts of *REMs*. In recent decades, we observe increasing applications of these compounds in technology and science. They are used for production of pure *REMs*, their alloys and other compounds (materials for glass and ceramics dyeing, luminescence and laser technique, corrosion inhibition, hydrogen storage and nuclear fuel reprocessing). Also, organic chemists found a spectacularly high number of reactions catalyzed by *REM* chlorides. Potentially, medical chemists look to apply these compounds as imaging agents in computer tomography and the prevention of renal stones. Also, knowledge about the solubility in multicomponent systems as well as the related equilibrium phase diagrams is essential for identification of complex compounds of *REM* chlorides with other salts and many organic compounds. This may likewise improve the extraction and refining of *REM* compounds. Recently, several papers related to the solubilities of *REM* chlorides in binary, ternary and quaternary aqueous systems have been published each year. Some selected solubility data of *REM* chlorides in aqueous systems were previously collected,<sup>1-3</sup> but no systematic evaluation of all relevant results was carried out. This work attempts to systematize the immense experimental material acquired in this field. We hope that the present volume will serve as a useful guide for research and technology that involves *REMCl*<sub>3</sub>.

This volume continues the evaluation series of solubilities of *REM* salts in water (nitrates,<sup>4</sup> sulfates,<sup>5</sup> ethylsulfates,<sup>6</sup> iodates<sup>7</sup> and other halides<sup>8</sup>) and *REM* halides in non-aqueous solvents<sup>9</sup>. These projects have been connected with activity of the IUPAC Solubility Data Commission and Subcommittee. This volume will be published in three parts:

Part 1 (this paper): Scandium group (Sc, Y, La)

Part 2: Light lanthanides (Ce-Eu)

Part 3: Heavy lanthanides (Gd-Lu)

## 1.2. Nature of the Equilibrium Solid Phases

Solubility and phase diagrams are mutually connected. Phase diagrams aid in understanding and interpretation of the solubility results, therefore their schematic sketches are included in every critical evaluation, except the *ScCl*<sub>3</sub>-H<sub>2</sub>O phase diagram because the corresponding data for this system are contradictory so far. It is common knowledge that the equilibrium solid phases in *REMCl*<sub>3</sub>-H<sub>2</sub>O systems near room temperature have constant hydrate numbers *r* of the chlorides: 7 for La, Ce and Pr, and 6 for Sc, Y and Nd–Lu as found by the method of Schreinemakers, chemical analysis and well confirmed by some crystallographic studies. The isotypic heptahydrates are triclinic,  $P\bar{1}$  space group and



isotypic hexahydrates are monoclinic, P2/n space group.<sup>3</sup> The heptahydrate structure is based on the 9-coordinate complex  $[REMCl_2(H_2O)_7]^+$  which forms dimeric  $[(H_2O)_7REMCl_2REM(H_2O)_7]^{4+}$  units. The hexahydrate structure contains 8-coordinate species  $[REMCl_2(H_2O)_6]^+$ .

Below 273 K, the hydration number in  $REMCl_3 \cdot nH_2O$  may be, depending on the system, 8, 9, 10 or even 15, as established in exhaustive thermal analysis studies by Sokolova *et al.*<sup>10</sup> Unfortunately, only a monoclinic structure and cell parameters for  $YbCl_3 \cdot 9H_2O$  were identified by x-ray diffraction. Therefore, it would be very useful to confirm the results of Sokolova in another laboratory. At temperatures higher than 380 K the isotypic  $REMCl_3 \cdot 3H_2O$  hydrates may be formed, and they were structurally investigated by Reuter *et al.*<sup>11</sup> who found orthorhombic structure, Pnma space group. The structure consists of  $[REMCl_{4/2}Cl(H_2O)_3]$  chains, where two  $REM^{3+}$  ions are connected via two  $Cl^-$  ions.

A comparison of the phase diagram shapes of the water-rich parts of the systems  $REMCl_3-H_2O$  showed quite smooth changes of their invariant points and temperature ranges of stability of the equilibrium solid phases through *REM* series. Such regularities allowed predicting<sup>12</sup> the  $PmCl_3-H_2O$ ,  $HoCl_3-H_2O$  and  $TmCl_3-H_2O$  phase diagrams which were earlier not experimentally investigated.

An extension of the phase diagrams to higher temperatures and more salt-rich parts is complicated by the hydrolysis reaction with the formation of oxychlorides (or hydroxychlorides), according to the scheme:



Teixeira da Silva<sup>13</sup> calculated the stability diagram of phases  $LaCl_3 \cdot 7H_2O$ ,  $LaCl_3$ ,  $LaOCl$  and  $La_2O_3$  depending on temperature and vapor pressures of  $H_2O$  and  $HCl$ ; it facilitates an estimation of the extent of reaction (1) at equilibrium conditions. Rafalski and Jonak<sup>14</sup> investigated kinetics of the hydrolysis reaction (1) of several  $REMCl_3$  and found that content of the oxychlorides formed during 3 min of dehydration was never higher than 1.5 mass % at temperatures lower than 450 K and pressure between 0.01 and 0.04 MPa. This means that solubility determinations and thermal analysis at temperatures higher than 400 K should be performed relatively fast; this condition may be practically satisfied only for the latter method of experiments.

### 1.3. Solubility as a Function of Temperature

The solubility values are represented in phase diagrams by the liquidus lines. In the binary  $REMCl_3-H_2O$  systems, the liquiduses are known from eutectic points, much below 273 K, to more than 370 K and even up to 653 K, as in the case of  $YCl_3$ . This group of salts may be classified as quite well soluble. There are some general features of the solubility data of  $REMCl_3$  in water when temperature is changed. Typically, a content of ~0.05 mole fraction of  $REMCl_3$  in mixtures with  $H_2O$  decreases the melting eutectic temperature to the range 209-

231 K. Above the eutectic points, the solubilities increase moderately with temperature. Increases of the solubility are quite insignificant near room temperature. Unexpectedly, large increases of the solubility have recently been observed above 370 K when temperatures of the peritectic (or congruent) decomposition of  $REMCl_3 \cdot 6H_2O$  or  $REMCl_3 \cdot 7H_2O$  phases are approached. The strongly bent shapes of the liquiduses require use of an extended form of the solubility equation because the familiar, linear equation

$$\ln x = A + BT^{-1} \quad (2)$$

(where  $x$  is the salt solubility expressed in mole fraction,  $T$  the absolute temperature in K,  $A$  and  $B$  constants), could be used only in very narrow temperature ranges. Therefore, we tried to use the advanced form of solubility equation, applied for salts within the IUPAC solubility evaluation projects,<sup>15</sup> to describe more adequately the liquidus:

$$\ln \{ x^v (1-x)^r (v+r)^{v+r} r^{-r} [1 + (v-1)x]^{-(v+r)} \} = A + BT^{-1} + C \ln T + DT, \quad (3)$$

where  $v$  is the number of ions produced upon salt dissociation and  $r$  is the number of water molecules in the equilibrium solid phase. The constants  $A$ ,  $B$ ,  $C$  and  $D$  are derived from a fitting procedure when a minimum of 4 solubility results at various temperatures as well as  $v$  and  $r$  are known. Due to the many parameters used and significant scatter of the solubility data from various laboratories for a selected system, application of Eq. (3) quite frequently resulted in unexplained convexities on the solubility curves. In such situations, we used the truncated form of Eq. (3), without the fourth term  $DT$ , to obtain monotonic dependencies of the solubility on temperature.

Equations (2) and (3) are valid between the eutectic and peritectic or between two peritectic points when the hydration number in the equilibrium solid phase is constant. It would be possible to establish the hydration number in the solid phase in the course of the fitting procedure using Eq. (3), but the solubility results should be then sufficiently precise and minimally scattered.

#### 1.4. Quality of the Solubility Results

Except for the results from the laboratory of Spedding and coworkers, who carried out the most careful and multi-faceted determinations, the precision and accuracy achieved in other laboratories were rarely better than 1 %. There are several reasons why the solubility data for  $REMCl_3$  are seldom precise. The equilibria between the crystal hydrates and the saturated solutions are very slow and times of days are needed to obtain constant results from the subsequent solubility determinations. Harkot<sup>16,17</sup> investigated the rate of polythermal crystallization of  $LaCl_3 \cdot 7H_2O$  and found that the crystallization process was also complicated in the liquid phase by complex chemical and structural transformations of polycondensated forms of the salt. A possibility of formation of two crystal hydrates increases if additionally a crystallization occurs near a peritectic temperature.

Solubilities of  $REMCl_3$  are pH-sensitive. The saturated solutions of the pure stoichiometric salts (1:3) are acidic,  $1 < \text{pH} < 2$ .<sup>18</sup> An increase of pH leads to the formation of hydroxychlorides which, according to Ref. 19, should be less soluble than pure  $REMCl_3$ . A decrease of pH by addition of HCl made also a regular decrease of  $REMCl_3$  solubility that was observed for many systems investigated. Instead, addition of acetic acid made quite marginal changes of the solubilities of  $REMCl_3$ ; the recalculated molal concentrations (in other words: the molar ratio of the salt to water) were practically constant up to 60 mass % of acetic acid, suggesting that an unchanged hydrate of  $REMCl_3$  would exist in the saturated solutions independently of acetic acid concentration. Complexation of  $REM$  ions by Cl ions in the solutions of  $REMCl_3$ , according to the discussion of Oakes *et al.*,<sup>20</sup> seems to be negligible. Thus, one may conclude that the use of only very pure salts of proper stoichiometry guarantees reliable and precise solubility determinations.

Sometimes the solubility data were only graphically presented. When they were found to be important, the results were read out from the figures by the compilers. The precision of such a procedure was always indicated in the data sheets under the heading Estimated Error. The suggested solubilities at different temperatures, tabulated in the Critical Evaluations, belong to various classes of precision. If there is an agreement of at least two independent studies within experimental errors, the solubility values are assigned to the Recommended category. The Tentative values were suggested if one reliable result was reported or if the mean value of at least two studies was outside their error limits. In the tabulation, four, three and two significant figures of the selected solubility values are assigned for precision that is better than  $\pm 0.1$ ,  $\pm 1$  and  $\pm 10$  %, respectively.

Most of the solubility data were reported in the literature in mass % and seldom in terms of molality. For the sake of uniformity, all essential data were converted from mass % into mol  $\text{kg}^{-1}$  (molal units), using the IUPAC 2005 standard atomic masses. Critical evaluations also contain results of the saturating concentrations for the binary  $REMCl_3$ - $H_2O$  systems expressed in mole fractions because the phase diagrams were plotted in such units.

### 1.5. Solubility as a Function of $REM$ Atomic Number

Dependence of the solubility of  $REMCl_3$  on  $REM$  atomic number is presented in Fig. 1. The selected solubility data at 273, 298 and 333 K are used for its construction.

[Fig. 1. Selected solubility data for  $REMCl_3$ , ordered according to atomic number of  $REM$ , at 273, 298 and 333 K.]

One may easily observe that the solubility at three temperatures is almost constant for La, Ce and Pr; these elements form the heptahydrates as the equilibrium solid phases. For  $NdCl_3$ , the heptahydrate is the solid phase at 273 K whereas the hexahydrate is the equilibrium solid phase at 298 and 333 K; however, the solubility of  $NdCl_3$  is higher than of

$\text{PrCl}_3$  at 273 and 298 K but not at 333 K when a significant relative decrease of the solubility of  $\text{NdCl}_3$  is observed. In the case of the rest of  $\text{RE}M\text{Cl}_3$ , which form hexahydrates at 298 and 333 K, parabola-like dependencies of the solubility versus atomic number (or decreasing ionic radius of  $\text{REM}$ ) are observed. The shape of the analogous dependence at 273 K is similar, however between Er and Lu the octahydrates or nonahydrates are formed as the equilibrium solid phases. A minimum of the solubility was observed for Tb at all three temperatures. Because the three dependencies shown in Fig. 1 are almost parallel, the temperature dependencies of the solubility for any individual  $\text{RE}M\text{Cl}_3$  should be quite similar.

The solubility values for  $\text{YCl}_3$ , which should be near those for  $\text{HoCl}_3$ , as was observed for the nitrates,<sup>4</sup> sulfates<sup>5</sup> and ethylsulfates,<sup>6</sup> are atypically close to the values for  $\text{YbCl}_3$ . The solubility values determined for  $\text{ScCl}_3$  at 273 and 298 K are strongly scattered and significantly higher than for the rest of  $\text{RE}M\text{Cl}_3$ ; they do not show any smooth dependence of the solubility, therefore no quantitative conclusions related to this system may be drawn. Due to the much higher ionic potential of Sc, its compounds may display atypical physico-chemical properties.

## 1.6. Acknowledgements and History of the Project

The evaluators and compilers gratefully acknowledge the encouraging help of the IUPAC Commission V.8. and later Subcommittee on Solubility and Equilibrium Data. The project was initiated in the early eighties by Mioduski during the catalytic chairmanship of A.S. Kertes and continued during the chairmanships of J.W. Lorimer, M. Salomon, D. Shaw and H. Gamsjaeger. Advice and suggestions of M. Salomon and J.W. Lorimer were very fruitful. The first outline of the volume was ready in 1989. The sudden death of Mioduski in 2000 stopped the project for several years, although Mioduski had gathered more than a half of the material for this volume. In 2004 Gumiński started to complete the literature from the last two decades (and sometimes earlier), supplement and rearrange the critical evaluations and data sheets, check numerical data and retype the whole material into electronic form. Realization of the final stage of the volume preparation was made possible by Zeng, who found many papers in Chinese, translated them, calculated solubility equations, plotted the phase diagram figures and improved the Critical Evaluations. Therefore, Gumiński is very thankful to H. Gamsjaeger and W. Voigt for the successful involvement of Zeng in the project.

The authors of this volume also acknowledge the important inputs of J.A. Rard, K.A. Gschneider, J.E. Powell, V. Valyashko, M. Skripkin, H.U. Borgstedt, G. Brunisholz, J.C.G. Buenzli, L. Niinisto, G.R. Choppin, J. Burges, J. Kijowski, T. Moeller, J.M. Haschke, Z.X. Tang, Y.Sh. Chen, Y.L. Xu and K.L. Loening, who provided the authors with reprints of hardly available papers and reports.

## 1.7. References for the Preface

- <sup>1</sup> H. Stephen and T. Stephen, *Solubilities of Inorganic and Organic Compounds* (Pergamon, Oxford, 1979).
- <sup>2</sup> J. Burgess and J. Kijowski, *Adv. Inorg. Chem. Radiochem.* **24**, 57 (1981).
- <sup>3</sup> *Gmelins Handbook*, Sc, Y, La and Ln Chlorides, Springer-Verlag, Berlin, **39**, Part C4a, 1982.
- <sup>4</sup> S. Siekierski, T. Mioduski and M. Salomon, *IUPAC Solubility Data Series, Vol. 13, Sc, Y, La and Ln Nitrates* (Pergamon, Oxford, 1983).
- <sup>5</sup> T. Mioduski, *Chem. Anal. (Warsaw)* **43**, 457 (1998).
- <sup>6</sup> T. Mioduski, *J. Radioanal. Nucl. Chem. Lett.* **128**, 351 (1988).
- <sup>7</sup> H. Miyamoto, *Current Topics Sol. Chem.* **1**, 81 (1994).
- <sup>8</sup> T. Mioduski, unpublished materials.
- <sup>9</sup> T. Mioduski and M. Salomon, *IUPAC Solubility Data Series, Vol. 22, Sc, Y, La, and Ln Halides in Nonaqueous Solvents* (Pergamon, Oxford, 1985).
- <sup>10</sup> N. P. Sokolova, *Radiokhimiya* **30**, 435 (1988).
- <sup>11</sup> G. Reuter, H. Fink and H. J. Seifert, *Z. Anorg. Chem.* **620**, 665 (1994).
- <sup>12</sup> C. Gumiński, *Arch. Metall. Mater.* **51**, 617 (2006).
- <sup>13</sup> F. Teixeira da Silva, *Trans. Inst. Min. Metall., Ser. C* **114**, C70 (2005).
- <sup>14</sup> W. Rafalski and K. Jonak, *Przemysl Chem.* **48**, 735 (1969).
- <sup>15</sup> J. W. Lorimer and R. Cohen-Adad, *Thermodynamics of Solubility*, in *The Experimental Determination of Solubilities*, G. T. Hefter and R. P. T. Tomkins, Eds., (Wiley, Chichester, 2003), p. 19.
- <sup>16</sup> J. Harkot, *Polish J. Chem.* **63**, 337 (1989).
- <sup>17</sup> J. Harkot, *Polish J. Chem.* **64**, 53 (1990).
- <sup>18</sup> F. H. Spedding, J. A. Rard and V. Saeger, *J. Chem. Eng. Data* **19**, 373 (1974).
- <sup>19</sup> L. G. Sillen and A. Martell, *Stability Constants*, Special Publications no 17 and 25 (The Chemical Society, London, 1964 and 1971).
- <sup>20</sup> C. S. Oakes, J. A. Rard and D. G. Archer, *J. Chem. Eng. Data* **49**, 313 (2004).

## 2. Solubility of Scandium Chloride

### 2.1. Critical Evaluation of the Solubility of ScCl<sub>3</sub> in Aqueous Solutions

---

Components:	Evaluators:
(1) Scandium chloride; ScCl <sub>3</sub> ; [10361-84-9]	T. Mioduski, Institute of Chemistry and Nuclear Technology, Warsaw, Poland &
(2) Water; H <sub>2</sub> O; [7732-18-5]	C. Gumiński, Department of Chemistry, University of Warsaw, Poland
	November 2007

---

The ScCl<sub>3</sub>-H<sub>2</sub>O system was not intensively studied and results of the corresponding investigations are frequently controversial. The solubility of ScCl<sub>3</sub> in water has been determined by Fischer *et al.*<sup>21-23</sup> at 273 K, Samodelov<sup>24</sup> at 298 K and Suzuki *et al.*<sup>25</sup> at 298.2 K. Table 1 summarizes their results.

[Table 1. The experimental solubility data of ScCl<sub>3</sub> in water at two temperatures]

The lower solubility limit that could be estimated from the determination of Ref. 21 seems to be relatively far from saturation, and this value may be rejected. In the subsequent publications by Fischer *et al.*,<sup>22,23</sup> two solubility values were reported for nonahydrate as the equilibrium solid phase at 273 K. The experimental result in Ref. 22 was higher than the one in Ref. 23 which was obtained from a smoothing procedure. This difference was not commented on by the authors. The still higher solubility value was reported by Samodelov<sup>24</sup> at 298 K. These results,<sup>24</sup> expressed in mass % and molar concentrations of ScCl<sub>3</sub>, were inconsistent by 0.5 %. Molalities in the corresponding data sheet were calculated by the compilers using the solubility data expressed in mass % ScCl<sub>3</sub>. Moreover, the experimental solubility result of 6.22 mol kg<sup>-1</sup> is slightly higher than the theoretical composition of the nonahydrate of 6.17 mol kg<sup>-1</sup> (the solubility of ScCl<sub>3</sub> in its own crystallization water) invalidating the experimental result of Ref. 24. The situation was not clarified in the precise determination of Suzuki *et al.*,<sup>25</sup> who found significantly lower solubility of ScCl<sub>3</sub> at 298.2 K and specified ScCl<sub>3</sub>·6H<sub>2</sub>O [20662-14-0] as the equilibrium solid phase. The previous measurements<sup>21-24</sup> were not commented on in Ref. 25. All authors equilibrated the solutions with crystals for 1-2 d which, by comparison to the lanthanide chloride–water systems, seems to be too short a period. Therefore, at present, it is impossible to suggest the most probable solubility value which should be equal to or higher than 3.83 and equal to or lower than 6.17 mol kg<sup>-1</sup> ScCl<sub>3</sub> between 273 and 298 K.

Also, the solute entity does not clarify the confusing situation. ScCl<sub>3</sub>·9H<sub>2</sub>O was found as the equilibrium solid phase at 273 and 298 K in several works.<sup>22-24,26</sup> Unexpectedly, ScCl<sub>3</sub>·6H<sub>2</sub>O was reported as the solute at 298 K by Ref. 25. In addition to the equilibrium

solids analysis in Refs. 22-24, Lim *et al.*<sup>26</sup> claimed that  $\text{ScCl}_3 \cdot 7\text{H}_2\text{O}$  is the “maximally” hydrated salt at room temperature and determined its crystal structure as monoclinic (which is typical of  $\text{LnCl}_3 \cdot 6\text{H}_2\text{O}$ ). The single crystals in Ref. 26 were deposited from concentrated aqueous solutions, being slightly acidified to avoid hydrolysis.

Hygroscopic  $\text{ScCl}_3 \cdot 6\text{H}_2\text{O}$  (most frequently recrystallized from concentrated HCl solutions) was the starting substance for several dehydration studies.<sup>27-33</sup> The results differed greatly with respect to the temperature and nature of the intermediate products.  $\text{ScCl}_3 \cdot 5\text{H}_2\text{O}$  [114364-35-1] was found in the investigations of Refs. 31 and 32,  $\text{ScCl}_3 \cdot 4\text{H}_2\text{O}$  [64618-80-0] in Refs. 32 and 33,  $\text{ScCl}_3 \cdot 3\text{H}_2\text{O}$  [114364-36-2] in Ref. 28,  $\text{ScCl}_3 \cdot 2\text{H}_2\text{O}$  [114364-37-3] in Ref. 33 and  $\text{ScCl}_3 \cdot 1.5\text{H}_2\text{O}$  in Ref. 30. Further heating resulted in formation of  $\text{ScOCl}$ ,<sup>27-29,31,33,34</sup> which was finally decomposed to  $\text{Sc}_2\text{O}_3$  above 830 K. Melting of  $\text{ScCl}_3 \cdot 6\text{H}_2\text{O}$  was found at 333 K,<sup>27,31</sup> 343-353 K,<sup>28</sup> 346 K,<sup>33</sup> 341 K<sup>34</sup> and 336-339 K,<sup>35</sup> pointing to a relatively good agreement in this matter. However, taking into account all these experimental observations, no even provisional  $\text{ScCl}_3\text{-H}_2\text{O}$  phase diagram may be constructed at present.

Addition of HCl to the  $\text{ScCl}_3$  solution caused a systematic decrease of the  $\text{ScCl}_3$  solubility at 273 K<sup>22,23</sup> as well as at 298 K,<sup>24</sup> the solubility increased again at higher HCl concentrations, but this effect was stronger at 273 K. The authors of Refs. 22 and 23 observed that  $\text{ScCl}_3 \cdot 9\text{H}_2\text{O}$  was the solute up to 10 mol  $\text{kg}^{-1}$  HCl, whereas Ref. 24 found it up to only 2.1 mol  $\text{dm}^{-3}$ ; above these HCl concentrations, both authors found the hexahydrate as the equilibrium solid phase.

Summarizing these observations, it is impossible at present to suggest any quantitative solubility values of  $\text{ScCl}_3$  as well as an unquestionable composition of the equilibrium solid phase at a selected temperature. The only statement that may be made at present is that  $\text{ScCl}_3$  is a well soluble salt, its hexahydrate melts at about 340 K and dehydrates gradually. Further studies on this system are strongly advised.

