COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Hexachloro-1,3-butadiene; C_4Cl_6; [87-68-3]

ORIGINAL MEASUREMENTS:

Shevtsova, Z.N.; Korshunov, B.G.; Safonov, V.V.; Kogan, L.M.; Gudkova, V.I.

Zh. Neorg. Khim. 1968, *13*, 3096-9; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1968, *13*, 1596-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition, densities, viscosities and refractive indices of saturated solutions.

$t/^\circ C$	solubility[a] mass %	mol kg^{-1}	$d/g\ cm^{-3}$	η/P	n_D^{20}	nature of the solid phase
25	0.040	0.00163	1.681	0.0382	1.5564	$LaCl_3.4H_2O$
50	0.042	0.00171	1.642	0.0305	1.5554	$LaCl_3.4H_2O$
75	0.057	0.00233	1.614	0.0244	1.5