## References

- <sup>21</sup> W. Fischer, J. Wernet and M. Zumbusch-Pfisterer, *Z. Anorg. Chem.* **258**, 157 (1949).
- <sup>22</sup> W. Fischer and G. Buhler, *Z. Anorg. Chem.* **285**, 156 (1956).
- <sup>23</sup> W. Fischer, H. Bauer, I. Dillo, T. Molaug, J. Nier, H. Rohrer and K. Trovaag, *Z. Anorg. Chem.* **357**, 177 (1968).
- <sup>24</sup> A. P. Samodelov, *Zh. Neorg. Khim.* **10**, 1735 (1965).
- <sup>25</sup> Y. Suzuki, R. Yoshino, H. Saitoh, K. Fukushi and Y. Sonehara, *J. Alloys Comp.* **180**, 383 (1992).
- <sup>26</sup> K. C. Lim, B. W. Skelton and A. H. White, *Aust. J. Chem.* **53**, 875 (2000).
- <sup>27</sup> W. W. Wendlandt, *J. Inorg. Nucl. Chem.* **5**, 118 (1957).
- <sup>28</sup> F. Petru and F. Kutek, *Collect. Czech. Chem. Commun.* **25**, 1143 (1960).
- <sup>29</sup> F. Petru, J. Hradilova and B. Hajek, *Collect. Czech. Chem. Commun.* **33**, 2720 (1968).

- <sup>30</sup> W. Crookes, Phil. Trans. Roy. Soc. **A 209**, 15 (1908).
- <sup>31</sup> I. V. Arkhangelskii, L. N. Komissarova, V. M. Shatskii and N. P. Shepelev, Zh. Neorg. Khim. **17**, 310 (1972).
- <sup>32</sup> G. A. Melson, D. J. Olszanski and A. K. Rahimi, Spectrochim. Acta **A 33**, 301 (1977).
- <sup>33</sup> S. V. Aleksandrovskii, Zh. Prikl. Khim. **70**, 1761 (1997).
- <sup>34</sup> K. H. Schröder, Dissertation, Münster, Germany, 1955; as quoted in Ref. 3.
- <sup>35</sup> *Aldrich 2007-2008 Handbook of Fine Chemicals*, Sigma-Aldrich Corp., St. Louis, 2170 (2007).



## 2.2. Data for ScCl<sub>3</sub> in Aqueous Systems

Components:	Original Measurements:
(1) Scandium chloride; ScCl <sub>3</sub> ; [10361-84-9]	<sup>21</sup> W. Fischer, J. Wernet, and M. Zambusch-Pfisterer, Z. Anorg. Chem. <b>258</b> , 157 (1949).
(2) Hydrochloric acid; HCl; [7647-01-0]	
(3) Water; H <sub>2</sub> O; [7732-18-5]	
(4) Diethyl ether; C <sub>4</sub> H <sub>10</sub> O; [60-29-7]	
(5) Ethanol; C <sub>2</sub> H <sub>6</sub> O; [64-17-5]	

Variables:	Prepared by:
Solvents	T. Mioduski & C. Gumiński
Temperature: 273 K or room	

### Experimental values

Solubility of ScCl<sub>3</sub> in water and aqueous mixed solvents at 0 °C or room temperature

Solvent	g Sc <sub>2</sub> O <sub>3</sub> / 100 cm <sup>3</sup> of saturated solution	c <sub>1</sub> / mol dm <sup>-3a</sup>
H <sub>2</sub> O	>16	>2.3
40 % HCl	>1.5	>0.22
40 % HCl + diethyl ether (1:1)	>4	>0.6
43 % HCl in 95 % ethanol <sup>b</sup>	1.25 <sup>b</sup>	0.18 <sup>b</sup>

<sup>a</sup> calculated by the compilers from Sc<sub>2</sub>O<sub>3</sub> content used by the authors as the starting material for the experiments

<sup>b</sup> at room temperature

The equilibrium solid phases were not specified.

The authors observed that the degree of ScCl<sub>3</sub> co-precipitation with NaCl, NH<sub>4</sub>Cl or MgCl<sub>2</sub> was very low.

### Auxiliary Information

#### Method/Apparatus/Procedure:

The solvents were saturated with HCl gas in a glass apparatus. Filtrates of the saturated and non-saturated solutions were analyzed gravimetrically for Sc content after its precipitation as Sc<sub>2</sub>O<sub>3</sub>.

#### Source and Purity of Materials:

The materials used were of reagent-grade purity.

#### Estimated Error:

Nothing specified.

<b>Components:</b>	<b>Original Measurements:</b>
(1) Scandium chloride; ScCl <sub>3</sub> ; [10361-84-9]	<sup>22</sup> W. Fischer and G. Buhler, Z. Anorg. Chem.
(2) Hydrochloric acid; HCl; [7647-01-0]	<b>285</b> , 156 (1956).
(3) Water; H <sub>2</sub> O; [7732-18-5]	
<b>Variables:</b>	<b>Prepared by:</b>
Concentration of HCl: 0-30.2 mass %	T. Mioduski & C. Gumiński
One temperature: 273 K	

### Experimental Values

Solubility of ScCl<sub>3</sub> in water and HCl solutions at 0 °C

Equilibration time / d	100w <sub>2</sub>	m <sub>2</sub> <sup>a</sup>	100w <sub>1</sub>	m <sub>1</sub> <sup>a</sup>	n <sup>b</sup>
3 <sup>c</sup>	0	0	43.2	5.03	6.2
3 <sup>d</sup>	0.3	0.47	41.3	4.67	6.8
2 <sup>d</sup>	2.1	0.99	39.9	4.45	6.4
2 <sup>d</sup>	4.8	2.1	32.1	3.36	9.6
3 <sup>d</sup>	8.2	3.8	32.7	3.66	6.7
2 <sup>c</sup>	12.2	5.6	28.3	3.14	5.8
2 <sup>d</sup>	14.2	6.2	23.2	2.45	9.1
3 <sup>c</sup>	18.2	8.4	22.0	2.43	6.2
2 <sup>d</sup>	19.7	8.8	18.9	2.03	8.9
3 <sup>d</sup>	22.5	10.3	18.0	2.00	5.9
1 <sup>c</sup>	23.4	10.8	17.2	1.91	6.5
2 <sup>c</sup>	24.6	11.4	16.1	1.79	6.2
3 <sup>d</sup>	24.9	11.6	16.4	1.85	6.2
2 <sup>c</sup>	25.5	11.9	15.7	1.76	6.4
3 <sup>d</sup>	28.3	13.6	14.8	1.72	6.1
2	30.2	16.1	18.5	2.38	6.1
2	28.8	16.7	23.8	3.32	5.7
3	28.3	16.4	24.3	3.39	6.0
3	28.0	16.1	24.3	3.37	6.2
3	27.6	15.9	24.7	3.42	5.9
3	25.8	15.2	27.7	3.94	6.2

<sup>a</sup> calculated by the compilers

<sup>b</sup> number of water molecules in the equilibrium solid formula ScCl<sub>3</sub>·nH<sub>2</sub>O

<sup>c</sup> equilibrium approached from supersaturation by precipitation of the solid excess after the solution was cooled to 0 °C

<sup>d</sup> equilibrium approached from undersaturation by addition of the solid solute to HCl solution

The authors stated that  $\text{ScCl}_3 \cdot 6\text{H}_2\text{O}$  and  $\text{ScCl}_3 \cdot 9\text{H}_2\text{O}$  are the equilibrium solid phases at HCl concentrations higher and lower than 23 mass %, respectively; the solute  $\text{ScCl}_3 \cdot 9\text{H}_2\text{O}$  was experimentally identified in only 3 of a total of 10 tests performed.

Based on the experimental data presented above, the authors of Ref. 23 reported smoothed values of  $\text{ScCl}_3$  solubility in HCl solutions at 0 °C.

Smoothed values of solubility of  $\text{ScCl}_3$  in water and aqueous solutions of HCl at 0 °C

$m_2$	$m_1$ / g-equivalent $\text{kg}^{-1}$	$m_1^a$	Equilibrium solid phase
0	12.1	4.0	$\text{ScCl}_3 \cdot 9\text{H}_2\text{O}$
5	8.0	2.7	$\text{ScCl}_3 \cdot 9\text{H}_2\text{O}$
10	6.0	2.0	$\text{ScCl}_3 \cdot 9\text{H}_2\text{O}$
14	5.2	1.7	$\text{ScCl}_3 \cdot 6\text{H}_2\text{O}$
15.2	11.8	3.9	$\text{ScCl}_3 \cdot 6\text{H}_2\text{O}$

<sup>a</sup> calculated by the compilers

### Auxiliary Information

#### Method/Apparatus/Procedure:

Isothermal method was used. A hydrate of  $\text{ScCl}_3$  was equilibrated with the solvents by mixing for 1-3 d at the selected temperature. The saturated solutions were filtered through glass wool. Sc was precipitated as  $\text{Sc}(\text{OH})_3$  with  $\text{NH}_3$  and determined gravimetrically as  $\text{Sc}_2\text{O}_3$ . Total chloride content was found from a potentiometric titration. Composition of the equilibrium solid was graphically determined by the dry residue method of Schreinemakers.

#### Source and Purity of Materials:

$\text{Sc}_2\text{O}_3$  was purified from Zr, Hf, Fe, Al, Ln and finally contained 0.1 %  $\text{ZrO}_2$ . It was further transferred into  $\text{ScCl}_3$ , probably in a form of hydrate.

#### Estimated Error:

Solubility: nothing specified;  $\pm(2-7)$  % (as estimated by the compilers from the data scatter on a graphical presentation of all results).

Temperature: nothing specified.

<b>Components:</b> (1) Scandium chloride; ScCl <sub>3</sub> ; [10361-84-9] (2) Hydrochloric acid; HCl; [7647-01-0] (3) Water; H <sub>2</sub> O; [7732-18-5]	<b>Original Measurements:</b> <sup>24</sup> A.P. Samodelov, Zh. Neorg. Khim. <b>10</b> , 1735 (1965).
<b>Variables:</b> Concentration of HCl: 0-18.2 mass % One temperature: 298 K	<b>Prepared by:</b> T. Mioduski & C. Gumiński

### Experimental Values

Solubility of ScCl<sub>3</sub> in H<sub>2</sub>O and HCl solutions at 25 °C

$\rho$ / kg dm <sup>-3</sup>	100w <sub>2</sub>	c <sub>2</sub>	100w <sub>1</sub>	c <sub>1</sub>	m <sub>1</sub> <sup>a</sup>	Equilibrium solid <sup>b</sup>
1.420	0	0	48.50	4.65	6.22	A
1.415	0.01	0.04	48.10	4.54	6.13	A
1.417	0.20	0.06	47.10	4.45	5.91	A
1.413	0.46	0.18	46.20	4.36	5.72	A
1.408	0.80	0.34	44.30	4.16	5.33	A
1.403	1.28	0.50	44.80	4.20	5.49	A
1.398	2.10	0.81	39.70	3.69	4.51	A
1.396	2.20	0.82	42.80	3.98	5.14	A
1.393	2.58	1.00	40.70	3.78	4.74	A
1.391	2.85	1.10	40.80	3.78	4.78	A
1.387	3.32	1.28	38.60	3.57	4.39	A
1.390	3.11	1.20	40.20	3.72	4.69	A
1.385	4.00	1.54	38.00	3.51	4.33	A
1.382	4.31	1.58	38.80	3.58	4.51	A
1.381	4.93	1.91	37.50	3.45	4.30	A
1.380	5.07	1.94	38.70	5.56 <sup>c</sup>	4.55	A
1.372	5.36	2.05	37.60	3.45	4.36	A
1.370	5.54	2.12	37.60	3.44	4.37	A
1.368	5.67	2.17	37.30	3.42	4.32	B
1.367	5.92	2.26	36.80	3.36	4.25	B
1.367	6.43	2.40	36.00	3.28	4.13	B
1.364	6.60	2.50	35.60	3.24	4.07	B
1.355	7.45	2.80	34.30	3.10	3.89	B
1.354	7.50	2.82	33.20	3.00	3.70	B
1.363	7.62	2.87	33.45	3.02	3.75	B
1.350	8.00	3.00	33.30	3.00	3.75	B
1.345	8.46	3.16	32.00	2.86	3.55	B

1.341	8.87	3.30	32.00	2.86	3.58	B
1.338	9.15	3.40	30.50	2.72	3.34	B
1.335	9.45	3.51	30.95	2.85	3.43	B
1.333	9.60	3.56	30.20	2.68	3.31	B
1.331	9.64	3.64	30.00	2.66	3.28	B
1.331	9.92	3.66	31.50	2.81	3.55	B
1.328	10.13	3.74	30.80	2.72	3.45	B
1.323	10.65	3.92	29.70	2.62	3.29	B
1.320	10.90	4.00	29.55	2.60	3.28	B
1.316	11.22	4.10	29.20	2.56	3.24	B
1.312	11.42	4.30	29.10	2.55	3.23	B
1.309	12.10	4.40	28.40	2.48	3.15	B
1.306	12.40	4.50	28.70	2.50	3.22	B
1.305	13.80	5.00	27.60	2.40	3.11	B
1.302	14.40	5.20	26.60	2.31	2.98	B
1.300	15.22	5.50	26.00	2.25	2.92	B
1.302	15.90	5.75	25.80	2.24	2.92	B
1.303	16.30	5.90	26.20	2.28	3.01	B
1.305	16.55	6.00	26.42	2.30	3.06	B
1.311	17.30	6.30	26.80	2.34	3.17	B
1.317	18.60	6.52	27.50	2.41	3.37	B
1.320	18.00	6.60	27.50	2.42	3.33	B
1.323	18.20	6.70	29.20	2.78	3.70	B

<sup>a</sup> calculated by the compilers

<sup>b</sup> A = ScCl<sub>3</sub>·9H<sub>2</sub>O; B = ScCl<sub>3</sub>·6H<sub>2</sub>O

<sup>c</sup> misprinted value, the correct result should be 3.56 mol dm<sup>-3</sup>

Extrapolation of the solubility results to zero HCl concentration, when ScCl<sub>3</sub>·6H<sub>2</sub>O would be the solute, points to ScCl<sub>3</sub> solubility in water of ~58 mass %, as metastable solution.

### Auxiliary Information

#### Method/Apparatus/Procedure:

Equilibrium was reached from above (by increasing HCl concentration in ScCl<sub>3</sub> solution that made precipitation of ScCl<sub>3</sub> hydrate excess) or from below (by addition of ScCl<sub>3</sub> into HCl solution). 8-10 h was found to be sufficient time to reach equilibrium, but the solutions were agitated for another 20-25 h. HCl was added in gaseous form into the system. ScCl<sub>3</sub> content was determined gravimetrically and spectrographically, but no details were reported. HCl content was found by titration with alkali hydroxide solution after addition of dry NaF to precipitate ScF<sub>3</sub>·3NaF in the solution and to avoid hydrolysis. Composition of the solid

phases was determined by the residue method of Schreinemakers and chemical analysis. Then Sc was precipitated with  $\text{NH}_3$ , the resulting  $\text{Sc}(\text{OH})_3$  ignited and weighed as  $\text{Sc}_2\text{O}_3$ . Content of Cl was determined gravimetrically as  $\text{AgCl}$ .

**Source and Purity of Materials:**

$\text{ScCl}_3$  was prepared by chlorination of  $\text{Sc}_2\text{O}_3$  (99 % pure), purified by thiocyanate extraction with ether, mixed with wood charcoal at 1273-1373 K.

Gaseous HCl was obtained by a "conventional method" to saturate the solutions.

Water purity was not specified.

**Estimated Error:**

Solubility: nothing specified;  $\pm(1-3)$  % (as estimated by the compilers from the scatter of the results on their graphical presentation).

Temperature: precision  $\pm 0.05$  K.

---

<b>Components:</b>	<b>Original Measurements:</b>
(1) Scandium chloride; ScCl <sub>3</sub> ; [10361-84-9]	<sup>25</sup> Y. Suzuki, R. Yoshino, H. Saitoh, K. Fukushi and Y. Sonehara, J. Alloys Comp. <b>180</b> , 383 (1992).
(2) Water; H <sub>2</sub> O; [7732-18-5]	
<b>Variables:</b>	<b>Prepared by:</b>
One temperature: 298.2 K	C. Gumiński

---

### Experimental Values

Solubility of ScCl<sub>3</sub> in H<sub>2</sub>O at 25.0 °C was found to be 3.8307 mol kg<sup>-1</sup>. The equilibrium solid phase was stated as ScCl<sub>3</sub>·6H<sub>2</sub>O but no analysis of this phase was reported.

---

### Auxiliary Information

#### Method/Apparatus/Procedure:

Isopiestic measurements in isothermal conditions were performed. A stainless steel chamber contained 6 Pt cups placed in a Cu block. The cups contained either reference or sample solutions. The chamber was thermostated. A Pt gauze was added to each cup to promote equilibration. The chamber was slowly evacuated and then gently rotated. The molalities at equilibrium were obtained from the weight of stock solution in a cup and the weight of solution in a cup after equilibration. The equilibrium periods ranged from 1 to 2 d and was accepted to be when the concentration of two samples of each solution agreed to within ±0.15 %. Concentrations of ScCl<sub>3</sub> and CaCl<sub>2</sub> (used as the reference) were determined gravimetrically.

#### Source and Purity of Materials:

The solutions of ScCl<sub>3</sub> were prepared from HCl solution (reagent grade) and Sc<sub>2</sub>O<sub>3</sub> 99.9+ %. Source of H<sub>2</sub>O was not specified. CaCl<sub>2</sub> was of reagent grade, probably recrystallized.

#### Estimated Error:

Solubility: precision better than ±0.15 %.

---

### 3. Solubility of Yttrium Chloride

#### 3.1. Critical Evaluation of the Solubility of $\text{YCl}_3$ in Aqueous Solutions

---

<b>Components:</b>	<b>Evaluators:</b>
(1) Yttrium chloride; $\text{YCl}_3$ ; [10361-92-9]	T. Mioduski, Institute of Nuclear Chemistry and Technology, Warsaw, Poland &
(2) Water; $\text{H}_2\text{O}$ ; [7732-18-5]	C. Gumiński, Department of Chemistry, University of Warsaw, Poland &
	D. Zeng, College of Chemistry and Chemical Engineering, Hunan University, P.R. China
	July, 2007

---

Data for the solubility of  $\text{YCl}_3$  in water and aqueous solutions have been reported in many papers.<sup>21,23,36-64</sup> The solubility values were mainly determined by analytical methods at isothermal conditions.<sup>21,23,36-51,53-58,62-64</sup> Thermal analysis,<sup>60</sup> visual observation of last crystal dissolution,<sup>61</sup> isopiestic<sup>52,58,59</sup> and vapor-pressure measurements<sup>61</sup> were also used. Many studies dealt with ternary and even quaternary systems containing  $\text{YCl}_3$ , and the solubility in pure water was then given mostly as one point in a phase diagram. In several studies devoted to ternary systems and performed in the same laboratory, probably one solubility determination in water was performed and that result was further repeated; however, several times the corresponding values were slightly different. All solubility results for the  $\text{YCl}_3\text{-H}_2\text{O}$  system are summarized in Table 2.

[Table 2. Experimental values of solubility of  $\text{YCl}_3$  in  $\text{H}_2\text{O}$  as a function of temperature]

The experiments at low<sup>60</sup> and high temperatures<sup>61</sup> enriched our knowledge of the whole  $\text{YCl}_3\text{-H}_2\text{O}$  system, however the results obtained at low temperatures<sup>60</sup> show only qualitative agreement with the majority of the solubility data and there are some reservations about the interpretation of the results obtained in Ref. 60, as is pointed out in the corresponding data sheet. Doubtless,  $\text{YCl}_3\cdot 15\text{H}_2\text{O}$ ,  $\text{YCl}_3\cdot 9\text{H}_2\text{O}$ ,  $\text{YCl}_3\cdot 8\text{H}_2\text{O}$  and  $\text{YCl}_3\cdot 6\text{H}_2\text{O}$  [10025-94-2] equilibrium solid phases were found to be stable up to their peritectic decompositions at 250, 273, 276 (as reported in Ref. 60) and 426 K (as reported in Ref. 10), respectively; see the schematic phase diagram of the  $\text{YCl}_3\text{-H}_2\text{O}$  system in Fig. 2. The thermal arrests observed in Ref. 60 at ~221 K, placed between  $\text{YCl}_3\cdot 15\text{H}_2\text{O}$  and  $\text{YCl}_3\cdot 9\text{H}_2\text{O}$  compounds, were unexplained and might reflect either a decomposition temperature of another intermediate phase formed between  $\text{YCl}_3\cdot 15\text{H}_2\text{O}$  and  $\text{YCl}_3\cdot 9\text{H}_2\text{O}$  or a metastable eutectic reaction. The eutectic point at 0.042 mole fraction  $\text{YCl}_3$  and 231 K seems to be more probable than at 0.051 mole fraction  $\text{YCl}_3$  and 221 K.

[Fig. 2. Water-rich part of the  $\text{YCl}_3\text{-H}_2\text{O}$  equilibrium phase diagram]



The inflection on the liquidus between 276 and 300 K should not be attributed to any defined thermodynamic effect, but rather results from fitting of the experimental data because the solubility values obtained from thermal analysis<sup>60</sup> below 276 K are distinctly lower than the solubility results above 276 K. Concerning the high-temperature part of the diagram, the authors of Ref. 61 found a liquidus which was relatively flat at first and with a high slope above 458 K. Without further decisive experiments in this range, we are not able to designate the character of the reaction at 458 K: peritectic or congruent melting. The authors of Ref. 61 suggested that above 458 K the equilibrium solid phase was anhydrous  $\text{YCl}_3$ ; this statement also needs confirmation in a future experiment because, according to several papers, the dehydration of  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$ , upon heating or action of a drying agent, was found to be a stepwise process with the formation of  $\text{YCl}_3 \cdot 5.5\text{H}_2\text{O}$ ,<sup>65</sup>  $\text{YCl}_3 \cdot 5\text{H}_2\text{O}$  [114364-34-0],<sup>65,66</sup>  $\text{YCl}_3 \cdot 4\text{H}_2\text{O}$  [114364-33-9]<sup>66</sup>  $\text{YCl}_3 \cdot 3\text{H}_2\text{O}$  [114364-32-8],<sup>65-68</sup>  $\text{YCl}_3 \cdot 2.5\text{H}_2\text{O}$ ,<sup>65</sup>  $\text{YCl}_3 \cdot 2\text{H}_2\text{O}$  [114364-31-7],<sup>66-69</sup>  $\text{YCl}_3 \cdot \text{H}_2\text{O}$  [13470-35-4]<sup>65-71</sup> and finally dehydrated  $\text{YCl}_3$ .<sup>65-67,69,71</sup> The authors of Ref. 72 negated, after x-ray diffraction investigations, existence of  $\text{YCl}_3 \cdot 3\text{H}_2\text{O}$  in the  $\text{YCl}_3\text{-H}_2\text{O}$  system. Formation of  $\text{YCl}_3 \cdot 5.5\text{H}_2\text{O}$ ,  $\text{YCl}_3 \cdot 5\text{H}_2\text{O}$ ,  $\text{YCl}_3 \cdot 4\text{H}_2\text{O}$  and  $\text{YCl}_3 \cdot 2.5\text{H}_2\text{O}$  seems to be unlikely in the opinion of the evaluators.  $\text{YOCl}$  was identified as the final product of thermogravimetric experiments in Refs. 65, 71, 73 and 74. Further thermolysis of  $\text{YOCl}$  led to formation of  $\text{Y}_2\text{O}_3$ .<sup>65,74</sup>

The melting point of  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$  was determined by many investigators: 426 K,<sup>10</sup> 434-436 K,<sup>37</sup> 458 K,<sup>53</sup>  $458 \pm 2$  K,<sup>61</sup> 429-433 K,<sup>70</sup> 463 K,<sup>74</sup> 433 K,<sup>75</sup> 424.2-425.0 K<sup>76</sup> and 458 K.<sup>77</sup> The significant differences may be explained either by the determinations being performed in different experimental conditions (closed or open vessels, various heating rates), by the melting temperature being very sensitive to the purity of  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$  used, or by another reaction in the range 424-436 K.

The solubility results obtained with the hexahydrate as the equilibrium solute at temperatures below 276 K are treated, according to the phase-diagram studies of Ref. 60, as corresponding to a metastable equilibrium, and they are excluded from the further evaluation. Thus, the solubility data between the peritectics at 276 and 458 K with the hexahydrate as the equilibrium solid will be further assessed; however, a selection of recommended values is still problematic. The solubility of  $\text{YCl}_3$  in water is relatively high, but its increase with temperature is relatively small up to about 373 K and becomes significant between 373 K and the peritectic (or congruent) melting point at 458 K.<sup>61</sup> This would suggest that the system in this temperature range displays a tendency to immiscibility or  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$  (by similarity to  $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$ ) melts congruently and forms a eutectic between  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$  and  $\text{YCl}_3 \cdot 2\text{H}_2\text{O}$  at a temperature slightly lower than 458 K as is reflected in the phase diagram in Fig. 2. Unexpectedly, further increase of temperature above 458 K leads again to a relatively small increase of the solubility, similar to that observed near room temperature.

Although some authors<sup>51,52,59</sup> claimed to reach the precision of  $\pm 0.1$  %, the whole scatter of the solubility data at selected temperatures is between 5 and 8 %. It is very characteristic that except for the scattered results of Ref. 36, other data,<sup>37-39,45,55</sup> differing in absolute values, show quite similar slopes of the dependencies of the logarithm of the solubility versus reciprocal temperature. Concerning the method of analysis of the saturated solutions, it was observed that the precipitation of  $Y_2O_3$ <sup>37,38</sup> gave higher results of the analysis than the titration of Y ions with EDTA solution.<sup>45,55</sup>

There are too few reliable solubility results to propose solubility equations when  $YCl_3 \cdot 15H_2O$ ,  $YCl_3 \cdot 9H_2O$  and  $YCl_3 \cdot 8H_2O$  are the equilibrium solid phases. Therefore, we concentrated on finding the proper equation between 276 and 458 K when the equilibrium solute is  $YCl_3 \cdot 6H_2O$ , as well as above 458 K when the equilibrium solid phase is probably  $YCl_3 \cdot 2H_2O$ . For construction of the solubility equations, we used the selected solubility values and the general form of solubility equation expressed by Eq. (3) (see Sec. 1.3). Based on least-square fitting, the following solubility equations were obtained:

$$\ln\{x_1^4(1-x_1)^6(4+6)^{4+6}6^{-6}[1+(4-1)x_1]^{-(4+6)}\} = 797.150 - 22972.8T^{-1} - 137.273\ln T - 0.20535T \quad (4)$$

for  $YCl_3 \cdot 6H_2O$  equilibrium solid phase between 276 and 458 K, where  $x_1$  is the solubility of  $YCl_3$  in water expressed in mole fraction and  $T$  the absolute temperature in K, and

$$\ln\{x_1^4(1-x_1)^2(4+2)^{4+2}2^{-2}[1+(4-1)x_1]^{-(4+2)}\} = 217.8634 - 9956.3T^{-1} - 34.619\ln T + 0.0331T \quad (5)$$

for  $YCl_3 \cdot 2H_2O$  equilibrium solid phase between 458 and 653 K, assuming that such solid phase is in equilibrium in this temperature range. The following solubility data, deviating by more than 3 % from the mean values, were not taken into account in the fitting procedure: Refs. 23, 36, 38 and 55 at 273 K, Ref. 37 at 283 K, Ref. 37 at 293 K, Refs. 38, 39, 43, 58 and 62 at 298 K, Refs. 37 and 47 at 303 K and all data from Refs. 36 and 45 at higher temperatures. The selected solubility values are collected in Table 3.

[Table 3. Recommended (R), tentative (T) and doubtful (D) solubilities of  $YCl_3$  in  $H_2O$  at selected temperatures]

The solubility of  $YCl_3$  in HCl solutions was measured in seven papers.<sup>21,23,37,42,45,63,64</sup> There is no doubt that addition of HCl to aqueous solution decreases the  $YCl_3$  solubility. A precise comparison of the data is rather difficult because of different experimental temperatures and different expressions of HCl content. The data of Ref. 37 show concentrations almost twice as high as the interpolated data of Refs. 23, 42 and 45; the latter three papers show concordant results. There is quite good agreement between the results of Ref. 42 and single points of Refs. 63 and 64 at 298 K. It is unexpected that, at the highest HCl concentration of 45 mass % and temperature of 373 K,<sup>45</sup> no partial dehydration of hexahydrate was seen.

Solubility in the ternary  $YCl_3$ - $LnCl_3$ - $H_2O$  systems at 298 K was studied by the group of Nikolaev for La,<sup>54</sup> Ce,<sup>40,41,50</sup> Pr,<sup>54</sup> Nd,<sup>54</sup> Eu,<sup>44</sup> Gd<sup>54,57</sup> and Ho.<sup>58</sup> All lanthanide chlorides

showed similar solubilities in a molal scale. According to these results, the heptahydrates of  $\text{LaCl}_3$ ,  $\text{CeCl}_3$  and  $\text{PrCl}_3$  do not form solid solutions with  $\text{YCl}_3$  hexahydrate of different structure, but at high  $\text{YCl}_3$  contents La, Ce or Pr form a series of solid solutions of the type  $(\text{Y,Ln})\text{Cl}_3 \cdot 6\text{H}_2\text{O}$ , thus adopting the structure of  $\text{YCl}_3$  hexahydrate. The  $\text{YCl}_3$ - $\text{NdCl}_3$ - $\text{H}_2\text{O}$  system contained a continuous series of solid solutions  $(\text{Y,Nd})\text{Cl}_3 \cdot 6\text{H}_2\text{O}$  as the equilibrium solid phase. Hydrated chlorides of Eu and Gd co-crystallized with  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$ ; at low  $\text{YCl}_3$  concentrations, the corresponding solid solutions based on  $\text{EuCl}_3 \cdot 6\text{H}_2\text{O}$  or  $\text{GdCl}_3 \cdot 6\text{H}_2\text{O}$  and at high  $\text{YCl}_3$  concentrations on  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$ , a solid miscibility break in between was attributed to a significant difference in the ionic radii of Gd or Eu and Y. For similar radii, such as for Y and Ho in the  $\text{YCl}_3$ - $\text{HoCl}_3$ - $\text{H}_2\text{O}$  system, the solid phase was reported to be a continuous solid solution  $(\text{Y,Ho})\text{Cl}_3 \cdot 6\text{H}_2\text{O}$  in the whole composition range. In the  $\text{YCl}_3$ - $\text{YF}_3$ - $\text{H}_2\text{O}$  system,<sup>53</sup> formation of  $\text{YCl}_3 \cdot 4\text{YF}_3 \cdot 9\text{H}_2\text{O}$  was observed.

Neither  $\text{KCl}$ <sup>39</sup> nor  $\text{MgCl}_2$ <sup>56</sup> formed double salts or solid solutions with  $\text{YCl}_3$  hexahydrate in the corresponding ternary systems at 298 K; the systems were found to be of eutonic type. In the  $\text{CdCl}_2$ - $\text{YCl}_3$ - $\text{H}_2\text{O}$  system,<sup>63</sup> the authors found a formation of two double salts  $4\text{CdCl}_2 \cdot \text{YCl}_3 \cdot 13\text{H}_2\text{O}$  and  $5\text{CdCl}_2 \cdot 2\text{YCl}_3 \cdot 26\text{H}_2\text{O}$  as well as a metastable  $8\text{CdCl}_2 \cdot \text{YCl}_3 \cdot 15\text{H}_2\text{O}$ .

The quaternary systems  $\text{YCl}_3$ - $\text{CeCl}_3$ - $\text{EuCl}_3$ - $\text{H}_2\text{O}$ <sup>46,54</sup> and  $\text{YCl}_3$ - $\text{EuCl}_3$ - $\text{HoCl}_3$ - $\text{H}_2\text{O}$ <sup>78</sup> at 298 K were also investigated. Solid phases in the first system were dominated by solid solutions. In the second system, a succession of solid solutions  $(\text{Y,Eu,Ho})\text{Cl}_3 \cdot 6\text{H}_2\text{O}$  was determined. Ref. 78 is not compiled because the results were not presented in numerical form and were condensed in hardly readable figures. Wang *et al.*<sup>64</sup> measured solubilities in the  $\text{YCl}_3$ - $\text{CsCl}$ - $\text{HCl}$ - $\text{H}_2\text{O}$  system at 298 K and observed crystallization of two double salts,  $\text{YCl}_3 \cdot 4\text{CsCl} \cdot 10\text{H}_2\text{O}$  and  $2\text{YCl}_3 \cdot 3\text{CsCl} \cdot 14\text{H}_2\text{O}$ , which released water gradually at elevated temperatures forming finally anhydrous compounds of the same salt stoichiometries. Qiao *et al.*<sup>63</sup> observed formation of the same stable double salts in the quaternary  $\text{CdCl}_2$ - $\text{YCl}_3$ - $\text{HCl}$ - $\text{H}_2\text{O}$  as well as in the ternary  $\text{CdCl}_2$ - $\text{YCl}_3$ - $\text{H}_2\text{O}$  system.

$\text{YCl}_3$  solubility was also investigated in solutions containing organic substances. The authors of Ref. 21 observed 20 times lower  $\text{YCl}_3$  solubility in 40 mass %  $\text{HCl}$  dissolved in a 1:1 mixture of diethyl ether and water, but 4 times higher solubility was observed in 43 mass %  $\text{HCl}$  dissolved in 95 mass % ethanol; both values are compared to the solubility of  $\text{YCl}_3$  in aqueous 40 mass %  $\text{HCl}$  solution at 273 K ( $1.1 \times 10^{-3}$  mol  $\text{kg}^{-1}$ ). The ternary systems of  $\text{YCl}_3$ - $\text{H}_2\text{O}$  with urea,<sup>47</sup> acetamide,<sup>48</sup> thiourea<sup>49</sup> and selenourea<sup>49</sup> were investigated at 303 K and with  $\beta$ -phenylalanine<sup>62</sup> at 298 K. Analysis of the equilibrium solid phases in these systems pointed to adduct formation between  $\text{YCl}_3$  and urea, acetamide or  $\beta$ -phenylalanine but not with thiourea and selenourea; the latter systems were of a simple eutonic type.

## References

<sup>10</sup> N. P. Sokolova, Radiokhimiya **30**, 435 (1988).

<sup>21</sup> W. Fischer, J. Wernet and M. Zumbusch-Pfisterer, Z. Anorg. Chem. **258**, 157 (1949).

- <sup>23</sup> W. Fischer, H. Bauer, I. Dillo, T. Molaug, J. Nier, H. Rohrer and K. Trovaag, *Z. Anorg. Chem.* **357**, 177 (1968).
- <sup>36</sup> M. C. Crew, H. E. Steinert and B. S. Hopkins, *J. Phys. Chem.* **29**, 34 (1925).
- <sup>37</sup> M. D. Williams, H. C. Fogg and C. James, *J. Am. Chem. Soc.* **47**, 297 (1925).
- <sup>38</sup> J. E. Powell, *U.S. Atom. Ener. Comm. Rep. IS-15*, p. 10 (1959).
- <sup>39</sup> Z. N. Shevtsova and In Vei-Tszyuan, *Zh. Neorg. Khim.* **8**, 1749 (1963).
- <sup>40</sup> A. V. Nikolaev, A. A. Sorokina and V. G. Tsubanov, *Dokl. Akad. Nauk SSSR* **172**, 1333 (1967).
- <sup>41</sup> A. V. Nikolaev, A. A. Sorokina, A. Ya. Vilenskaya and V. G. Tsubanov, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* **1967**, no 6, 5.
- <sup>42</sup> Z. N. Shevtsova, L. S. Nam and B. G. Korshunov, *Zh. Neorg. Khim.* **13**, 1682 (1968).
- <sup>43</sup> V. S. Petelina, N. I. Nikurashina and G. V. Illarionova, *Issledovaniya v Oblasti Khimii Redkozemelnykh Elementov, Saratovskii Universitet, Saratov* **3**, 48 (1971).
- <sup>44</sup> A. V. Nikolaev and A. A. Sorokina, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* **1971**, no 4, 61.
- <sup>45</sup> A. N. Zelikman, N. V. Baryshnikov and A.I. Khokhlov, *Zh. Neorg. Khim.* **16**, 2023 (1971).
- <sup>46</sup> A. V. Nikolaev, A. A. Sorokina and N. G. Yudina, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* **1972**, no 5, 28.
- <sup>47</sup> G. A. Ashimkulova, K. Sulaimankulov, Sh. T. Turdaliev and K. Noguev, *Zh. Neorg. Khim.* **18**, 2011 (1973).
- <sup>48</sup> G. A. Ashimkulova, *Geterogennye Ravnovesiya v Sistemakh Neorganicheskikh i Organicheskikh Soedinenii* (Ilim, Frunze, 1974), p. 21.
- <sup>49</sup> G. A. Ashimkulova, K. Noguev and K. Sulaimankulov, *Zh. Neorg. Khim.* **19**, 2588 (1974).
- <sup>50</sup> A. V. Nikolaev, L. G. Kosobudskaya and A. A. Sorokina, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* **1975**, no 5, 53.
- <sup>51</sup> F. H. Spedding, V. W. Saeger, K. A. Gray, P. K. Boneau, M. A. Brown, C. W. DeKock, J. L. Baker, L. E. Shiers, H. O. Weber and A. Habenschuss, *J. Chem. Eng. Data* **20**, 72 (1975).
- <sup>52</sup> F. H. Spedding, H. O. Weber, V. W. Saeger, H. H. Petheram, J.A. Rard and A. Habenschuss, *J. Chem. Eng. Data* **21**, 341 (1976).
- <sup>53</sup> N. Levina, G. Dadabaeva, D. D. Ikrami and A. S. Paramzin, *Dep. VINITI N-478-77*, 27 pp. (1977).
- <sup>54</sup> A. V. Nikolaev, A. A. Sorokina, V. N. Lubkova and N. G. Yudina, *Osnovy Vzaimodeistviya Solei Redkozemelnykh Elementov v Vode* (Nauka, Novosibirsk, 1977), p. 36, 45, 46, 53, 72, 79, 161.
- <sup>55</sup> A. V. Nikolaev, A. A. Sorokina, N. P. Sokolova, G. S. Kotlyar-Shapiro, N. P. Anoshina, L. I. Bagryantseva and A. P. Olovyanishnikova, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* **1977**, no 4, 84.

- <sup>56</sup> A. V. Nikolaev, A. A. Sorokina, P. A. Stabnikov, N. P. Sokolova and G. S. Kotlyar-Shapiro, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* **1977**, no 6, 44.
- <sup>57</sup> N. P. Sokolova, I. I. Yakovlev and I. N. Komissarova, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* **1980**, no 6, 45.
- <sup>58</sup> A. A. Sorokina and N. G. Yudina, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* **1981**, no 5, 51.
- <sup>59</sup> J. A. Rard and F. H. Spedding, *J. Chem. Eng. Data* **27**, 454 (1982).
- <sup>60</sup> N. P. Sokolova, *Zh. Neorg. Khim.* **28**, 782 (1983).
- <sup>61</sup> M. A. Urusova and V. M. Valyashko, *Zh. Neorg. Khim.* **38**, 1074 (1993).
- <sup>62</sup> F. Ren, Sh. L. Gao, G. H. Chu and I. Zh. Shiq, *Yingyong Huaxue (Chin. J. Appl. Chem.)* **17**, 203 (2000).
- <sup>63</sup> Zh. P. Qiao, L. H. Zhuo and H. Wang, *Wuji Huaxue Xuebao (Chin. J. Inorg. Chem.)* **20**, 929 (2004).
- <sup>64</sup> H. Wang, J. X. Duan, X. Q. Ran and Sh. Y. Gao, *Chin. J. Chem.* **22**, 1128 (2004).
- <sup>65</sup> L. Ya. Markovskii, E. Ya. Pesina, Yu. A. Omelchenko and Yu. D. Kondrashev, *Zh. Neorg. Khim.* **14**, 14 (1969).
- <sup>66</sup> N. V. Baryshnikov, A. V. Polonskii and A. I. Khokhlov, *Zh. Neorg. Khim.* **20**, 3177 (1975); A. V. Polonskii, N. V. Baryshnikov and L. A. Voskresenskaya, *Zh. Neorg. Khim.* **21**, 317 (1976).
- <sup>67</sup> J. Wang, Z. Guo, Y. Chen, S. Huang and Z. Wang, *J. Northwest Univ.* **20**, 49 (1990); as quoted in Ref. 68.
- <sup>68</sup> V. V. Hong and J. Sundström, *Thermochim. Acta* **307**, 37 (1997).
- <sup>69</sup> G. Haeseler and F. Matthes, *J. Less-Common Met.* **9**, 133 (1965).
- <sup>70</sup> C. Matignon, *Ann. Chim. Phys.* **8**, 433 (1906).
- <sup>71</sup> M. Z. Su and P. G. Li, *Huaxue Tongbao* **1979**, no 4, 34.
- <sup>72</sup> H. J. Seifert and S. Funke, *Thermochim. Acta* **320**, 1 (1998).
- <sup>73</sup> W. W. Wendlandt, *J. Inorg. Nucl. Chem.* **9**, 136 (1959).
- <sup>74</sup> L. G. Sokolova, A. V. Lapitskaya, A. F. Bolshakov, S. B. Pirkes and B. V. Abalduyev, *Zh. Neorg. Khim.* **26**, 1736 (1981); B. V. Abalduyev, A. F. Bolshakov, L. G. Sokolova, A. V. Lapitskaya and S. B. Pirkes, *Issledovaniya v Oblasti Khimii Redkozemelnykh Elementov (Saratovskii Universitet, Saratov, 1975)*, p. 47.
- <sup>75</sup> W. W. Wendlandt and R. G. Sewell, *Texas J. Sci.* **8**, 231 (1961).
- <sup>76</sup> Yu. G. Sakharova and T. A. Ezhova, *Zh. Neorg. Khim.* **21** 551 (1976); *Russ. J. Inorg. Chem.* **21**, 296 (1976).
- <sup>77</sup> W. W. Wendlandt and J. L. Bear, *Anal. Chim. Acta* **21**, 439 (1959).
- <sup>78</sup> A. V. Nikolaev, A. A. Sorokina and N. G. Yudina, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* **1983**, no 6, 116.

### 3.2. Data for the $\text{YCl}_3\text{-H}_2\text{O}$ System

<b>Components:</b>	<b>Original Measurements:</b>
(1) Yttrium chloride; $\text{YCl}_3$ ; [10361-92-9]	<sup>36</sup> M.C. Crew, H.E. Steinert and B.S. Hopkins,
(2) Water; $\text{H}_2\text{O}$ ; [7732-18-5]	J. Phys. Chem. <b>29</b> , 34 (1925).
<b>Variables:</b>	<b>Prepared by:</b>
Temperature: 273–353 K	T. Mioduski & C. Gumiński

#### Experimental Values

Solubility of  $\text{YCl}_3$  in water at various temperatures

$t / ^\circ\text{C}$	g $\text{YCl}_3$ / 100 g $\text{H}_2\text{O}$	$100w_1^a$	$m_1^a$
0	74.3	42.6	3.81
	72.7	42.1	3.72
16	76.5	43.4	3.92
	75.1	42.9	3.85
25.1	75.4	43.0	3.86
	75.3	43.0	3.86
45	77.0	43.5	3.94
	76.3	43.3	3.91
60	77.0	43.5	3.94
	77.6	43.7	3.97
80	78.1	43.9	4.00
	78.1	43.9	4.00

<sup>a</sup> calculated by the compilers

The authors tested that the degree of hydration of the solute was not changed between 298 and 353 K, and it may be assumed that the equilibrium solid was  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$ . At lower temperatures, the equilibrium solid could be nona- or octahydrate of  $\text{YCl}_3$ , but 5 h of equilibration seems to be insufficient time in the opinion of the compilers.

#### Auxiliary Information

##### Method/Apparatus/Procedure:

Isothermal method was used. Both solute and solvent contained in an Erlenmeyer flask were immersed in a thermostat and vigorously shaken at intervals. Degree of saturation was controlled periodically and equilibration of 5 h was found to be sufficient. At higher temperatures, the saturation point was reached by cooling the supersaturated solution from higher temperature. Duplicate samples were drawn off for analysis. Amount of Y was precipitated with oxalic acid. The precipitate was filtered, washed, dried and ignited to the oxide. The factor 2.9043 was used for converting the oxide to the chloride.

##### Source and Purity of Materials:

An initial source of Y contained traces of Ho and Er. It was purified by precipitation as oxalate and twice as hydroxide (in the compilers' opinion, the separation may not be effective).  $Y(OH)_3$  was converted to  $Y_2O_3$ , dissolved in HCl and the solution evaporated to crystallization. The crystals were filtered off, centrifuged, washed and centrifuged again. Freshly distilled conductivity water, protected from air, was used. All other reagents were carefully purified.

**Estimated Error:**

Solubility: precision  $\pm 1.5$  %.

Temperature: no better than  $\pm 0.1$  K (by the compilers).

---

<b>Components:</b>	<b>Original Measurements:</b>
(1) Yttrium chloride; YCl <sub>3</sub> ; [10361-92-9]	<sup>37</sup> M.D. Williams, H.C. Fogg and C. James, J. Am. Chem. Soc. <b>47</b> , 297 (1925).
(2) Hydrochloric acid; HCl; [7647-01-0]	
(3) Water; H <sub>2</sub> O; [7732-18-5]	
<b>Variables:</b>	<b>Prepared by:</b>
Solvent: H <sub>2</sub> O or ~21 mass % HCl	T. Mioduski, M. Salomon & C. Gumiński
Temperature: 283–323 K	

### Experimental Values

Solubility of YCl<sub>3</sub> in water at various temperatures

<i>t</i> / °C	100 <i>w</i> (Y <sub>2</sub> O <sub>3</sub> )	100 <i>w</i> <sub>1</sub>	<i>m</i> <sub>1</sub> <sup>a</sup>
10	25.37	43.85	4.000
20	25.50	44.08	4.037
30	25.64	44.33	4.078
40	25.86	44.70	4.140
50	26.07	45.07	4.202

<sup>a</sup> calculated by the compilers

The authors used YCl<sub>3</sub>·6H<sub>2</sub>O for the solubility tests, and they assumed that the equilibrium solid has the same formula. No analysis of the solute after the tests was reported.

The melting point of YCl<sub>3</sub>·6H<sub>2</sub>O was found to be 161-163 °C.

Solubility of YCl<sub>3</sub> in aqueous HCl solution of density 1.1051 g cm<sup>-3a</sup> at various temperatures

<i>t</i> / °C	100 <i>w</i> (Y <sub>2</sub> O <sub>3</sub> )	100 <i>w</i> <sub>1</sub>
10	14.09	24.35
15	14.12	24.41
20	14.25	24.63
30	14.74	25.48
40	15.34	26.52
50	15.94	27.55

<sup>a</sup> This density, if measured at 293 K, corresponds to an aqueous solution containing ~21 mass % HCl.

### Auxiliary Information

#### Method/Apparatus/Procedure:

Isothermal method was used. Samples were placed in bottles, brought to a temperature either above or below the experimental temperature. They were sealed, placed in a thermostat and rotated for 9 h or more. About 1 cm<sup>3</sup> of the saturated solution was withdrawn, diluted to 150 cm<sup>3</sup> and precipitated with oxalic acid. The mixture was allowed to stand for 12 h or precipitated with oxalic acid. The mixture was allowed to stand for 12 h or more, then



the precipitate was washed, ignited to  $Y_2O_3$  and weighed. The analyses were made in duplicate; the samples were taken from the same bottle.

**Source and Purity of Materials:**

The solute was prepared by dissolving highly purified  $Y_2O_3$  in HCl, evaporating the solution to the point of crystallization, cooling in ice and saturating with HCl. The crystals were filtered, washed with saturated HCl solution and recrystallized when necessary. The crystals were dried in vacuum over  $H_2SO_4$  and NaOH.

**Estimated Error:**

Nothing specified, no estimation possible.

---

<b>Components:</b>	<b>Original Measurements:</b>
(1) Yttrium chloride; YCl <sub>3</sub> ; [10361-92-9]	<sup>38</sup> J.E. Powell, U.S. Atom. Ener. Comm. Rep. IS-15, p. 10 (1959).
(2) Water; H <sub>2</sub> O; [7732-18-5]	

<b>Variables:</b>	<b>Prepared by:</b>
Temperature: 273–333 K	T. Mioduski & C. Gumiński

### Experimental Values

Solubility of YCl<sub>3</sub> in water at various temperatures

<i>t</i> / °C	g YCl <sub>3</sub> ·H <sub>2</sub> O / 100 g H <sub>2</sub> O	<i>m</i> <sub>1</sub> <sup>a</sup>
0	199	3.84
25	217	4.03
60	233	4.20

<sup>a</sup> calculated by the compilers

The equilibrium solid phase was reported to be YCl<sub>3</sub>·6H<sub>2</sub>O for all temperatures; this was assured by seeding the supersaturated solutions with YCl<sub>3</sub>·6H<sub>2</sub>O crystals. However, the equilibrium solid phase seems to be YCl<sub>3</sub>·9H<sub>2</sub>O or YCl<sub>3</sub>·8H<sub>2</sub>O at 273 K as shown on the equilibrium diagram.

### Auxiliary Information

#### Method/Apparatus/Procedure:

Saturated solutions were heated with an excess of YCl<sub>3</sub>·6H<sub>2</sub>O until the salt was completely dissolved and then cooled to the desired temperature. Small crystals of YCl<sub>3</sub>·6H<sub>2</sub>O were added to the supersaturated solutions, which were further equilibrated isothermally for 24-28 h. Aliquots of the saturated solutions were analyzed gravimetrically as the oxide for Y content. The salt was first evaporated to dryness, HNO<sub>3</sub> added and heated gradually to yield finally Y<sub>2</sub>O<sub>3</sub>, as reported in [J.E. Powell and H.R. Burkholder, J. Inorg. Nucl. Chem. **14**, 65 (1960)].

#### Source and Purity of Materials:

About 99.9 % pure Y<sub>2</sub>O<sub>3</sub> was obtained by ion exchange method [J.E. Powell and F.H. Spedding, Chem. Eng. Progr. Symp. Ser. **55**, 101 (1959)]. It was dissolved in a slight excess of aqueous HCl solution. The resulting moist salt was recrystallized several times before use.

#### Estimated Error:

Nothing specified.

<b>Components:</b>	<b>Original Measurements:</b>
(1) Yttrium chloride; YCl <sub>3</sub> ; [10361-92-9] or	<sup>43</sup> V.S. Petelina, N.I. Nikurashina and G.V.
(2) Lanthanum chloride; LaCl <sub>3</sub> ; [10099-58-8]	Illarionova, <i>Issledovaniya v Oblasti Khimii</i>
(3) Water; H <sub>2</sub> O; [7732-18-5]	Redkozemelnykh Elementov, Saratovskii
	Universitet, Saratov <b>3</b> , 48 (1971).

<b>Variables:</b>	<b>Prepared by:</b>
Salt: YCl <sub>3</sub> or LaCl <sub>3</sub>	T. Mioduski & C. Gumiński
One temperature: 298 K	

---

### Experimental Values

Solubility of YCl<sub>3</sub> in water at 25 °C was reported to be 45.08 mass % or 4.204 mol kg<sup>-1</sup> (as recalculated by the compilers). The equilibrium solid phase was found to be YCl<sub>3</sub>·6H<sub>2</sub>O.

Solubility of LaCl<sub>3</sub> in water at 25 °C was reported to be 46.26 mass % or 3.510 mol kg<sup>-1</sup> (as recalculated by the compilers). The equilibrium solid phase was found to be LaCl<sub>3</sub>·8H<sub>2</sub>O. It seems that these data contain two typographical errors: the solubility should be 49.26 mass % LaCl<sub>3</sub> or 3.958 mol kg<sup>-1</sup> (as recalculated by the compilers) and the solid phase formula should be LaCl<sub>3</sub>·7H<sub>2</sub>O as may be deduced from another paper from the same laboratory [V.S. Petelina, R.V. Mertslin, N.I. Nikurashkina and L.K. Sedova, *Issledovaniya v Oblasti Khimii Redkozemelnykh Elementov* (Saratovskii Universitet, Saratov, 1969), p. 85.].

---

### Auxiliary Information

#### Method/Apparatus/Procedure:

Isothermal method was used. The metals in the saturated solutions were determined gravimetrically as oxides by the oxalate method.

#### Source and Purity of Materials:

Oxides of the metals (of unknown purity) were dissolved in HCl (1:1) solutions. The solutions were evaporated until crystallization. The products were dried in a desiccator and analyzed for the metal contents by the oxalate method.

#### Estimated Error:

Nothing specified.

---

ETC.

## 4. Solubility of Lanthanum Chloride

### 4.1. Critical Evaluation of the Solubility of LaCl<sub>3</sub> in Aqueous Solutions

---

**Components:**

- (1) Lanthanum chloride; LaCl<sub>3</sub>; [10099-58-8]  
(2) Water; H<sub>2</sub>O; [7732-18-5]

**Evaluators:**

T. Mioduski, Institute of Nuclear Chemistry and Technology, Warsaw, Poland,  
C. Gumiński, Department of Chemistry, University of Warsaw, Poland &  
D. Zeng, College of Chemistry and Chemical Engineering, Hunan University, P.R. China  
December 2007

---

Solubility phenomena in LaCl<sub>3</sub>-H<sub>2</sub>O and aqueous multicomponent LaCl<sub>3</sub> systems have been studied intensively,<sup>10,16,18,23,40-45,51-55,79-152</sup> and therefore this evaluation is the most voluminous of all aqueous rare earth metal chloride systems. Abundant information is available especially on the solubilities of LaCl<sub>3</sub> in aqueous ternary systems at selected temperatures with a wide variety of third components.

Three papers<sup>51,52,54</sup> were compiled with the YCl<sub>3</sub>-H<sub>2</sub>O system (Sec. 3). For various reasons, several reports were not compiled here in the form of data sheets. Only abstracts of two reports<sup>109,117</sup> were available to the compilers. For the paper by Kana and Akimbo,<sup>128</sup> related to glass-formation studies of LaCl<sub>3</sub> solutions, the solubility of LaCl<sub>3</sub> in water, about 3.9 mol kg<sup>-1</sup> at room temperature, was read out from a figure. Suzuki *et al.*<sup>135,136</sup> studied activity coefficients of solutions and the solubility of LaCl<sub>3</sub> in water; the solubility data in numerical form were not published and those read out from the figures are 3.81 mol kg<sup>-1</sup> at 293 K<sup>135</sup> and 3.88 mol kg<sup>-1</sup> at 298 K.<sup>136</sup> The paper of Petelina *et al.*<sup>43</sup> was not compiled due to erroneous results of the solubility and the equilibrium solid phase; see the table of collected solubility data below. Studies by Ren *et al.* were devoted to formation of complexes between La ion and crown ethers B15C5<sup>134</sup> and 18C6<sup>138</sup> in ethanol solution; water content in these investigations was marginal since water was introduced only with the starting salt LaCl<sub>3</sub>·3H<sub>2</sub>O, therefore no data sheets for these reports<sup>134,138</sup> are prepared.

Historically, the first study on solubility of LaCl<sub>3</sub> was dated surprisingly late – in 1920.<sup>79</sup> Solubilities of LaCl<sub>3</sub> in 1-5 mol dm<sup>-3</sup> NH<sub>4</sub>Cl solutions, with and without addition of NH<sub>3</sub>, when the starting substance was La<sub>2</sub>O<sub>3</sub>, at 288, 303 and 323 K, were presented in barely readable figures. The solubility values reported were many times lower than those observed at similar conditions in the subsequent studies and formulae of the equilibrium solid phases, which contained O, suggested rather a formation of oxychloride of La in these conditions. Therefore, Ref. 79 is not compiled.

The solubilities of LaCl<sub>3</sub> in aqueous systems were mainly determined by analytical methods (gravimetry, titration, refractometry, chromatography and spectrophotometry) at isothermal conditions. Spedding *et al.*<sup>52</sup> used the isopiestic technique. Harkot<sup>16</sup> analyzed the saturated solutions after polythermal crystallization. Friend and Hale<sup>80</sup> recorded the temperature of the last crystal dissolution. Thermal analysis, used by Sokolova<sup>10</sup> and Voigt,<sup>137</sup> allowed construction of the LaCl<sub>3</sub>-H<sub>2</sub>O equilibrium phase diagram on the water-rich side.

LaCl<sub>3</sub>·10H<sub>2</sub>O [117973-45-2], LaCl<sub>3</sub>·9H<sub>2</sub>O [117973-40-7], LaCl<sub>3</sub>·7H<sub>2</sub>O [10025-84-0], LaCl<sub>3</sub>·6H<sub>2</sub>O [17272-45-6] equilibrium solid phases were found<sup>10</sup> to exist up to 239, 250, 367 and 394 K, respectively. According to Ref. 137, existence of LaCl<sub>3</sub>·6H<sub>2</sub>O [17272-45-6] is questionable and this phase should rather be treated as metastable. Temperature of melting or the peritectic decomposition of LaCl<sub>3</sub>·7H<sub>2</sub>O was reported as 368 K in Refs. 82, 127 and 129, 364 K in Refs. 35, 362 K in Ref. 155, 365 K in Ref. 153, 364.6±1.4 K in Ref. 137 and 367±1 K in Refs. 10 and 75. The result of Ref. 137 was the mean value of many of the author's measurements and therefore it seems to be the most precise and is selected in this evaluation; the author of the report showed that LaCl<sub>3</sub>·7H<sub>2</sub>O melts congruently in contrast to the finding of Ref. 10. The much too low value of 330-332 K, given as the transition of solid into liquid LaCl<sub>3</sub>·7H<sub>2</sub>O in Ref. 154, seems to correspond to non-equilibrium conditions in the experiments. Several overestimated results of the melting point of LaCl<sub>3</sub>·7H<sub>2</sub>O were also published: 374.2 K in Ref. 156, 373 K in Ref. 74 and 383 K in Ref. 77. The melting temperature of LaCl<sub>3</sub>·6H<sub>2</sub>O was reported to be 393 K in Refs. 10 and 53, but the authors did not describe exact analyses of this compound.

According to many dehydration studies, water is released from LaCl<sub>3</sub>·7H<sub>2</sub>O gradually with the formation of LaCl<sub>3</sub>·3H<sub>2</sub>O [35564-84-2],<sup>68,71,82,137,145,157-160,162-165</sup> LaCl<sub>3</sub>·H<sub>2</sub>O [51305-40-9],<sup>68,71,82,137,145,157,158,160-165</sup> and finally anhydrous LaCl<sub>3</sub><sup>27,82,145,156-158,161-165</sup> at increasing temperatures, decreasing pressure or action of a dehydrating agent; a formation of LaCl<sub>3</sub>·6H<sub>2</sub>O,<sup>10,53,157</sup> LaCl<sub>3</sub>·5H<sub>2</sub>O [87720-86-3],<sup>158</sup> LaCl<sub>3</sub>·4H<sub>2</sub>O [10555-78-9]<sup>53</sup> and LaCl<sub>3</sub>·2H<sub>2</sub>O [87720-87-4]<sup>158,165</sup> intermediate forms were sometimes reported but generally not accepted. Many authors<sup>27,71,74,82,156,161</sup> observed formation of LaOCl as the final product of dehydration or as a by-product of anhydrous LaCl<sub>3</sub> formation.

Crystal structure of LaCl<sub>3</sub>·7H<sub>2</sub>O was investigated in Refs. 145, 153, 166-170 and of LaCl<sub>3</sub>·3H<sub>2</sub>O in Ref. 11. Authors of Ref. 53 investigated LaCl<sub>3</sub>·6H<sub>2</sub>O crystals by x-ray diffraction but no conclusions were drawn. Triclinic LaCl<sub>3</sub>·6H<sub>2</sub>O, reported in Ref. 3, should be treated in fact as LaCl<sub>3</sub>·7H<sub>2</sub>O.

All these experimental achievements were taken into account for construction of the refined water-rich part of the LaCl<sub>3</sub>-H<sub>2</sub>O phase diagram. Such a partial diagram is presented in Fig. 3.

[Figure 3. Water-rich part of the LaCl<sub>3</sub>-H<sub>2</sub>O equilibrium phase diagram]

The solubility data reported exclusively for the LaCl<sub>3</sub>-H<sub>2</sub>O binary system are the most valuable for this critical evaluation. The remaining solubilities compiled and reviewed here are relevant mostly to ternary systems containing LaCl<sub>3</sub> as one of components. In many such studies, the solubility of LaCl<sub>3</sub> in water was measured once by a given research group, prior to a series of papers, and this value was repeated in all subsequent studies by that group.

Thus the data reported for the ternary systems are generally regarded as somewhat less reliable.

All solubility results for the  $\text{LaCl}_3\text{-H}_2\text{O}$  system are collected in Table 4.

[Table 4. Experimental solubilities reported for the  $\text{LaCl}_3\text{-H}_2\text{O}$  system as a function of temperature]

Thus, the solubility of  $\text{LaCl}_3$  in water was observed to increase gradually up to 250 K, then it increased only slightly up to about 320 K and strongly up to the melting point of  $\text{LaCl}_3\cdot 7\text{H}_2\text{O}$ . Further increase of the solubility, when  $\text{LaCl}_3\cdot 3\text{H}_2\text{O}$  was the equilibrium phase, was relatively significant, but the solubility of  $\text{LaCl}_3\cdot \text{H}_2\text{O}$  was practically independent of temperature and the results in this range may be attributed to the formation of the oxychloride.

The data from the studies by Petelina *et al.*,<sup>43,87</sup> Harkot,<sup>16</sup> Nikolaev *et al.*,<sup>112</sup> Alieva and Sulaimankulov,<sup>92</sup> Zholalieva *et al.*,<sup>108,113</sup> Zelikman *et al.*,<sup>45</sup> Shi *et al.*,<sup>130</sup> Chen *et al.*,<sup>151</sup> Li *et al.*<sup>139</sup> and Wang *et al.*<sup>152</sup> are excluded from further evaluation. Probably, no equilibrium was achieved in their experimental conditions (as in the experimental conditions of Ref. 16), or the errors were connected with the analytical procedures used. No doubt, the most precise and accurate solubility data of  $\text{LaCl}_3$  in water were obtained by Powell and Burkholder<sup>82</sup> between 273 and 363 K (but no error limits were reported), whose results were confirmed by the less precise determinations of Brunisholz and Nozari,<sup>86</sup> Zhuravlev *et al.*,<sup>94,98-103,105,107</sup> Sheveleva *et al.*,<sup>96</sup> Tang *et al.*<sup>118-122</sup> ( $\pm 0.2\%$  and  $\pm 0.2\text{ K}$ ), Shevtsova *et al.*,<sup>42,81,83</sup> Nikolaev *et al.*<sup>44,54,55,104,110</sup> ( $\pm [2-3]\%$  and  $\pm 0.5\text{ K}$ ), Sergeeva and Karapetyants,<sup>106</sup> Kost *et al.*,<sup>114,116,125-127,129</sup> Zwietasch *et al.*,<sup>123</sup> Berecz and Török,<sup>132</sup> Friend and Hale<sup>80</sup> at  $>323\text{ K}$  ( $\pm$  "several" % and  $\pm 0.2\text{ K}$ ), Volkov *et al.*,<sup>90</sup> Sokolova<sup>10</sup> at 273-322 K ( $\pm 0.5\%$  and  $\pm 1\text{ K}$ ), Voigt<sup>137</sup> ( $\pm 0.2\%$  and  $\pm 0.5\text{ K}$ ), Chen *et al.*,<sup>131</sup> Xu *et al.*,<sup>133</sup> Liang *et al.*,<sup>140</sup> Cui *et al.*,<sup>141</sup> Liu *et al.*,<sup>143,145</sup> Ren *et al.*<sup>147</sup> and the especially precise values obtained by Spedding *et al.*<sup>18,51,52,111</sup> at 298.15 K ( $\pm 0.1\%$  and  $\pm 0.02\text{ K}$ ).

The liquidus points obtained by Sokolova<sup>10</sup> at temperatures lower than 273 K may be treated as the tentative solubilities of the corresponding phases; however, the results at 367 and 394 K should be placed at significantly more salt-rich values. It seems that the phase relations established by Voigt<sup>137</sup> are much more convincing and reliable in this temperature range.

Due to insufficient data, no solubility equations may be formulated below 250 K when the equilibrium solid phase is either  $\text{LaCl}_3\cdot 10\text{H}_2\text{O}$  or  $\text{LaCl}_3\cdot 9\text{H}_2\text{O}$ . For  $\text{LaCl}_3\cdot 7\text{H}_2\text{O}$ , as the equilibrium solid phase between 250 and 365 K, one may describe the solubility behavior based on the general form (see Eq. (3) in Sec. 1) by fitting the equation to the most precise experimental data<sup>10,18,51,52,82,111,132,137,151</sup> and get the following parameters:

$$\ln\{x_1^4(1-x_1)^7(4+7)^{4+7}7^{-7}[1+(4-1)x_1]^{-(4+7)}\}=188.251-3771.3T^{-1}-35.139\ln T+0.08053T. \quad (6)$$

For  $\text{LaCl}_3\cdot 3\text{H}_2\text{O}$  between 353 and 448 K, the following solubility equation was fitted to the experimental data of Ref. 137:

$$\ln\{x_1^4(1-x_1)^3(4+3)^{4+3}3^{-3}[1+(4-1)x_1]^{-(4+3)}\} = -99.661 + 3727.6T^{-1} - 15.764\ln T - 0.01104T. \quad (7)$$

The constants in both equations were obtained by a fitting procedure with the least-square method using the selected solubility results from Table 4. The solubility values obtained for  $\text{LaCl}_3 \cdot \text{H}_2\text{O}$  as the equilibrium solid phase were not approximated with a solubility equation.

The recommended, tentative and doubtful solubilities of  $\text{LaCl}_3$  in water, which were selected by the compilers and obtained from the solubility equations, are collected in Table 5.

[Table 5. Recommended (R), tentative (T) and doubtful (D) solubilities of  $\text{LaCl}_3$  in  $\text{H}_2\text{O}$  at selected temperatures; the data between 250 and 493 K were confirmed by the fitting equations]

Harkot<sup>16,17</sup> investigated the polythermal crystallization process of  $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$  in water in the temperature range 291.2 to 362.9 K and detected formation of polycondensation type structures of the solute in the saturated solution that deviated slightly from the salt stoichiometry. Due to the large scatter of the solubility data obtained in Ref. 16 and 17, one should be cautious about the quantitative conclusions drawn by this author.

Powell and Burkholder<sup>82</sup> measured metastable solubilities of  $\text{LaCl}_3 \cdot 6\text{H}_2\text{O}$  in the temperature range 313-345.7 K; these values have a tentative character. The solubilities of  $\text{LaCl}_3 \cdot 6\text{H}_2\text{O}$  were always higher than those of  $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$ .

The solubilities of  $\text{LaCl}_3$  in HCl solutions were measured in several studies<sup>23,42,45,80,132,142,148,149,151</sup> as a function of HCl concentration and temperature. As pointed out by a summary,<sup>132</sup> the solubilities decreased smoothly with increasing HCl concentration at all temperatures between 273 and 363 K, however at the highest HCl concentrations a small increase of the solubility was observed. Scatter of the experimental solubility results from all sources increased from only a few % in water to about 15 % at the highest HCl contents,<sup>12</sup> indicating that the data related to the ternary systems are less precise. The equilibrium solid phase identified at these experimental conditions was independently found to be  $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$ .

The solubilities of  $\text{LaCl}_3$  in solutions of acetic acid at 303 K were unexpectedly found to be independent of the acetic acid concentration (up to 49 mass %) when the solubilities were expressed in molal concentrations.<sup>138</sup> This may suggest that the same solvate (hydrate) of  $\text{LaCl}_3$  may exist in the whole range of acetic acid concentrations.

The ternary  $\text{LaCl}_3\text{-MCl}_3\text{-H}_2\text{O}$  systems, with M being: Li,<sup>84,129</sup> Na,<sup>81,85,115,126,127</sup> K,<sup>83,89,125,126</sup>  $\text{NH}_4$ ,<sup>83,114,125</sup> Rb<sup>96,116</sup> or Cs,<sup>91,126,142</sup> were investigated. The first four systems were found to be of a simple eutonic type. One congruently soluble salt was formed in the  $\text{LaCl}_3\text{-RbCl}_3\text{-H}_2\text{O}$  system and two salts in the case of the  $\text{LaCl}_3\text{-CsCl}_3\text{-H}_2\text{O}$  system. No double chloride formation was seen in the quaternary systems  $\text{LaCl}_3\text{-LiCl}_3\text{-KCl}_3\text{-H}_2\text{O}$ ,<sup>97</sup>  $\text{LaCl}_3\text{-NaCl}_3\text{-NH}_4\text{Cl}_3\text{-H}_2\text{O}$ <sup>115</sup> and  $\text{LaCl}_3\text{-KCl}_3\text{-NH}_4\text{Cl}_3\text{-H}_2\text{O}$ ,<sup>115</sup> however, two double chlorides were isolated in the

LaCl<sub>3</sub>-NaCl-CsCl-H<sub>2</sub>O system.<sup>91</sup> The corresponding details may be found in the respective data compilations.

The ternary LaCl<sub>3</sub>-MCl<sub>2</sub>-H<sub>2</sub>O systems, with M being Mg,<sup>110</sup> Ca,<sup>81,90</sup> Sr<sup>90</sup> and Ba,<sup>90</sup> were reported to be of a simple eutonic type. The formation of two incongruently soluble double chlorides was observed in the LaCl<sub>3</sub>-CdCl<sub>2</sub>-H<sub>2</sub>O system.<sup>152</sup> In the case of the quaternary LaCl<sub>3</sub>-CdCl<sub>2</sub>-HCl (9.7 mass %)-H<sub>2</sub>O<sup>148</sup> as well as the LaCl<sub>3</sub>-ZnCl<sub>2</sub>-HCl (7 mass %)-H<sub>2</sub>O<sup>149</sup> systems, two congruently soluble double chlorides and three double chlorides were reported to be formed, respectively. Formation of three double chlorides was observed in the quaternary LaCl<sub>3</sub>-CsCl-HCl (13, 23 %)-H<sub>2</sub>O and LaCl<sub>3</sub>-CsCl-acetic acid (42 %)-H<sub>2</sub>O systems.<sup>142</sup>

Ternary LaCl<sub>3</sub>-LnCl<sub>3</sub>-H<sub>2</sub>O systems have been intensively studied by the group of Nikolaev *et al.*, with Ln being: Y,<sup>54</sup> Ce,<sup>40,41</sup> Eu<sup>44</sup> and Yb<sup>104</sup> at 298 K, as well as by Brunisholz and Nozari<sup>86</sup> for Nd and Sm at 293 K. The quaternary systems LaCl<sub>3</sub>-NdCl<sub>3</sub>-SmCl<sub>3</sub>-H<sub>2</sub>O<sup>86</sup> and LaCl<sub>3</sub>-CeCl<sub>3</sub>-EuCl<sub>3</sub>-H<sub>2</sub>O<sup>54,93</sup> were also investigated at 293 K and 298 K, respectively. It was found that no double salts were formed in these systems. If the second Ln chloride also formed a heptahydrate, solid solutions or mixed crystals (La,Ln)Cl<sub>3</sub>·7H<sub>2</sub>O were the equilibrium solid phases. However, if Ln chloride formed a hexahydrate, a discontinuity between two forms, (La,Ln)Cl<sub>3</sub>·7H<sub>2</sub>O and (Ln,Ln)Cl<sub>3</sub>·6H<sub>2</sub>O, was observed at certain salt compositions.

Mixed crystals were also found in the ternary LaCl<sub>3</sub>-LaBr<sub>3</sub>-H<sub>2</sub>O system.<sup>123</sup> A double salt of formula LaCl<sub>3</sub>·LaF<sub>3</sub>·9H<sub>2</sub>O was identified in the LaCl<sub>3</sub>-LaF<sub>3</sub>-H<sub>2</sub>O system.<sup>53</sup>

Ternary systems containing LaCl<sub>3</sub>, H<sub>2</sub>O and the following organic compounds have been investigated: acetic acid,<sup>138</sup> urea,<sup>92</sup> methylamine hydrochloride,<sup>94</sup> trimethylamine hydrochloride,<sup>94</sup> hydrazine dihydrochloride,<sup>95</sup> ethanediamine dihydrochloride,<sup>95</sup> hydroxyloamine hydrochloride,<sup>98</sup> dimethylamine hydrochloride,<sup>98</sup> hexamethylenediamine dihydrochloride,<sup>99</sup> piperazine dihydrochloride,<sup>100</sup> 1,2-ethanediamine-N,N,N',N'-teramethyl dihydrochloride,<sup>100</sup> 1,2-benzenediamine dihydrochloride,<sup>100</sup> 1,3-benzenediamine dihydrochloride,<sup>100</sup> triethylamine hydrochloride,<sup>102</sup> triethanolamine hydrochloride,<sup>102</sup> diethylamine hydrochloride,<sup>103</sup> diethanolamine hydrochloride,<sup>103</sup> butylamine hydrochloride,<sup>102</sup> aniline hydrochloride,<sup>105</sup> piperidine hydrochloride,<sup>105</sup> hexamethyleneimine hydrochloride,<sup>105</sup> acetone,<sup>106</sup> pyridine hydrochloride,<sup>107</sup> quinoline hydrochloride,<sup>107</sup> acetamide,<sup>108,120</sup> choline chloride,<sup>109</sup> acetylurea,<sup>113,121</sup> thiourea,<sup>113,118</sup> 1-acetyl-2-thiourea,<sup>122</sup> hexamethylenetetramine hydrochloride,<sup>119</sup> 2,3-dimethyl-1-phenyl-3-pyrazolin-5-one,<sup>124</sup> phthalimidoantipyrine,<sup>150</sup> phenylalanine,<sup>147</sup> 4-(4-chlorobenzoyl)aminoantipyrine,<sup>146</sup> fluorouracil,<sup>145</sup> N,N'-bis(antipyrine-4-yl)oxalamide,<sup>144</sup> NN'-bis(antipyrine-4'-yl)-hexenedicarboxamide,<sup>141</sup> phthalimidoantipyrine,<sup>140</sup> 4-chloroacetylantipyrine,<sup>133</sup> glycine,<sup>131</sup> glutamic acid,<sup>131</sup> serine<sup>131,143</sup> and nicotinamide.<sup>130</sup> Details related to formation of complex compounds, by the compounds mentioned above with LaCl<sub>3</sub>, may be found in the corresponding data sheets.



## References

- <sup>10</sup> N. P. Sokolova, Radiokhimiya **30**, 435 (1988).
- <sup>12</sup> C. Gumiński, Arch. Metall. Mater. **51**, 617 (2006).
- <sup>16</sup> J. Harkot, Polish J. Chem. **63**, 337 (1989).
- <sup>17</sup> J. Harkot, Polish J. Chem. **64**, 53 (1990).
- <sup>18</sup> F. H. Spedding, J. A. Rard and V. Saeger, J. Chem. Eng. Data **19**, 373 (1974).
- <sup>23</sup> W. Fischer, H. Bauer, I. Dillo, T. Molaug, J. Nier, H. Rohrer and K. Trovaag, Z. Anorg. Chem. **357**, 177 (1968).
- <sup>40</sup> A. V. Nikolaev, A. A. Sorokina and V. G. Tsubanov, Dokl. Akad. Nauk SSSR **172**, 1333 (1967).
- <sup>41</sup> A. V. Nikolaev, A. A. Sorokina, A. Ya. Vilenskaya and V. G. Tsubanov, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 6, 5 (1967).
- <sup>42</sup> Z. N. Shevtsova, L. S. Nam and B. G. Korshunov, Zh. Neorg. Khim. **13**, 1682 (1968).
- <sup>43</sup> V. S. Petelina, N. I. Nikurashina and G. V. Illarionova, Issledovaniya v Oblasti Khimii Redkozemelnykh Elementov, Saratovskii Universitet, Saratov **3**, 48 (1971).
- <sup>44</sup> A. V. Nikolaev and A. A. Sorokina, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 4, 61 (1971).
- <sup>45</sup> A. N. Zelikman, N. V. Baryshnikov and A. I. Khokhlov, Zh. Neorg. Khim. **16**, 2023 (1971).
- <sup>51</sup> F. H. Spedding, V. W. Saeger, K. A. Gray, P. K. Boneau, M. A. Brown, C. W. DeKock, J. L. Baker, L. E. Shiers, H. O. Weber and A. Habenschuss, J. Chem. Eng. Data **20**, 72 (1975).
- <sup>52</sup> F. H. Spedding, H. O. Weber, V. W. Saeger, H. H. Petheram, J. A. Rard and A. Habenschuss, J. Chem. Eng. Data **21**, 341 (1976).
- <sup>53</sup> N. Levina, G. Dadabaeva, D. D. Ikrami and A. S. Paramzin, Dep. VINITI N-478-77, 27 pp. (1977).
- <sup>54</sup> A. V. Nikolaev, A. A. Sorokina, V. N. Lubkova and N. G. Yudina, *Osnovy Vzaimodeistviya Solei Redkozemelnykh Elementov v Vode* (Nauka, Novosibirsk, 1977), p. 36, 45, 46, 53, 72, 79, 161.
- <sup>55</sup> A. V. Nikolaev, A. A. Sorokina, N. P. Sokolova, G. S. Kotlyar-Shapiro, N. P. Anoshina, L. I. Bagryantseva and A. P. Olovyanishnikova, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 4, 84 (1977).
- <sup>74</sup> L. G. Sokolova, A. V. Lapitskaya, A. F. Bolshakov, S. B. Pirkes and B. V. Abalduev, Zh. Neorg. Khim. **26**, 1736 (1981); B. V. Abalduev, A. F. Bolshakov, L. G. Sokolova, A. V. Lapitskaya and S. B. Pirkes, Issledovaniya v Oblasti Khimii Redkozemelnykh Elementov, Saratovskii Universitet, Saratov, 1975, p. 47.
- <sup>75</sup> W. W. Wendlandt and R. G. Sewell, Texas J. Sci. **8**, 231 (1961).
- <sup>77</sup> W. W. Wendlandt and J. L. Bear, Anal. Chim. Acta **21**, 439 (1959).
- <sup>79</sup> W. Prandtl and J. Rauchenberger, Ber. Dtsch. Chem. Ges. **53**, 843 (1920).
- <sup>80</sup> J. N. Friend and R. W. Hale, J. Chem. Soc. **1940**, 670.

- <sup>81</sup> Z. N. Shevtsova, V. S. Zelova and L. I. Ushakova, Nauch. Dokl. Vyssh. Shkoly, Khim. Khim. Tekhnol. **3**, 417 (1958).
- <sup>82</sup> J. E. Powell and H. R. Burkholder, J. Inorg. Nucl. Chem. **14**, 65 (1960).
- <sup>83</sup> Z. N. Shevtsova, L. I. Zhizhina and L. E. Eltsberg, Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol. **4**, 176 (1961).
- <sup>84</sup> E. N. Khutorskoi and A. D. Sheveleva, Ucheb. Zap. Perm. Univ. **178**, 49 (1968).
- <sup>85</sup> E. N. Khutorskoi and A. D. Sheveleva, Ucheb. Zap. Perm. Univ. **178**, 57 (1968).
- <sup>86</sup> G. Brunisholz and M. Nozari, Helv. Chim. Acta **52**, 2303 (1969).
- <sup>87</sup> V. S. Petelina, R. V. Mertslin, N. I. Nikurashina and L. K. Sedova, Issledovaniya v Oblasti Khimii Redkozemelnykh Elementov, Saratovskii Universitet, Saratov, **1969**, p. 85.
- <sup>88</sup> Z. F. Alieva, O. V. Agashkin and K. Sulaimankulov, Izv. Akad. Nauk Kirgiz. SSR, no 5, 38 (1970).
- <sup>89</sup> E. N. Khutorskoi and A. D. Sheveleva, Ucheb. Zap. Perm. Univ. **207**, 11 (1970).
- <sup>90</sup> A. A. Volkov, M. P. Borodina and A. I. Shapiro, Ucheb. Zap. Perm. Univ. **229**, 3 (1970).
- <sup>91</sup> A. D. Sheveleva, N. A. Torgashina and E. N. Khutorskoi, Ucheb. Zap. Perm. Univ. **229**, 21 (1970).
- <sup>92</sup> Z. F. Alieva and K. S. Sulaimankulov, Izv. Akad. Nauk Kaz. SSR, Ser. Khim. **20**, no 6, 16 (1970).
- <sup>93</sup> A. V. Nikolaev, A. A. Sorokina, N. G. Yudina and V. N. Lubkova, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 5, 35 (1972).
- <sup>94</sup> E. F. Zhuravlev, R. V. Yurkevich and N. G. Kalegina, Zh. Neorg. Khim. **17**, 2536 (1972).
- <sup>95</sup> R. V. Yurkevich and E. F. Zhuravlev, Zh. Neorg. Khim. **17**, 3063 (1972).
- <sup>96</sup> A. D. Sheveleva, K. I. Mochalov, E. N. Khutorskoi and N. A. Torgashina, Ucheb. Zap. Perm. Univ. **289**, 3 (1973).
- <sup>97</sup> A. D. Sheveleva, K. I. Mochalov and N. A. Torgashina, Ucheb. Zap. Perm. Univ. **289**, 15 (1973).
- <sup>98</sup> E. F. Zhuravlev, R. V. Yurkevich and N. G. Kalegina, Zh. Neorg. Khim. **18**, 1109, (1973).
- <sup>99</sup> E. F. Zhuravlev, R. V. Yurkevich, A. G. Samatov and L. F. Shlentova, Zh. Neorg. Khim. **18**, 2247 (1973).
- <sup>100</sup> E. F. Zhuravlev, R. V. Yurkevich and N. A. Rasskazova, Zh. Neorg. Khim. **19**, 534 (1974).
- <sup>101</sup> E. F. Zhuravlev, R. V. Yurkevich and N. A. Rasskazova, Zh. Neorg. Khim. **19**, 1977 (1974).
- <sup>102</sup> E. F. Zhuravlev, R. V. Yurkevich and N. G. Kalegina, Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol. **17**, 498 (1974).
- <sup>103</sup> E. F. Zhuravlev, R. V. Yurkevich and N. A. Rasskazova, Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol. **17**, 1123 (1974).
- <sup>104</sup> A. V. Nikolaev, A. A. Sorokina and N. L. Kozyreva, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 6, 86 (1974).

- <sup>105</sup> R. V. Yurkevich and E. F. Zhuravlev, *Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol.* **18**, 140 (1975).
- <sup>106</sup> Ts. B. Sergeeva and M. Kh. Karapetyants, *Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol.* **19**, 846 (1976).
- <sup>107</sup> R. V. Yurkevich, E. F. Zhuravlev and N. A. Rasskazova, *Zh. Neorg. Khim.* **21**, 2234 (1976).
- <sup>108</sup> Z. Zholaliev, K. Sulaimankulov and K. Noguev, *Zh. Neorg. Khim.* **21**, 2290 (1976).
- <sup>109</sup> E. F. Zhuravlev, R. V. Yurkevich and N. A. Rasskazova, *Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol.* **19**, 1297 (1976).
- <sup>110</sup> A. A. Sorokina, N. P. Sokolova, G. S. Kotlyar-Shapiro and P. A. Stabnikov, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 4, 91 (1977).
- <sup>111</sup> F. H. Spedding, C. W. DeKock, G. W. Pepple and A. Habenschuss, *J. Chem. Eng. Data* **22**, 58 (1977).
- <sup>112</sup> A. V. Nikolaev, A. A. Sorokina, N. P. Sokolova, G. S. Kotlyar-Shapiro and L.I. Bagryantseva, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 1, 46 (1978).
- <sup>113</sup> Z. N. Zholaliev and K. Sulaimankulov, *Zh. Neorg. Khim.* **23**, 1367 (1978).
- <sup>114</sup> L. L. Kost, Yu. V. Shirai and V. G. Shevchuk, *Zh. Neorg. Khim.* **25**, 579 (1980).
- <sup>115</sup> V. G. Shevchuk and Yu. V. Shirai, *Zh. Neorg. Khim.* **25**, 843 (1980).
- <sup>116</sup> Yu. V. Shirai and V. G. Shevchuk, *Zh. Neorg. Khim.* **26**, 1940 (1981).
- <sup>117</sup> G. O. Karapetyan, B. I. Spesivtsev, N. O. Khvostova and O. V. Yanush, *SPSTL Dep. Doc.* 916 Khp-D81, p. 188 (1981); *Chem. Abstr.* **98**, 78951d (1982).
- <sup>118</sup> Z. X. Tang, Zh. Zh. Guo, P. H. Chen, J. H. Ma and Y. Sh. Chen, *Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.)* **3**, 447 (1982).
- <sup>119</sup> Z. X. Tang and Y. Sh. Chen, *Zhongguo Xitu Xuebao (J. Chin. Rare Earth Soc.)* **1**, 43 (1983).
- <sup>120</sup> Z. X. Tang, Zh. Zh. Guo, P. H. Chen, J. H. Ma and Y. Sh. Chen, *Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.)* **4**, 419 (1983).
- <sup>121</sup> Z. X. Tang, Zh. Zh. Guo, P. H. Chen, J.H. Ma and Y.Sh. Chen, *Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.)* **4**, 631 (1983).
- <sup>122</sup> Z. X. Tang, Zh. Zh. Guo, P.H. Chen, J. H. Ma and Y. Sh. Chen, *Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.)* **4**, 634 (1983).
- <sup>123</sup> K. J. Zwietasch, E. M. Kirmse and I. Krech, *Z. Chem.* **24**, 144 (1984).
- <sup>124</sup> Y. Zh. Zou, Z. X. Tang and Y. Sh. Chen, *Huaxue Xuebao (Acta Chim. Sinica)* **42**, 913 (1984).
- <sup>125</sup> D. A. Storozhenko, Yu. V. Shirai and N. M. Lazorenko, *Zh. Neorg. Khim.* **31**, 537 (1986).
- <sup>126</sup> Yu. V. Shirai, *Zh. Neorg. Khim.* **31**, 1049 (1986).
- <sup>127</sup> D. A. Storozhenko, Yu. V. Shirai and L. M. Lazorenko, *Zh. Neorg. Khim.* **31**, 1610 (1986).
- <sup>128</sup> H. Kanno and Y. Akama, *J. Phys. Chem.* **91**, 1263 (1987).

- <sup>129</sup> D. A. Storozhenko, Yu. V. Shirai and N. F. Eskova, Zh. Neorg. Khim. **33**, 2438 (1988).
- <sup>130</sup> Q. H. Shi, J. Q. Chen and L. L. Wang, Huaxue Shijie **29**, 484 (1988).
- <sup>131</sup> J. X. Chen, X. Q. Ran, Zh. Zh. Guo and Y. Sh. Chen, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) **11**, 555 (1990).
- <sup>132</sup> E. Berecz and T. I. Török, Z. Anorg. Chem. **583**, 177 (1990).
- <sup>133</sup> X. L. Xu, Z. X. Tang, Zh. Zh. Guo and Y. Sh. Chen, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) **12**, 728 (1991).
- <sup>134</sup> D. H. Ren, J. Zhang, J. Ch. Sun, C. M. Yin and Z. R. Liu, Wuji Huaxue Xuebao (Chin. J. Inorg. Chem.) **8**, 209 (1992).
- <sup>135</sup> K. Fukushi, A. Mutoh, K. Ishikawa and Y. Suzuki, J. Alloys Comp. **192**, 290 (1993).
- <sup>136</sup> K. Fukushi, K. Iwamoto, K. Kobayashi, K. Ishikawa and Y. Suzuki, J. Alloys Comp. **207/208**, 468 (1994).
- <sup>137</sup> H. Voigt, *Thermodynamische, thermoanalytische und strukturelle Untersuchungen an Lanthanhalogenid-Hydraten*, Rep. Vo5112/1-2 (Bergakademie, Freiberg, Germany, 1994).
- <sup>138</sup> Q. D. Li, H. F. Xue and D. H. Ren, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) **8**, 1172 (1995).
- <sup>139</sup> Y. H. Li, X. Q. Ran and P. H. Chen, Yan Hu Yan Jiu (J. Salt Lake Sci.) **3**, no 2, 40 (1995).
- <sup>140</sup> H. B. Liang, Sh. J. Li, G. L. Gao, N. H. Cao and Z. X. Tang, Shaanxi Shifan Daxue Xuebao, Ziran Kexueban (J. Shaanxi Normal Univ., Natur. Sci.) **25**, no 3, 61 (1997).
- <sup>141</sup> B. Cui, Z. X. Tang, Zh. Zh. Guo and K. X. Chen, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) **18**, 1742 (1997).
- <sup>142</sup> Y. H. Li, X. Q. Ran and P. H. Chen, Zhongguo Xitu Xuebao (J. Chin. Rare Earth Soc.) **15**, no 2, 102 (1997); J. Rare Earths **15**, no 2, 113 (1997).
- <sup>143</sup> J. R. Liu, M. Ji, Zh. G. Li and Sh. G. Gao, Xibei Daxue Xuebao, Ziran Kexueban (J. Northwest Univ., Natur. Sci.) **28**, no 1, 44 (1998).
- <sup>144</sup> H. B. Liang, W. H. Han, G. L. Gao, D. J. Zhang and Q. Bo, Shaanxi Shifan Daxue Xuebao, Ziran Kexueban (J. Shaanxi Normal Univ., Natur. Sci.) **27**, no 1, 73 (1999).
- <sup>145</sup> Sh. P. Liu, G. Q. Huang, Ch. D. Zhang and H. Y. Wang, Neimenggu Daxue Xuebao, Ziran Kexueban (Acta Sci. Natur. Univ. NeMongol) **30**, no 1, 53 (1999).
- <sup>146</sup> H. B. Liang, W. L. Wang, D. D. Hu and W. P. Zhang, Zhongguo Xitu Xuebao **17**, no 1, 81 (1999); J. Rare Earths **17**, no 1, 64 (1999).
- <sup>147</sup> F. Ren, Sh. L. Gao and Q. Zh. Shi, Xiamen Daxue Xuebao, Ziran Kexueban (J. Xiamen Univ., Natur. Sci.) **39**, 485 (2000).
- <sup>148</sup> L. Li, H. Wang, Sh. P. Xia, M. Ch. Hu and Sh. Y. Gao, Wuji Huaxue Xuebao (Chin. J. Inorg. Chem.) **19**, 201 (2003).
- <sup>149</sup> Zh. P. Qiao, L. H. Zhuo, Sh. Sh. Zhang and H. Wang, Wuji Huaxue Xuebao (Chin. J. Inorg. Chem.) **19**, 303 (2003).

- <sup>150</sup> Zh. F. Hao, H. B. Liang, Z. X. Tang, L. Yu, J. Yu and Y. W. Fang, *Huaxue Xuebao (Acta Chim. Sinica)* **61**, 853 (2003).
- <sup>151</sup> Y. G. Chen, Zh. Fang, Q. R. Zhang, K. Y. Yuan and Sh. F. Wang, *Xiyou Jinshu (Chin. J. Rare Met.)* **27**, 601 (2003).
- <sup>152</sup> H. Wang, X. F. Wang, F. X. Dong, X. Q. Ran, L. Li and Sh. Y. Gao, *J. Chem. Eng. Data* **52**, 731 (2007).
- <sup>153</sup> A. Habenschuss and F. H. Spedding, *Cryst. Struct. Commun.* **8**, 511 (1979).
- <sup>154</sup> N. P. Sokolova and E. A. Ukraintseva, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 1, 24 (1984).
- <sup>155</sup> J. G. Tang, C. K. S. Lee and L. A. Belfiore, *J. Polym. Sci, Part B: Polym. Phys.* **41**, 2200 (2003).
- <sup>156</sup> H. B. Chen, P. Zh. Yang, Ch. Y. Zhou, Ch. Y. Jiang and J. G. Pan, *Cryst. Growth Design* **6**, 809 (2006).
- <sup>157</sup> A. N. Zelikman, N. V. Baryshnikov, A. N. Khokhlov, *Izv. Vyssh. Ucheb. Zaved., Tsvetn. Metall.*, no 6, 89 (1971).
- <sup>158</sup> Zh. Gong, P. H. Chen, Zh. Zh. Guo, J. H. Ma and Y. Sh. Chen, *Huaxue Xuebao (Acta Chim. Sinica)* **41**, 391 (1983).
- <sup>159</sup> N. P. Sokolova and E. A. Ukraintseva, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 1, 24 (1984).
- <sup>160</sup> V. V. Hong and D. V. Minh, *Tap Chi Hoa Hoc (J. Chem.)* **36**, no 4, 44 (1998).
- <sup>161</sup> G. C. Zhu, F. P. Li and M. G. Xiao, *Trans. Nonferrous Met. Soc. China* **13**, 1454 (2003).
- <sup>162</sup> Zh. Gong, P. H. Chen, Zh. Zh. Guo, J. H. Ma and Y. Sh. Chen, *Zhongguo Xitu Xuebao (J. Chin. Rare Earth Soc.)* **3**, no 2, 13 (1985).
- <sup>163</sup> Zh. Gong, P. H. Chen, Zh. Zh. Guo, J. H. Ma and Y. Sh. Chen, *Huaxue Xuebao (Acta Chim. Sinica)* **41**, 463 (1983).
- <sup>164</sup> Zh. Gong, P. H. Chen, Zh. Zh. Guo, J. H. Ma and Y. Sh. Chen, *Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.)* **4**, 493 (1983).
- <sup>165</sup> S. J. Ashcroft and C. T. Mortimer, *J. Less-Comm. Met.* **14**, 403 (1968).
- <sup>166</sup> V. I. Ivernova, V. P. Tarasova and M. M. Umanskii, *Izv. Akad. Nauk SSSR, Ser. Fiz.* **15**, 164 (1951).
- <sup>167</sup> J. H. Burns and J. R. Peterson, *Inorg. Chem.* **10**, 147 (1971).
- <sup>168</sup> C. Brouty and P. Herpin, *Compt. Rend. C* **272**, 2049 (1971).
- <sup>169</sup> V. V. Bakakin, R. F. Klevtsova and L. P. Soloveva, *Zh. Strukt. Khim.* **15**, 820 (1974).
- <sup>170</sup> C. J. Kepert, B. W. Skelton and A. H. White, *Austr. J. Chem.* **47**, 385 (1994).

## 4.2. Data for the LaCl<sub>3</sub>-H<sub>2</sub>O System

<b>Components:</b>	<b>Original Measurements:</b>
(1) Lanthanum chloride; LaCl <sub>3</sub> ; [10099-58-8]	<sup>10</sup> N.P. Sokolova, Radiokhimiya <b>30</b> , 435
(2) Water; H <sub>2</sub> O; [7732-18-5]	(1988).
<b>Variables:</b>	<b>Prepared by:</b>
Composition: 0–66.5 mass % LaCl <sub>3</sub>	C. Gumiński

### Experimental Values

Melting temperatures of LaCl<sub>3</sub>-H<sub>2</sub>O mixtures as read from a figure and recalculated to mol % by the compiler

100w <sub>1</sub>	100x <sub>1</sub>	t / °C	Equilibrium solid phase
59.0	9.56	121 <sup>a</sup>	LaCl <sub>3</sub> ·6H <sub>2</sub> O + LaCl <sub>3</sub> ·nH <sub>2</sub> O (n<6)
53.0 <sup>a</sup>	7.65	94 <sup>a</sup>	LaCl <sub>3</sub> ·7H <sub>2</sub> O + LaCl <sub>3</sub> ·6H <sub>2</sub> O
51.5	7.24	49	LaCl <sub>3</sub> ·7H <sub>2</sub> O
49.0	6.66	25	as above
48.1	6.38	10	as above
47.3	6.19	0	as above
47.1	6.14	–9	as above
47.0 <sup>a</sup>	6.12	–23 <sup>a</sup>	LaCl <sub>3</sub> ·9H <sub>2</sub> O + LaCl <sub>3</sub> ·7H <sub>2</sub> O
45.2 <sup>a</sup>	5.71	–34 <sup>a</sup>	LaCl <sub>3</sub> ·10H <sub>2</sub> O + LaCl <sub>3</sub> ·9H <sub>2</sub> O
44.0	5.46	–44	LaCl <sub>3</sub> ·10H <sub>2</sub> O
41.5 <sup>a</sup>	4.95	–64 <sup>a</sup>	H <sub>2</sub> O(s) + LaCl <sub>3</sub> ·10H <sub>2</sub> O
37.8	4.27	–49	H <sub>2</sub> O(s)
35.5	3.89	–41	as above
32.3	3.39	–32	as above
24.3	2.30	–15	as above
15.3	1.31	–6	as above
9.3	0.75	–2	as above
0 <sup>a</sup>	0	0 <sup>a</sup>	as above

<sup>a</sup> numerical result

### Auxiliary Information

#### Method/Apparatus/Procedure:

Mixtures of the components were investigated by means of differential thermal analysis. The samples were prepared in sealed glass ampoules. They were heated and cooled between –120 and ~130 °C with rate of 0.2-0.5 K/min<sup>-1</sup>. The heating curves were recorded with the use of a thermocouple. Stoichiometry of the hydrates was determined from extent of the thermal effects by the method of Tammann for 10-18 samples of compositions changed by

0.3-0.5 mass %. Samples of stoichiometric hydrates (annealed 1-2 d within their temperature existence ranges) were additionally investigated many times by thermal analysis.

**Source and Purity of Materials:**

As in [N.P. Sokolova, K.A. Khaldoyanidi and I.I. Yakovlev, Zh. Neorg. Khim. **25**, 2584 (1980).],  $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$  was prepared by dissolving  $\text{La}_2\text{O}_3$  (99.9 % pure) in excess of HCl solution. The product was twice recrystallized from HCl (“very pure”) and from water.

**Estimated Error:**

Solubility: precision better than  $\pm 0.3$  mass %; reading-out procedure  $\pm 0.5$  mass %.

Composition of the hydrates: precision within 2-3 %.

Temperature: precision  $\pm 1$  K; reading-out procedure  $\pm 1$  K.

---

**Source and Purity of Materials:**

Nothing specified, presumably as in Ref. 54. Heptahydrate of La and hexahydrate of Yb chlorides were prepared by dissolving the corresponding oxides (99.9+ % pure) in HCl solution (of special purity). The salts were twice recrystallized from HCl solution and then from water. They were dried in air at temperatures below 35 °C. Compositions of the salts were confirmed by chemical analysis: metals – by titration with EDTA and chloride – by the Volhard method.

**Estimated Error:**

Solubility: precision  $\pm(1.0-1.5)$  %.<sup>54</sup>

Temperature: nothing specified.

---



LaCl<sub>3</sub>·2C<sub>5</sub>H<sub>5</sub>N·HCl·3H<sub>2</sub>O (since the authors reported about dichloride, the correct formula of the adduct should be written as LaCl<sub>3</sub>·2C<sub>5</sub>H<sub>5</sub>N·2HCl·3H<sub>2</sub>O); C = LaCl<sub>3</sub>·7H<sub>2</sub>O

The compound B was found to be congruently soluble. Its solubility was determined to be 64.0 and 64.8 mass % at 20 and 40 °C, respectively; the corresponding values expressed in molalities, as calculated by the compilers, are 3.08 and 3.19 mol kg<sup>-1</sup>.

Composition of saturated solutions in the ternary LaCl<sub>3</sub>-C<sub>9</sub>H<sub>7</sub>N·HCl-H<sub>2</sub>O system at 20 and 40 °C

<i>t</i> / °C	100 <i>w</i> <sub>1</sub>	<i>m</i> <sub>1</sub> <sup>a</sup>	100 <i>w</i> <sub>3</sub>	<i>m</i> <sub>3</sub> <sup>a</sup>	Equilibrium solid phase <sup>b</sup>
20	0	0	83.0	29.5	D
	1.5	0.39	83.0	32.3	D
	2.5	0.70	83.0	34.6	D
	3.0	0.87	83.0	35.8	D + E
	8.7	1.59	69.0	18.7	E
	10.5	1.78	65.5	16.5	E
	18.3	2.38	50.4	9.72	E
	27.0	2.94	35.5	5.72	E
	34.0	3.30	24.0	3.45	E
	38.3	3.54	17.6	2.41	E + C
	39.5	3.62	16.0	2.17	C
	41.7	3.59	11.0	1.40	C
	44.0	3.66	7.0	0.86	C
	44.5	3.67	6.0	0.73	C
	45.0	3.54	3.2	0.37	C
48.5	3.84	0	0	C	
40	0	0	85.0	34.2	D
	1.5	0.44	84.6	36.8	D
	2.5	0.76	84.0	37.6	D
	4.0	1.25	83.0	38.6	D + E
	6.5	1.71	78.0	30.4	E
	14.0	2.38	62.0	15.6	E
	24.5	3.17	44.0	8.43	E
	32.5	3.58	30.5	4.98	E
	35.5	3.62	24.5	3.70	E
	38.3	3.84	21.0	3.12	E
	41.0	3.89	16.0	2.25	E + C
	41.7	3.88	14.5	2.00	C

44.0	3.90	10.0	1.31	C
45.7	3.88	6.3	0.79	C
48.3	4.04	3.0	0.37	C
50.5	4.16	0	0	C

<sup>a</sup> molalities calculated by the compilers

<sup>b</sup> D = C<sub>9</sub>H<sub>7</sub>N·HCl; E = LaCl<sub>3</sub>·C<sub>9</sub>H<sub>7</sub>N·HCl·7H<sub>2</sub>O; C = LaCl<sub>3</sub>·7H<sub>2</sub>O

The compound E was found to be congruently soluble. Its solubility was determined to be 62.4 and 63.4 mass % at 20 and 40 °C; the corresponding values expressed in molalities, as calculated by the compilers, are 4.04 and 4.22 mol kg<sup>-1</sup>, respectively.

### Auxiliary Information

#### Method/Apparatus/Procedure:

The method of isothermal sections of the phase diagram with refractometric analysis was used.<sup>94</sup> Mixtures of selected compositions along directed section of the phase diagram were shaken for a long time until refractive indexes of the solutions remained constant. Compositions of the saturated solutions and the corresponding solid phases were determined from inflection points on plots of the refractive index versus composition. The refractive indexes were measured in a thermostated refractometer. Both adduct compounds were studied by x-ray diffraction, thermogravimetry and thermal analysis.

#### Source and Purity of Materials:

LaCl<sub>3</sub>·7H<sub>2</sub>O was obtained by repeated recrystallization of anhydrous LaCl<sub>3</sub> ("pure" grade). Water content of 33.92 mass % in the product confirmed the formula of heptahydrate.<sup>94</sup> C<sub>5</sub>H<sub>5</sub>N·HCl was prepared by neutralization of C<sub>5</sub>H<sub>5</sub>N with HCl. The melting point of the product after double crystallization was 82.5 °C (the reported value of 145-147 °C in the Aldrich Catalogue of Chemicals, 2005 is not in agreement) and its density measured in a pycnometer was 1.39 g cm<sup>-3</sup>.

C<sub>9</sub>H<sub>7</sub>N·HCl was prepared by neutralization of C<sub>9</sub>H<sub>7</sub>N with HCl. The melting point of the product after double crystallization was 95 °C and its density measured in a pycnometer was 1.28 g cm<sup>-3</sup>.

#### Estimated Error:

Nothing specified.

<b>Components:</b>	<b>Original Measurements:</b>
(1) Lanthanum chloride; LaCl <sub>3</sub> ; [10099-58-8]	<sup>124</sup> Y.Zh. Zou, Z.X. Tang and Y.Sh. Chen,
(2) Antipyrine; 2,3-dimethyl-1-phenyl-3-pyrazolin-5-one; C <sub>11</sub> H <sub>12</sub> N <sub>2</sub> O; [60-80-0]	Huaxue Xuebao (Acta Chim. Sinica) <b>42</b> , 913 (1984).
(3) Water; H <sub>2</sub> O; [7732-18-5]	
<b>Variables:</b>	<b>Prepared by:</b>
Composition of mixtures	T. Mioduski & C. Gumiński
Temperature: 273 and 293 K	

### Experimental Values

Although the whole range of composition was investigated at 0 and 20 °C, the results were presented only in a barely readable figure. Compositions of two eutonic points were reported numerically at both temperatures.

Compositions of saturated solutions at the eutonic points in the ternary LaCl<sub>3</sub>-C<sub>11</sub>H<sub>12</sub>N<sub>2</sub>O-H<sub>2</sub>O system at 0 and 20 °C

<i>t</i> / °C	100 <i>w</i> <sub>1</sub>	<i>m</i> <sub>1</sub> <sup>a</sup>	100 <i>w</i> <sub>2</sub>	<i>m</i> <sub>2</sub> <sup>a</sup>	Equilibrium solid phases <sup>b</sup>
0	34.5	4.69	35.5	6.28	A + B
	21.36	4.24	58.09	15.02	B + C
20	31.04	5.35	45.31	10.18	A + B
	19.32	4.42	62.87	18.75	B + C

<sup>a</sup> molalities calculated by the compilers

<sup>b</sup> A = LaCl<sub>3</sub>·7H<sub>2</sub>O; B = LaCl<sub>3</sub>·6C<sub>11</sub>H<sub>12</sub>N<sub>2</sub>O·7H<sub>2</sub>O; C = C<sub>11</sub>H<sub>12</sub>N<sub>2</sub>O (antipyrine)

The compound B was found to be incongruently soluble.

### Auxiliary Information

#### Method/Apparatus/Procedure:

Equilibrium between the solutes and the solution was ascertained by constancy of the refractive index of the solutions measured in a thermostated refractometer. Both saturated solutions and solid phases were analyzed. La was determined by titration with EDTA solution using xylenol orange as an indicator. N in antipyrine was determined by the Kjeldahl method. Composition of solid phases was found by the graphical method of Schreinemakers. Composition of the eutonic points was confirmed by break points on the isotherms of refractive index versus composition.

#### Source and Purity of Materials:

LaCl<sub>3</sub>·7H<sub>2</sub>O was prepared by dissolving La<sub>2</sub>O<sub>3</sub> (99.99 % pure) in HCl solution and subsequent crystallization of the product.

Antipyrine was purified prior to use.

**Estimated Error:**

Solubility: precision  $\pm 0.1$  mass %.

Temperature: precision  $\pm 0.2$  K; stability  $\pm 0.05$  K.

Refractive index:  $\pm 0.0002$ .

---

<b>Components:</b>	<b>Original Measurements:</b>
(1) Lanthanum chloride; $\text{LaCl}_3$ ; [10099-58-8]	<sup>133</sup> X.L. Xu, Z.X. Tang, Zh.Zh. Guo and Y.Sh. Chen, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) <b>12</b> , 728 (1991).
(2) 4-chloroacetylantipyrine; 4-(chloroacetyl)-1,2-dihydro-1,5-dimethyl-2-phenyl-3H-pyrazol-3-one; $\text{C}_{13}\text{H}_{13}\text{ClN}_2\text{O}_2$ ; [6630-73-5]	
(3) Water; $\text{H}_2\text{O}$ ; [7732-18-5]	
<b>Variables:</b>	<b>Prepared by:</b>
Composition of mixtures	C. Gumiński & D. Zeng
One temperature: 303 K	

### Experimental Values

Composition of saturated solutions in the ternary  $\text{LaCl}_3\text{-C}_{13}\text{ClH}_{13}\text{N}_2\text{O}_2\text{-H}_2\text{O}$  system at 30 °C

$100w_1$	$m_1^a$	$100w_2$	$m_2^a$	Equilibrium solid phase <sup>b</sup>
0	0	0.11	0.0042	A
13.50	0.637	0.10	0.0044	A
27.48	1.547	0.10	0.0052	A
33.14	2.025	0.14	0.0079	A
40.90	2.833	0.24	0.015	A
45.90	3.483	0.37	0.026	A
48.39	3.844	0.28	0.021	A + B
49.50	3.997	0	0	B

<sup>a</sup> molalities calculated by the compilers

<sup>b</sup> A =  $\text{C}_{13}\text{ClH}_{13}\text{N}_2\text{O}_2$ ; B =  $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$

The system was found to be of eutonic type.

### Auxiliary Information

#### Method/Apparatus/Procedure:

The components were equilibrated in isothermal conditions. Equilibrium in the system was checked by periodic measurement of refractive index of the liquid phase. It was found that equilibrium was reached after 20 d. Content of La in the saturated solution was determined by titration with EDTA solution. Content of  $\text{C}_{13}\text{ClH}_{13}\text{N}_2\text{O}_2$  was determined as a sum from gravimetry and ultraviolet spectrometry: the wet residue was dissolved in boiling water, then cooled for the crystallization, dried and weighed, subsequently the rest of the compound in the solution was determined by ultraviolet spectrometry. Composition of the solid phases was determined by the method of Schreinemakers.

**Source and Purity of Materials:**

$\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$  was prepared from  $\text{La}_2\text{O}_3$  and the stoichiometric amount of HCl solution. The mixture was stirred, evaporated on a water bath, cooled and crystallized adding a small crystal of the product. The solid and the solution were separated. The crystals were recrystallized and stored in a dry container.

$\text{C}_{13}\text{ClH}_{13}\text{N}_2\text{O}_2$  was prepared in this laboratory and twice recrystallized.

Distilled water was used.

**Estimated Error:**

Solubility: nothing specified.

Temperature: stability  $\pm 0.1$  K; stability in the refractive index measurements  $\pm 0.2$  K.

Refractive index: precision  $\pm 0.0002$ .

---

<b>Components:</b>	<b>Original Measurements:</b>
(1) Lanthanum chloride; LaCl <sub>3</sub> ; [10099-58-8]	<sup>140</sup> H.B. Liang, Sh.J. Li, G.L. Gao, N.H. Cao
(2) Phthalimidoantipyrine; 2-(2,3-dihydro-1,5-dimethyl-3-oxo-2-phenyl)-1H-pyrazol-4-yl-1H-isoindole-1,3(2H)-dione; C <sub>19</sub> H <sub>15</sub> N <sub>3</sub> O <sub>3</sub> ; [101896-02-0]	and Z.X. Tang, Shanxi Shifan Daxue Xuebao, Ziran Kexueban (J Shaanxi Norm. Univ., Nat. Sci. Edit.) <b>25</b> , no 3, 61 (1997).
(3) Water; H <sub>2</sub> O; [7732-18-5]	

<b>Variables:</b>	<b>Prepared by:</b>
Composition of mixtures	C. Gumiński & D. Zeng
One temperature: 303 K	

### Experimental Values

Composition of saturated solutions in the ternary LaCl<sub>3</sub>-C<sub>19</sub>H<sub>15</sub>N<sub>3</sub>O<sub>3</sub>-H<sub>2</sub>O system at 30 °C

100w <sub>1</sub>	m <sub>1</sub> <sup>a</sup>	100w <sub>2</sub>	m <sub>2</sub> <sup>a</sup>	Equilibrium solid phase <sup>b</sup>
0	0	0.28	0.0084	A
12.43	0.580	0.19	0.0065	A
27.49	1.549	0.16	0.0066	A
35.90	2.289	0.15	0.0070	A
40.26	2.753	0.12	0.0060	A
43.52	3.149	0.13	0.0069	A
44.97	3.339	0.11	0.0060	A
47.90	3.759	0.14	0.0081	A
49.39	3.989	0.13	0.0077	A + B
49.50	3.997	0	0	B

<sup>a</sup> molalities calculated by the compilers

<sup>b</sup> A = C<sub>19</sub>H<sub>15</sub>N<sub>3</sub>O<sub>3</sub>; B = LaCl<sub>3</sub>·7H<sub>2</sub>O

The system was found to be of a simple eutonic type. The same solubility results were later published in Ref. 150.

### Auxiliary Information

#### Method/Apparatus/Procedure:

Mixtures of the components were equilibrated in a thermostated water bath for 40 d. Reaching equilibrium in the system was monitored by measurements of refractive index of the solutions at regular time intervals. Content of La was determined by titration with EDTA solution. Content of C<sub>19</sub>H<sub>15</sub>N<sub>3</sub>O<sub>3</sub> was determined by ultraviolet spectrometry. Composition of wet solid phases was determined by the method of Schreinemakers and confirmed by a chemical analysis.

**Source and Purity of Materials:**

$\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$  was prepared by dissolution of  $\text{La}_2\text{O}_3$  (99.95 % pure) in a stoichiometric amount of HCl solution of analytical purity.

$\text{C}_{19}\text{H}_{15}\text{N}_3\text{O}_3$  was synthesized in the laboratory of the authors.

Doubly distilled water was used.

**Estimated Error:**

Solubility: nothing specified.

Temperature: precision  $\pm 0.05$  (in the thermostat); precision  $\pm 0.2$  K (in the refractometer).

---



<b>Components:</b>	<b>Original Measurements:</b>
(1) Lanthanum chloride; LaCl <sub>3</sub> ; [10099-58-8]	<sup>145</sup> Sh.P. Liu, G.Q. Huang, Ch.D. Zhang and
(2) Fluorouracil; 5-fluoro-2,4(1H,3H)- pyrimidinedione; C <sub>4</sub> H <sub>3</sub> FN <sub>2</sub> O <sub>2</sub> ; [51-21-8]	H.Y. Wang, Neimenggu Daxue Xuebao, Ziran Kexueban (Acta Sci. Nat. Univ. NeMongol) <b>30</b> , no. 1, 53 (1999).
(3) Water; H <sub>2</sub> O; [7732-18-5]	
<b>Variables:</b>	<b>Prepared by:</b>
Composition of mixtures	C. Gumiński & D. Zeng
One temperature: 303 K	

### Experimental Values

Composition of saturated solutions in the ternary LaCl<sub>3</sub>-C<sub>4</sub>H<sub>3</sub>FN<sub>2</sub>O<sub>2</sub>-H<sub>2</sub>O system at 30 °C

100w <sub>1</sub>	m <sub>1</sub> <sup>a</sup>	100w <sub>2</sub>	m <sub>2</sub> <sup>a</sup>	Equilibrium solid phase <sup>b</sup>
0	0	1.380	0.108	A
2.350	0.099	1.335	0.107	A
9.616	0.439	1.147	0.099	A
12.11	0.569	1.061	0.094	A
21.75	1.148	1.021	0.102	A
28.97	1.687	1.009	0.111	A
34.25	2.161	1.122	0.133	A
35.63	2.335	2.157	0.267	A + B
36.56	2.421	1.856	0.232	B
38.39	2.611	1.652	0.212	B
39.41	2.714	1.383	0.180	B
42.31	3.042	0.983	0.133	B
46.12	3.556	0.997	0.145	B
47.93	3.895	1.901	0.291	B + C
48.51	3.910	0.906	0.138	C
49.63	4.017	0	0	C

<sup>a</sup> molalities calculated by the compilers

<sup>b</sup> A = C<sub>4</sub>H<sub>3</sub>FN<sub>2</sub>O<sub>2</sub>; B = LaCl<sub>3</sub>·4C<sub>4</sub>H<sub>3</sub>FN<sub>2</sub>O<sub>2</sub>·7H<sub>2</sub>O; C = LaCl<sub>3</sub>·7H<sub>2</sub>O

Thermal and thermogravimetric analysis of the compounds B and C revealed gradual dehydration of these compounds to LaCl<sub>3</sub>·4C<sub>4</sub>H<sub>3</sub>FN<sub>2</sub>O<sub>2</sub>·3H<sub>2</sub>O and finally LaCl<sub>3</sub>·4C<sub>4</sub>H<sub>3</sub>FN<sub>2</sub>O<sub>2</sub>, and also to LaCl<sub>3</sub>·3H<sub>2</sub>O, LaCl<sub>3</sub>·H<sub>2</sub>O and finally LaCl<sub>3</sub>, respectively. Temperatures of the corresponding steps were observed at 73.9 and 104.8 °C, and also at 85.9, 119.8 and 156.8 °C, respectively. The x-ray diffractogram for LaCl<sub>3</sub>·7H<sub>2</sub>O powder was different from the one published in the Powder Diffraction File.

## Auxiliary Information

---

### Method/Apparatus/Procedure:

The components in proper amounts were continuously mixed for 8 d. The mother solution and the solid phase were analyzed: La – by titration with EDTA solution, Cl – by potentiometric titration with  $\text{AgNO}_3$  solution and  $\text{C}_4\text{H}_3\text{FN}_2\text{O}_2$  by infrared spectrometry. After combustion of the compounds A or B, an F-ion selective electrode was used for determination of F content. The compounds A, B and C were characterized by thermal analysis, thermogravimetry, x-ray diffraction, Fourier-transform infrared and NMR spectroscopy. Thermal analysis was performed in an Ar atmosphere with scan rate of 10 K/min between 10 and 410 °C, using  $\alpha\text{Al}_2\text{O}_3$  as a reference. The compound B was dried with NaOH before the tests and was subjected to elemental analysis.

### Source and Purity of Materials:

$\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$  was obtained from  $\text{La}_2\text{O}_3$  (99.9 % pure) which was dissolved in a slight excess of HCl solution. The product was recrystallized twice.

$\text{C}_4\text{H}_3\text{FN}_2\text{O}_2$  was a commercial product (99.7 % pure) with melting point of 280 °C.

All other chemicals were chemically or analytically pure.

### Estimated Error:

Nothing specified.

---

<b>Components:</b>	<b>Original Measurements:</b>
(1) Lanthanum chloride; LaCl <sub>3</sub> ; [10099-58-8]	<sup>146</sup> H.B. Liang, W.L. Wang, D.D. Hu and W.P.
(2) 4-(4-chlorobenzoyl)aminoantipyrine; 4-chloro-N-(2,3-dichloro-1,5-dimethyl-3-oxo-2-phenyl-1H-pyrazol-4-yl)-benzamide; C <sub>18</sub> H <sub>16</sub> ClN <sub>3</sub> O <sub>2</sub> ; [56866-88-7]	Zhang, J. Rare Earths <b>17</b> , no 1, 81 (1999); Zhongguo Xitu Xuebao <b>17</b> , no 1, 64 (1999).
(3) Water; H <sub>2</sub> O; [7732-18-5]	

<b>Variables:</b>	<b>Prepared by:</b>
Composition of mixtures	C. Gumiński
One temperature: 303 K	

### Experimental Values

Composition of saturated solutions in the ternary LaCl<sub>3</sub>-C<sub>18</sub>H<sub>16</sub>N<sub>3</sub>O<sub>2</sub>Cl-H<sub>2</sub>O system at 30 °C

100w <sub>1</sub>	m <sub>1</sub> <sup>a</sup>	100w <sub>2</sub>	m <sub>2</sub> <sup>a</sup>	Equilibrium solid phase <sup>b</sup>
0	0	0.03	0.0009	A
8.23	0.376	0.08	0.003	A
17.26	0.852	0.10	0.0035	A
31.00	1.833	0.05	0.002	A
40.06	2.727	0.04	0.002	A
46.28	3.519	0.09	0.005	A
48.94	3.915	0.09	0.005	A + B
49.50	4.000	0	0	B

<sup>a</sup> molalities calculated by the compiler

<sup>b</sup> A = C<sub>18</sub>H<sub>16</sub>N<sub>3</sub>O<sub>2</sub>Cl; B = LaCl<sub>3</sub>·7H<sub>2</sub>O

The system was found to be of simple eutonic type.

### Auxiliary Information

#### **Method/Apparatus/Procedure:**

Mixtures of the components were prepared in appropriate proportions. They were equilibrated in a thermostat. Equilibrium in the system was ascertained by monitoring refractive indexes of the solutions in a thermostated refractometer. The saturated solution and the wet residue were separated when the refractive index of a selected solution was constant in time. Content of La in the liquid phase was found by titration with EDTA solution. Content of C<sub>18</sub>H<sub>16</sub>N<sub>3</sub>O<sub>2</sub>Cl was determined by means of ultraviolet spectrophotometry, as in [K. Cheng, H. Liang and B. Cui, Chem. Reagents (in Chinese) **19**, 239 (1997)].

Compositions of the solid phases were found by the method of Schreinemakers.

#### **Source and Purity of Materials:**

$\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$  was prepared from  $\text{La}_2\text{O}_3$  (99.9 % pure) and HCl solution (analytically pure).

Crystals of the product were recrystallized.

$\text{C}_{18}\text{H}_{16}\text{N}_3\text{O}_2\text{Cl}$  was prepared by the authors, as described in [K. Cheng, H. Liang and B. Cui, Chem. Reagents (in Chinese) **19**, 239 (1997).].

Doubly distilled water was used.

**Estimated Error:**

Solubility: nothing specified.

Temperature: precision  $\pm 0.05$  K (in the thermostat), precision  $\pm 0.2$  K (in the refractometer).

---

**4.4.8.  $\text{LaCl}_3$ -Amino acids- $\text{H}_2\text{O}$  Systems**

Etc.

## 5. Cumulative References

- <sup>1</sup>H. Stephen and T. Stephen, *Solubilities of Inorganic and Organic Compounds* (Pergamon, Oxford, 1979).
- <sup>2</sup>J. Burgess and J. Kijowski, *Adv. Inorg. Chem. Radiochem.* **24**, 57 (1981).
- <sup>3</sup>Gmelins Handbook, Sc, Y, La and Ln Chlorides, Springer-Verlag, Berlin, **39**, Part C4a, 1982.
- <sup>4</sup>S. Siekierski, T. Mioduski and M. Salomon, *IUPAC Solubility Data Series*, Vol. 13, Sc, Y, La and Ln Nitrates (Pergamon, Oxford, 1983).
- <sup>5</sup>T. Mioduski, *Chem. Anal. (Warsaw)* **43**, 457 (1998).
- <sup>6</sup>T. Mioduski, *J. Radioanal. Nucl. Chem. Lett.* **128**, 351 (1988).
- <sup>7</sup>H. Miyamoto, *Current Topics Sol. Chem.* **1**, 81 (1994).
- <sup>8</sup>T. Mioduski, unpublished materials.
- <sup>9</sup>T. Mioduski and M. Salomon, *IUPAC Solubility Data Series*, Vol. 22, Sc, Y, La, and Ln Halides in Nonaqueous Solvents (Pergamon, Oxford, 1985).
- <sup>10</sup>N. P. Sokolova, *Radiokhimiya* **30**, 435 (1988).
- <sup>11</sup>G. Reuter, H. Fink and H. J. Seifert, *Z. Anorg. Chem.* **620**, 665 (1994).
- <sup>12</sup>C. Gumiński, *Arch. Metall. Mater.* **51**, 617 (2006).
- <sup>13</sup>F. Teixeira da Silva, *Trans. Inst. Min. Metall., Ser. C* **114**, C70 (2005).
- <sup>14</sup>W. Rafalski and K. Jonak, *Przemysl Chem.* **48**, 735 (1969).
- <sup>15</sup>J. W. Lorimer and R. Cohen-Adad, *Thermodynamics of Solubility*, in *The Experimental Determination of Solubilities*, G. T. Hefter and R. P. T. Tomkins, Eds. (Wiley, Chichester, 2003), p. 19.
- <sup>16</sup>J. Harkot, *Polish J. Chem.* **63**, 337 (1989).
- <sup>17</sup>J. Harkot, *Polish J. Chem.* **64**, 53 (1990).
- <sup>18</sup>F. H. Spedding, J. A. Rard and V. Saeger, *J. Chem. Eng. Data* **19**, 373 (1974).
- <sup>19</sup>L. G. Sillen and A. Martell, *Stability Constants*, Special Publications no 17 and 25 (The Chemical Society, London, 1964 and 1971).
- <sup>20</sup>C. S. Oakes, J. A. Rard and D. G. Archer, *J. Chem. Eng. Data* **49**, 313 (2004).
- <sup>21</sup>W. Fischer, J. Wernet and M. Zumbusch-Pfisterer, *Z. Anorg. Chem.* **258**, 157 (1949).
- <sup>22</sup>W. Fischer and G. Buhler, *Z. Anorg. Chem.* **285**, 156 (1956).
- <sup>23</sup>W. Fischer, H. Bauer, I. Dillo, T. Molaug, J. Nier, H. Rohrer and K. Trovaag, *Z. Anorg. Chem.* **357**, 177 (1968).
- <sup>24</sup>A. P. Samodelov, *Zh. Neorg. Khim.* **10**, 1735 (1965).

- <sup>25</sup>Y. Suzuki, R. Yoshino, H. Saitoh, K. Fukushi and Y. Sonehara, *J. Alloys Comp.* **180**, 383 (1992).
- <sup>26</sup>K. C. Lim, B. W. Skelton and A. H. White, *Aust. J. Chem.* **53**, 875 (2000).
- <sup>27</sup>W. W. Wendlandt, *J. Inorg. Nucl. Chem.* **5**, 118 (1957).
- <sup>28</sup>F. Petru and F. Kutek, *Collect. Czech. Chem. Commun.* **25**, 1143 (1960).
- <sup>29</sup>F. Petru, J. Hradilova and B. Hajek, *Collect. Czech. Chem. Commun.* **33**, 2720 (1968).
- <sup>30</sup>W. Crookes, *Phil. Trans. Roy. Soc.* **A 209**, 15 (1908).
- <sup>31</sup>I. V. Arkhangel'skii, L. N. Komissarova, V. M. Shatskii and N. P. Shepelev, *Zh. Neorg. Khim.* **17**, 310 (1972).
- <sup>32</sup>G. A. Melson, D. J. Olszanski and A. K. Rahimi, *Spectrochim. Acta* **A 33**, 301 (1977).
- <sup>33</sup>S. V. Aleksandrovskii, *Zh. Prikl. Khim.* **70**, 1761 (1997).
- <sup>34</sup>K. H. Schröder, Dissertation, Münster, Germany, 1955; as quoted in Ref. 3.
- <sup>35</sup>*Aldrich 2007-2008 Handbook of Fine Chemicals* (Sigma-Aldrich Corp., St. Louis, 2007), p. 2170.
- <sup>36</sup>M. C. Crew, H. E. Steinert and B. S. Hopkins, *J. Phys. Chem.* **29**, 34 (1925).
- <sup>37</sup>M. D. Williams, H. C. Fogg and C. James, *J. Am. Chem. Soc.* **47**, 297 (1925).
- <sup>38</sup>J. E. Powell, *U.S. Atom. Ener. Comm. Rep. IS-15*, 10 (1959).
- <sup>39</sup>Z. N. Shevtsova and In Vei-Tszyuan, *Zh. Neorg. Khim.* **8**, 1749 (1963).
- <sup>40</sup>A. V. Nikolaev, A. A. Sorokina and V. G. Tsubanov, *Dokl. Akad. Nauk SSSR* **172**, 1333 (1967).
- <sup>41</sup>A. V. Nikolaev, A. A. Sorokina, A. Ya. Vilenskaya and V. G. Tsubanov, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 6, 5 (1967).
- <sup>42</sup>Z. N. Shevtsova, L. S. Nam and B. G. Korshunov, *Zh. Neorg. Khim.* **13**, 1682 (1968).
- <sup>43</sup>V. S. Petelina, N. I. Nikurashina and G. V. Illarionova, *Issledovaniya v Oblasti Khimii Redkozemelnykh Elementov, Saratovskii Universitet, Saratov* **3**, 48 (1971).
- <sup>44</sup>A. V. Nikolaev and A. A. Sorokina, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 4, 61 (1971).
- <sup>45</sup>A. N. Zelikman, N. V. Baryshnikov and A.I. Khokhlov, *Zh. Neorg. Khim.* **16**, 2023 (1971).
- <sup>46</sup>A. V. Nikolaev, A. A. Sorokina and N. G. Yudina, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 5, 28 (1972).

- <sup>47</sup>G. A. Ashimkulova, K. Sulaimankulov, Sh. T. Turdaliev and K. Noguev, Zh. Neorg. Khim. **18**, 2011 (1973).
- <sup>48</sup>G. A. Ashimkulova, *Geterogennye Ravnovesiya v Sistemakh Neorganicheskikh i Organicheskikh Soedinenii* (Ilim, Frunze, 1974), p. 21.
- <sup>49</sup>G. A. Ashimkulova, K. Noguev and K. Sulaimankulov, Zh. Neorg. Khim. **19**, 2588 (1974).
- <sup>50</sup>A. V. Nikolaev, L. G. Kosobudskaya and A. A. Sorokina, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 5, 53 (1975).
- <sup>51</sup>F. H. Spedding, V. W. Saeger, K. A. Gray, P. K. Boneau, M. A. Brown, C. W. DeKock, J. L. Baker, L. E. Shiers, H. O. Weber and A. Habenschuss, J. Chem. Eng. Data **20**, 72 (1975).
- <sup>52</sup>F. H. Spedding, H. O. Weber, V. W. Saeger, H. H. Petheram, J.A. Rard and A. Habenschuss, J. Chem. Eng. Data **21**, 341 (1976).
- <sup>53</sup>N. Levina, G. Dadabaeva, D. D. Ikrami and A. S. Paramzin, Dep. VINITI N-478-77, 27 pp. (1977).
- <sup>54</sup>A. V. Nikolaev, A. A. Sorokina, V. N. Lubkova and N. G. Yudina, *Osnovy Vzaimodeistviya Solei Redkozemelnykh Elementov v Vode* (Nauka, Novosibirsk, 1977), p. 36, 45, 46, 53, 72, 79, 161.
- <sup>55</sup>A. V. Nikolaev, A. A. Sorokina, N. P. Sokolova, G. S. Kotlyar-Shapiro, N. P. Anoshina, L. I. Bagryantseva and A. P. Olovyanishnikova, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 4, 84 (1977).
- <sup>56</sup>A. V. Nikolaev, A. A. Sorokina, P. A. Stabnikov, N. P. Sokolova and G. S. Kotlyar-Shapiro, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 6, 44 (1977).
- <sup>57</sup>N. P. Sokolova, I. I. Yakovlev and I. N. Komissarova, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 6, 45 (1980).
- <sup>58</sup>A. A. Sorokina and N. G. Yudina, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 5, 51 (1981).
- <sup>59</sup>J. A. Rard and F. H. Spedding, J. Chem. Eng. Data **27**, 454 (1982).
- <sup>60</sup>N. P. Sokolova, Zh. Neorg. Khim. **28**, 782 (1983).
- <sup>61</sup>M. A. Urusova and V. M. Valyashko, Zh. Neorg. Khim. **38**, 1074 (1993).
- <sup>62</sup>F. Ren, Sh. L. Gao, G. H. Chu and I. Zh. Shiq, Yingyong Huaxue (Chin. J. Appl. Chem.) **17**, 203 (2000).
- <sup>63</sup>Zh. P. Qiao, L. H. Zhuo and H. Wang, Wuji Huaxue Xuebao (Chin. J. Inorg. Chem.) **20**, 929 (2004).
- <sup>64</sup>H. Wang, J. X. Duan, X. Q. Ran and Sh. Y. Gao, Chin. J. Chem. **22**, 1128 (2004).

- <sup>65</sup>L. Ya. Markovskii, E. Ya. Pesina, Yu. A. Omelchenko and Yu. D. Kondrashev, Zh. Neorg. Khim. **14**, 14 (1969).
- <sup>66</sup>N. V. Baryshnikov, A. V. Polonskii and A. I. Khokhlov, Zh. Neorg. Khim. **20**, 3177 (1975); A. V. Polonskii, N. V. Baryshnikov and L. A. Voskresenskaya, Zh. Neorg. Khim. **21**, 317 (1976).
- <sup>67</sup>J. Wang, Z. Guo, Y. Chen, S. Huang and Z. Wang, J. Northwest Univ. **20**, 49 (1990); as quoted in Ref. 68.
- <sup>68</sup>V. V. Hong and J. Sundström, Thermochim. Acta **307**, 37 (1997).
- <sup>69</sup>G. Haeseler and F. Matthes, J. Less-Common Met. **9**, 133 (1965).
- <sup>70</sup>C. Matignon, Ann. Chim. Phys. **8**, 433 (1906).
- <sup>71</sup>M. Z. Su and P. G. Li, Huaxue Tongbao, no 4, 34 (1979).
- <sup>72</sup>H. J. Seifert and S. Funke, Thermochim. Acta **320**, 1 (1998).
- <sup>73</sup>W. W. Wendlandt, J. Inorg. Nucl. Chem. **9**, 136 (1959).
- <sup>74</sup>L. G. Sokolova, A. V. Lapitskaya, A. F. Bolshakov, S. B. Pirkes and B. V. Abalduev, Zh. Neorg. Khim. **26**, 1736 (1981); B. V. Abalduev, A. F. Bolshakov, L. G. Sokolova, A. V. Lapitskaya and S. B. Pirkes, *Issledovaniya v Oblasti Khimii Redkozemelnykh Elementov* (Saratovskii Universitet, Saratov, 1975), p. 47.
- <sup>75</sup>W. W. Wendlandt and R. G. Sewell, Texas J. Sci. **8**, 231 (1961).
- <sup>76</sup>Yu. G. Sakharova and T. A. Ezhova, Zh. Neorg. Khim. **21** 551 (1976); Russ. J. Inorg. Chem. **21**, 296 (1976).
- <sup>77</sup>W. W. Wendlandt and J. L. Bear, Anal. Chim. Acta **21**, 439 (1959).
- <sup>78</sup>A. V. Nikolaev, A. A. Sorokina and N. G. Yudina, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 6, 116 (1983).
- <sup>79</sup>W. Prandtl and J. Rauchenberger, Ber. Dtsch. Chem. Ges. **53**, 843 (1920).
- <sup>80</sup>J. N. Friend and R. W. Hale, J. Chem. Soc. **1940**, 670.
- <sup>81</sup>Z. N. Shevtsova, V. S. Zelova and L. I. Ushakova, Nauch. Dokl. Vyssh. Shkoly, Khim. Khim. Tekhnol. **3**, 417 (1958).
- <sup>82</sup>J. E. Powell and H. R. Burkholder, J. Inorg. Nucl. Chem. **14**, 65 (1960).
- <sup>83</sup>Z. N. Shevtsova, L. I. Zhizhina and L. E. Eltsberg, Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol. **4**, 176 (1961).
- <sup>84</sup>E. N. Khutorskoi and A. D. Sheveleva, Ucheb. Zap. Perm. Univ. **178**, 49 (1968).
- <sup>85</sup>E. N. Khutorskoi and A. D. Sheveleva, Ucheb. Zap. Perm. Univ. **178**, 57 (1968).
- <sup>86</sup>G. Brunisholz and M. Nozari, Helv. Chim. Acta **52**, 2303 (1969).



- <sup>87</sup>V. S. Petelina, R. V. Mertslin, N. I. Nikurashina and L. K. Sedova, *Issledovaniya v Oblasti Khimii Redkozemelnykh Elementov* (Saratovskii Universitet, Saratov, 1969), p. 85.
- <sup>88</sup>Z. F. Alieva, O. V. Agashkin and K. Sulaimankulov, *Izv. Akad. Nauk Kirgiz. SSR*, no 5, 38 (1970).
- <sup>89</sup>E. N. Khutorskoi and A. D. Sheveleva, *Ucheb. Zap. Perm. Univ.* **207**, 11 (1970).
- <sup>90</sup>A. A. Volkov, M. P. Borodina and A. I. Shapiro, *Ucheb. Zap. Perm. Univ.* **229**, 3 (1970).
- <sup>91</sup>A. D. Sheveleva, N. A. Torgashina and E. N. Khutorskoi, *Ucheb. Zap. Perm. Univ.* **229**, 21 (1970).
- <sup>92</sup>Z. F. Alieva and K. S. Sulaimankulov, *Izv. Akad. Nauk Kaz. SSR, Ser. Khim.* **20**, no 6, 16 (1970).
- <sup>93</sup>A. V. Nikolaev, A. A. Sorokina, N. G. Yudina and V. N. Lubkova, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 5, 35 (1972).
- <sup>94</sup>E. F. Zhuravlev, R. V. Yurkevich and N. G. Kalegina, *Zh. Neorg. Khim.* **17**, 2536 (1972).
- <sup>95</sup>R. V. Yurkevich and E. F. Zhuravlev, *Zh. Neorg. Khim.* **17**, 3063 (1972).
- <sup>96</sup>A. D. Sheveleva, K. I. Mochalov, E. N. Khutorskoi and N. A. Torgashina, *Ucheb. Zap. Perm. Univ.* **289**, 3 (1973).
- <sup>97</sup>A. D. Sheveleva, K. I. Mochalov and N. A. Torgashina, *Ucheb. Zap. Perm. Univ.* **289**, 15 (1973).
- <sup>98</sup>E. F. Zhuravlev, R. V. Yurkevich and N. G. Kalegina, *Zh. Neorg. Khim.* **18**, 1109, (1973).
- <sup>99</sup>E. F. Zhuravlev, R. V. Yurkevich, A. G. Samatov and L. F. Shlentova, *Zh. Neorg. Khim.* **18**, 2247 (1973).
- <sup>100</sup>E. F. Zhuravlev, R. V. Yurkevich and N. A. Rasskazova, *Zh. Neorg. Khim.* **19**, 534 (1974).
- <sup>101</sup>E. F. Zhuravlev, R. V. Yurkevich and N. A. Rasskazova, *Zh. Neorg. Khim.* **19**, 1977 (1974).
- <sup>102</sup>E. F. Zhuravlev, R. V. Yurkevich and N. G. Kalegina, *Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol.* **17**, 498 (1974).
- <sup>103</sup>E. F. Zhuravlev, R. V. Yurkevich and N. A. Rasskazova, *Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol.* **17**, 1123 (1974).
- <sup>104</sup>A. V. Nikolaev, A. A. Sorokina and N. L. Kozyreva, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 6, 86 (1974).

- <sup>105</sup>R. V. Yurkevich and E. F. Zhuravlev, *Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol.* **18**, 140 (1975).
- <sup>106</sup>Ts. B. Sergeeva and M. Kh. Karapetyants, *Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol.* **19**, 846 (1976).
- <sup>107</sup>R. V. Yurkevich, E. F. Zhuravlev and N. A. Rasskazova, *Zh. Neorg. Khim.* **21**, 2234 (1976).
- <sup>108</sup>Z. Zholalieva, K. Sulaimankulov and K. Nogoiev, *Zh. Neorg. Khim.* **21**, 2290 (1976).
- <sup>109</sup>E. F. Zhuravlev, R. V. Yurkevich and N. A. Rasskazova, *Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol.* **19**, 1297 (1976).
- <sup>110</sup>A. A. Sorokina, N. P. Sokolova, G. S. Kotlyar-Shapiro and P. A. Stabnikov, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 4, 91 (1977).
- <sup>111</sup>F. H. Spedding, C. W. DeKock, G. W. Pepple and A. Habenschuss, *J. Chem. Eng. Data* **22**, 58 (1977).
- <sup>112</sup>A. V. Nikolaev, A. A. Sorokina, N. P. Sokolova, G. S. Kotlyar-Shapiro and L.I. Bagryantseva, *Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, no 1, 46 (1978).
- <sup>113</sup>Z. N. Zholalieva and K. Sulaimankulov, *Zh. Neorg. Khim.* **23**, 1367 (1978).
- <sup>114</sup>L. L. Kost, Yu. V. Shirai and V. G. Shevchuk, *Zh. Neorg. Khim.* **25**, 579 (1980).
- <sup>115</sup>V. G. Shevchuk and Yu. V. Shirai, *Zh. Neorg. Khim.* **25**, 843 (1980).
- <sup>116</sup>Yu. V. Shirai and V. G. Shevchuk, *Zh. Neorg. Khim.* **26**, 1940 (1981).
- <sup>117</sup>G. O. Karapetyan, B. I. Spesivtsev, N. O. Khvostova and O. V. Yanush, *SPSTL Dep. Doc. 916 Khp-D81*, p. 188 (1981); *Chem. Abstr.* **98**, 78951d (1982).
- <sup>118</sup>Z. X. Tang, Zh. Zh. Guo, P. H. Chen, J. H. Ma and Y. Sh. Chen, *Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.)* **3**, 447 (1982).
- <sup>119</sup>Z. X. Tang and Y. Sh. Chen, *Zhongguo Xitu Xuebao (J. Chin. Rare Earth Soc.)* **1**, 43 (1983).
- <sup>120</sup>Z. X. Tang, Zh. Zh. Guo, P. H. Chen, J. H. Ma and Y. Sh. Chen, *Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.)* **4**, 419 (1983).
- <sup>121</sup>Z. X. Tang, Zh. Zh. Guo, P. H. Chen, J.H. Ma and Y.Sh. Chen, *Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.)* **4**, 631 (1983).
- <sup>122</sup>Z. X. Tang, Zh. Zh. Guo, P.H. Chen, J. H. Ma and Y. Sh. Chen, *Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.)* **4**, 634 (1983).
- <sup>123</sup>K. J. Zwietasch, E. M. Kirmse and I. Krech, *Z. Chem.* **24**, 144 (1984).
- <sup>124</sup>Y. Zh. Zou, Z. X. Tang and Y. Sh. Chen, *Huaxue Xuebao (Acta Chim. Sinica)* **42**, 913 (1984).

- <sup>125</sup>D. A. Storozhenko, Yu. V. Shirai and N. M. Lazorenko, Zh. Neorg. Khim. **31**, 537 (1986).
- <sup>126</sup>Yu. V. Shirai, Zh. Neorg. Khim. **31**, 1049 (1986).
- <sup>127</sup>D. A. Storozhenko, Yu. V. Shirai and L. M. Lazorenko, Zh. Neorg. Khim. **31**, 1610 (1986).
- <sup>128</sup>H. Kanno and Y. Akama, J. Phys. Chem. **91**, 1263 (1987).
- <sup>129</sup>D. A. Storozhenko, Yu. V. Shirai and N. F. Eskova, Zh. Neorg. Khim. **33**, 2438 (1988).
- <sup>130</sup>Q. H. Shi, J. Q. Chen and L. L. Wang, Huaxue Shijie **29**, 484 (1988).
- <sup>131</sup>J. X. Chen, X. Q. Ran, Zh. Zh. Guo and Y. Sh. Chen, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) **11**, 555 (1990).
- <sup>132</sup>E. Berecz and T. I. Török, Z. Anorg. Chem. **583**, 177 (1990).
- <sup>133</sup>X. L. Xu, Z. X. Tang, Zh. Zh. Guo and Y. Sh. Chen, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) **12**, 728 (1991).
- <sup>134</sup>D. H. Ren, J. Zhang, J. Ch. Sun, C. M. Yin and Z. R. Liu, Wuji Huaxue Xuebao (Chin. J. Inorg. Chem.) **8**, 209 (1992).
- <sup>135</sup>K. Fukushi, A. Mutoh, K. Ishikawa and Y. Suzuki, J. Alloys Comp. **192**, 290 (1993).
- <sup>136</sup>K. Fukushi, K. Iwamoto, K. Kobayashi, K. Ishikawa and Y. Suzuki, J. Alloys Comp. **207/208**, 468 (1994).
- <sup>137</sup>H. Voigt, *Thermodynamische, thermoanalytische und strukturelle Untersuchungen an Lanthanhalogenid-Hydraten*, Rep. Vo5112/1-2 (Bergakademie, Freiberg, Germany, 1994).
- <sup>138</sup>Q. D. Li, H. F. Xue and D. H. Ren, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) **8**, 1172 (1995).
- <sup>139</sup>Y. H. Li, X. Q. Ran and P. H. Chen, Yan Hu Yan Jiu (J. Salt Lake Sci.) **3**, no 2, 40 (1995).
- <sup>140</sup>H. B. Liang, Sh. J. Li, G. L. Gao, N. H. Cao and Z. X. Tang, Shaanxi Shifan Daxue Xuebao, Ziran Kexueban (J. Shaanxi Normal Univ., Natur. Sci.) **25**, no 3, 61 (1997).
- <sup>141</sup>B. Cui, Z. X. Tang, Zh. Zh. Guo and K. X. Chen, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) **18**, 1742 (1997).
- <sup>142</sup>Y. H. Li, X. Q. Ran and P. H. Chen, Zhongguo Xitu Xuebao (J. Chin. Rare Earth Soc.) **15**, no 2, 102 (1997); J. Rare Earths **15**, no 2, 113 (1997).
- <sup>143</sup>J. R. Liu, M. Ji, Zh. G. Li and Sh. G. Gao, Xibei Daxue Xuebao, Ziran Kexueban (J. Northwest Univ., Natur. Sci.) **28**, no 1, 44 (1998).

- <sup>144</sup>H. B. Liang, W. H. Han, G. L. Gao, D. J. Zhang and Q. Bo, Shaanxi Shifan Daxue Xuebao, Ziran Kexueban (J. Shaanxi Normal Univ., Natur. Sci) **27**, no 1, 73 (1999).
- <sup>145</sup>Sh. P. Liu, G. Q. Huang, Ch. D. Zhang and H. Y. Wang, Neimenggu Daxue Xuebao, Ziran Kexueban (Acta Sci. Natur. Univ. NeMongol) **30**, no 1, 53 (1999).
- <sup>146</sup>H. B. Liang, W. L. Wang, D. D. Hu and W. P. Zhang, Zhongguo Xitu Xuebao **17**, no 1, 81 (1999); J. Rare Earths **17**, no 1, 64 (1999).
- <sup>147</sup>F. Ren, Sh. L. Gao and Q. Zh. Shi, Xiamen Daxue Xuebao, Ziran Kexueban (J. Xiamen Univ., Natur. Sci. **39**, 485 (2000).
- <sup>148</sup>L. Li, H. Wang, Sh. P. Xia, M. Ch. Hu and Sh. Y. Gao, Wuji Huaxue Xuebao (Chin. J. Inorg. Chem. **19**, 201 (2003).
- <sup>149</sup>Zh. P. Qiao, L. H. Zhuo, Sh. Sh. Zhang and H. Wang, Wuji Huaxue Xuebao (Chin. J. Inorg. Chem.) **19**, 303 (2003).
- <sup>150</sup>Zh. F. Hao, H. B. Liang, Z. X. Tang, L. Yu, J. Yu and Y. W. Fang, Huaxue Xuebao (Acta Chim. Sinica) **61**, 853 (2003).
- <sup>151</sup>Y. G. Chen, Zh. Fang, Q. R. Zhang, K. Y. Yuan and Sh. F. Wang, Xiyou Jinshu (Chin. J. Rare Met.) **27**, 601 (2003).
- <sup>152</sup>H. Wang, X. F. Wang, F. X. Dong, X. Q. Ran, L. Li and Sh. Y. Gao, J. Chem. Eng. Data **52**, 731 (2007).
- <sup>153</sup>A. Habenschuss and F. H. Spedding, Cryst. Struct. Commun. **8**, 511 (1979).
- <sup>154</sup>N. P. Sokolova and E. A. Ukraintseva, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 1, 24 (1984).
- <sup>155</sup>J. G. Tang, C. K. S. Lee and L. A. Belfiore, J. Polym. Sci, Part B: Polym. Phys. **41**, 2200 (2003).
- <sup>156</sup>H. B. Chen, P. Zh. Yang, Ch. Y. Zhou, Ch. Y. Jiang and J. G. Pan, Cryst. Growth Design **6**, 809 (2006).
- <sup>157</sup>A. N. Zelikman, N. V. Baryshnikov, A. N. Khokhlov, Izv. Vyssh. Ucheb. Zaved., Tsvetn. Matall., no 6, 89 (1971).
- <sup>158</sup>Zh. Gong, P. H. Chen, Zh. Zh. Guo, J. H. Ma and Y. Sh. Chen, Huaxue Xuebao (Acta Chim. Sinica) **41**, 391 (1983).
- <sup>159</sup>N. P. Sokolova and E. A. Ukraintseva, Izv. Sibir. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk, no 1, 24 (1984).
- <sup>160</sup>V. V. Hong and D. V. Minh, Tap Chi Hoa Hoc (J. Chem.) **36**, no 4, 44 (1998).
- <sup>161</sup>G. C. Zhu, F. P. Li and M. G. Xiao, Trans. Nonferrous Met. Soc. China **13**, 1454 (2003).

- <sup>162</sup>Zh. Gong, P. H. Chen, Zh. Zh. Guo, J. H. Ma and Y. Sh. Chen, Zhongguo Xitu Xuebao (J. Chin. Rare Earth Soc.) **3**, no 2, 13 (1985).
- <sup>163</sup>Zh. Gong, P. H. Chen, Zh. Zh. Guo, J. H. Ma and Y. Sh. Chen, Huaxue Xuebao (Acta Chim. Sinica) **41**, 463 (1983).
- <sup>164</sup>Zh. Gong, P. H. Chen, Zh. Zh. Guo, J. H. Ma and Y. Sh. Chen, Gaodeng Xuexiao Huaxue Xuebao (Chem. J. Chin. Univ.) **4**, 493 (1983).
- <sup>165</sup>S. J. Ashcroft and C. T. Mortimer, J. Less-Comm. Met. **14**, 403 (1968).
- <sup>166</sup>V. I. Ivernova, V. P. Tarasova and M. M. Umanskii, Izv. Akad. Nauk SSSR, Ser. Fiz. **15**, 164 (1951).
- <sup>167</sup>J. H. Burns and J. R. Peterson, Inorg. Chem. **10**, 147 (1971).
- <sup>168</sup>C. Brouty and P. Herpin, Compt. Rend. C **272**, 2049 (1971).
- <sup>169</sup>V. V. Bakakin, R. F. Klevtsova and L. P. Soloveva, Zh. Strukt. Khim. **15**, 820 (1974).
- <sup>170</sup>C. J. Kepert, B. W. Skelton and A. H. White, Austr. J. Chem. **47**, 385 (1994).

Appendix 3

Agenda Item 8

Presentation to SSED from the editor of JPCRD

Dr. Allan Harvey

Delivered by Dr. Mark Salomon in Dr. Harvey's absence

NIST


**Remarks to SSED from Editor of  
Journal of Physical and Chemical  
Reference Data**

Allan H. Harvey

Physical and Chemical Properties Division  
National Institute of Standards & Technology  
Boulder, Colorado 80305

CSTL

NIST



"Very sorry I could not meet with you in person."

Allan Harvey  
aharvey@boulder.nist.gov

CSTL

NIST

**History of J. Phys. Chem. Ref. Data**

- 1972: Founded as part of Standard Reference Data Act (US government law)
- NIST Standard Reference Data Program has mission to disseminate reference (critically evaluated) data
- Journal jointly published by NIST and American Institute of Physics (AIP).
- Published quarterly, about 2000 pages/yr
- Information at <http://jpcrd.aip.org>

CSTL

NIST

**Editors of J. Phys. Chem. Ref. Data**

- Founding editor David Lide (1972-1992)
- Jean Gallagher (1993-1996)
- Malcolm Chase (1996-2007)
- Bob Watters (NIST Management Representative, ~2002-present)
- Don Burgess (2007-present)
- Allan Harvey (2007-present)
- Burgess & Harvey share technical responsibility, Harvey deals with Solubility Data Series and most other thermodynamics-related papers.

CSTL

NIST

**Solubility Data Series in JPCRD**

- Started in 1998 with SDS volume 66
- Over 20 volumes so far, many multi-part.
- Typically accounts for 30-40% of JPCRD pages in a given year.

CSTL

NIST

**Current SDS Volumes in JPCRD**

- Volume 86 (Ethers and Ketones in Water). 6 Parts. Parts 1-3 (ethers) published in June 2008 issue. Parts 4-6 in proofs, probably will be in Sept. 2008 issue.
- Volume 87 (Rare Earth Metal Chlorides). 3 Parts. Part 1 (Scandium Group) at publisher, probably will be in Dec. 2008 issue. Parts 2 & 3 not yet submitted.

CSTL

**NIST**

**Recent JPCRD/SDS Changes**

- Articles in more efficient "portrait" layout
- AIP Publishing requires references to be numerical, in sequence through article
- End-of-article indexing dropped (seemed unnecessary when computer can search PDF file), making publication faster
- Harvey and Salomon working out procedures for proof correction with less redundancy

**CSTL**

**NIST**

**More General JPCRD Issues**

- New Editors not making drastic changes
- Reconvened Editorial Board and will solicit more advice
- Looking for papers in other areas of "physical & chemical reference data"
- Encouraging more use of Supplemental Material for lengthy, peripheral material (maybe not relevant to SDS?)

**CSTL**

**NIST**

**Policy on Multi-Part Volumes**

- Splitting into too many parts is inefficient
- Very long single papers (200+ pages) may be difficult to handle
- Some countries' systems reward multiple papers
- Any division into parts must be logical in terms of subject matter
- No part should be smaller than about 30-40 printed pages.
- **Consult with Allan Harvey & Mark Salomon after you know about how long the work is (# data sheets), before finalizing the papers.**

**CSTL**

**NIST**

**How Can I Make Publication of my Volume Faster & Easier?**

- **Follow author guidelines** ([jprcd.aip.org](http://jprcd.aip.org))
- References numbered in order cited, as complete as possible.
- Equations numbered consecutively, using Word Equation Editor (or Mathtype)
- Proper use of italics for variables, roman type for chemical formulas, etc.
- Figures in EPS, TIF, or PDF

**CSTL**

**NIST**

**How Can I Make Publication of my Volume Faster & Easier? (continued)**

- Use Table feature of Word for tables. Number tables consecutively, but tables within data sheets are unnumbered.
- Use symbols available in Word. Degree sign (<sup>°</sup>), not superscript letter o. Minus sign (-), not hyphen (-). Middle dot (·) [hydrate formulas], not superscript period. Multiplication sign (×), not letter x.
- Section headings for main divisions and crit. evals, not necessarily each system

**CSTL**

**NIST**

**Future Thoughts**

- We look forward to continuing to publish SDS articles in JPCRD
- SDS pages/year could be increased a little, but not a lot
- Project for on-line database with SDS data dormant – would be nice to restart if could be done more efficiently. NIST can supply website and possibly funds, probably not labor.

**CSTL**



Appendix 4  
Agenda Item 11(b)  
**Report of the Liquid-Liquid Group**

**DRAFT**

**Report of the Liquid-Liquid Group  
Solubility and Equilibrium Data Subcommittee  
International Union of Pure and Applied Chemistry**

**26 July 2008  
Trinity College  
Dublin Ireland**

**Present:** A Maczynski, M Goral, D Shaw

**Projects Completed Since Last Meeting:**

Vol. 82. Alcohols with Water – Revised and Updated, A. Maczynski, D.G. Shaw, M. Goral and B. Wisniewska-Gocłowska.

- Part 1. C<sub>4</sub> Alcohols with Water, *JPCRD*, **36(1)**, 59 (2007).
- Part 2. C<sub>5</sub> Alcohols with Water, *JPCRD*, **36(1)**, 133 (2007).
- Part 3. C<sub>6</sub> Alcohols with Water, *JPCRD*, **36(2)**, 399 (2007).
- Part 4. C<sub>7</sub> Alcohols with Water, *JPCRD*, **36(2)**, 445 (2007).
- Part 5. C<sub>8</sub> – C<sub>17</sub> Alcohols with Water, *JPCRD*, **36(3)**, 685 (2007).

Vol. 83. Acetonitrile: Ternary & Quaternary Systems. V.P. Sazonov, D.G. Shaw, A. Skrzecz, N.I. Lisov and N.V. Sazonov, *JPCRD*, **36 (3)**, 733, 2007

Vol. 86. Ethers and Ketones with Water. A. Maczynski, D.G. Shaw, M. Goral and B. Wisniewska-Gocłowska.

- Part 1. C<sub>2</sub>–C<sub>5</sub> Ethers with Water, *JPCRD*, **37(2)**, 1119 (2008)
- Part 2. C<sub>6</sub> Ethers with Water, *JPCRD*, **37(2)**, 1147 (2008)
- Part 3. C<sub>7</sub>-C<sub>14</sub> Ethers with Water, *JPCRD*, **37(2)**, 1169 (2008)
- Part 4. C<sub>4</sub> and C<sub>5</sub> Ketones with Water, in press
- Part 5. C<sub>6</sub> Ketones with Water, in press
- Part 6. C<sub>7</sub>-C<sub>12</sub> Ketones with Water, in press

**Projects in Preparation:**

2007-046-1-500 Mutual Solubility of Esters with Water, M Goral. Marian Goral reported that compilation is complete and evaluation is underway; completion is expected in mid-2009.

2007-047-1-500 Nitriles C+3: Binary and Multicomponent Systems, V Sazonov. Valerii Sazonov could not be present, but communication was brought by Vladimir Valyashko. Work is continuing but completion date needs to be revised to end of 2009.

**New Project:**

M Goral and A Maczynski suggested a volume on the mutual solubility of non-aromatic halocarbons with water.

Appendix 5

Axenda Item 11(c)

**Report on activities in the field of Solid Solubilities**

# Report on activities in the field of Solid Solubilities

## W. Voigt

Dublin, 27<sup>th</sup> July 2008

### Solubility data of compounds relevant to mobility of metals in the environment

#### **Alkaline earth metal carbonates. Alex de Visscher, J. Vanderdeelen, E. Königsberger ( 2002-031-1-500)**

Once more the importance of this volume for the actual, world-wide efforts to improve the understanding of the interactions of CO<sub>2</sub> in the environment is emphasized.

Progress has been made in the last year, consisting in updating new papers, writing an outline of an introduction to the volume. Thereby it became clear that evaluation work is faced with a number of problems. The most important point is to keep a balance between pure empirical judgment of data quality and the use of thermodynamic modelling to relate data at different conditions (temperature, CO<sub>2</sub> pressure, type and concentration of background electrolytes). In order to limit the time for finishing the volume it is proposed not to deliver a complete thermodynamic model of the aqueous carbonate + CO<sub>2</sub> system, but to restrict modelling on certain ranges of temperature, composition, where it is necessary for the selection of experimental data sets. Although solubility constants will be reported, they are not the final purpose of the volume.

It is furthermore suggested to divide the volume into two or three parts. Starting with beryllium and magnesium carbonate (I) and proceeding with calcium carbonate (II). After completion of this work it will be decided, whether a third volume will be needed for strontium and barium carbonate or not.

#### **Metal carbonates (Mn, Fe, Co, Ni, Cu, Zn, Ag, Cd, Hg, Pb) C. Magalhães, H. Gamsjäger and K. Sawada ( 2002-032-1-500)**

Due to other priorities work on this volume is also postponed.

### Solubility data of compounds relevant to human health.

#### **Solubility of hydroxybenzoic acids and hydroxybenzoates A. Goto, H. Miyamoto (2002-036-1-500)**

A. Goto delivered a CD containing all compilations and evaluations done so far. Considering the large number of compilation sheets (more than 350) it is suggested to divide the volume into at least 3 parts and to prepare as a first one "Solubility of hydrobenzoic acid and its salts in aqueous solution". Thereby reformatting into the new JPCRD format has to be done.

#### **Solubility of halogenated aromatic hydrocarbons A. Goto, R. Goto, M. Makino, and H. Miyamoto (2002-037-1-500)**

Due to the limited amount of data available for evaluation, at the Torino meeting the decision was made, to search for another way of publishing the collected data. Prof. Schüürmann was asked to check the possibility of using structure-property models established in his lab for correlating predicted solubilities with the data collected within this project. As a result it came out, that the correlation at 25 °C appears good for several incremental prediction methods, mostly within half to one order of magnitude. In addition it became known that in the lab of Prof. Schüürmann exists an evaluated data base for solubilities of halogenated aromatic hydrocarbons. Dr. Makino has to contact Prof. Schüürmann to clarify the quality of this data base and to develop an evaluation strategy based on the structure relationships.

**Solubility of substances related to urolithiasis. E. Königsberger and L.-C. Königsberger (2002-035-1-500)**

The authors ask for one year more prolongation.

**Rare Earth Metal Chlorides in Water and Aqueous Systems. T. Mioduski, C. Guminski and D. Zeng.**

**As promised at the Torino meeting the work about the chlorides will be finished this year. The volume will be divided into three parts:**

- Part 1. Scandium Group (Sc, Y, La)
- Part II: Light Lanthanides (Ce-Eu)
- Part III: Heavy Lanthanides (Gd-Lu)

Part I is already in print, part II will be ready within 2 months and part III at the end of 2008.

**Solubility in aqueous systems containing LiNO<sub>3</sub> and/or NaNO<sub>3</sub>. I. Eysseltova. (2007-044-1-500)**

Work on this volume is under way. The lithium nitrate part will be ready end of 2008. The sodium nitrate part is much more extensive and it will take the whole year 2009 to finish.

**Seawater system components**

As proposed in Turin 2007 W. Voigt and D. Zeng will submit a project application form for a volume solubility of “MgCl<sub>2</sub> and CaCl<sub>2</sub> in aqueous solutions”.

I. Eysseltova accepted to take over the responsibility for the volume of K<sub>2</sub>SO<sub>4</sub>-H<sub>2</sub>O at the Torino meeting. She has received the compilation sheets from Prof. R. Bouaziz in electronic form. However, since the work on the LiNO<sub>3</sub>/NaNO<sub>3</sub> project absorbs her capacity entirely the submission form for a K<sub>2</sub>SO<sub>4</sub> volume will be postponed.

**Proposal of new projects**

C. Guminski and D. Zeng propose the preparation of two new volumes entitled:

**Aqueous lanthanoide bromide systems. C. Guminski, D. Zeng, T. Mioduski,**

**Aqueous lanthanoide bromide systems. C. Guminski, D. Zeng, T. Mioduski,**

It will still be a continuation of the work started by T. Mioduski. Up-dates of the last 20 years have to be made. Thereby especially including new Chinese publications. For the bromide systems 100 compilation sheets and for the iodide approx. 50 compilation sheets are expected. Further members for the task group are needed. During the 13. ISSP colleagues from the Institute of Nuclear Waste Disposal at the Research Center Karlsruhe (V. Neck) will be asked

for collaboration, since there is a natural interest in lanthanide chemistry as analogues to actinides.

W. Voigt

Appendix 6  
Agenda Item 13  
**Project Expenses vs Budget**

## Project Expenses vs Budget

Through 28 May 2008	Actual	Budget	Over/ (Under) Budget	% of Budget	Planned End Date
<b>500-Analytical</b>					
Fransozini Fund-Income	1,490	1,758	(268)	85%	
NIST-2003 Gamsjäger	4,650	7,900	(3,250)	59%	
NIST-2004 Gamsjäger	0	8,900	(8,900)	0%	
SSED Wiley Royalties	1,592	1,591	1	100%	
2002-025-1-500 Hala	–	–	–	0%	<b>1-Jul-2004</b>
2002-031-1-500 Königsberger	1,500	1,500	–	100%	30-Jun-2008
2002-032-1-500 Gamsjäger	501	1,750	(1,249)	29%	<b>31-Dec-2007</b>
2002-035-1-500 Konigsberger	–	–	–	0%	<b>31-Dec-2007</b>
2002-036-1-500Goto/Miyamoto	0	1 000	(1 000)	0%	30-Jun-2008
2002-037-1-500 Goto	0	600	(600)	0%	30-Sep-2008
2002-038-1-500 Lorimer	–	–	–	0%	<b>31-Dec-2004</b>
2002-044-1-500 Scharlin	1,124	1,500	(376)	75%	<b>31-Dec-2007</b>
2002-045-1-500 Knox	–	–	–	0%	30-Jun-2008
2005-017-1-500C Shaw	2,800	2,800	–	100%	<b>Completed</b>
2005-033-1-500 Miyamoto	–	–	–	0%	<b>31-Dec-2006</b>
2006-032-1-500 Goral	–	–	–	0%	<b>31-Dec-2006</b>
2006-033-1-500 Guminski	–	–	–	0%	<b>1-Dec-2007</b>
2006-034-1-500 Clever	–	–	–	0%	<b>31-Dec-2007</b>
2007-044-1-500 Eysseltova	654	1,000	(346)	65%	31-Dec-2009
2007-045-1-500 Fogg	–	–	–	0%	30-Sep-2009
2007-046-1-500 Goral	–	–	–	0%	31-Dec-2009
2007-047-1-500 Sazonov	–	–	–	0%	31-Dec-2008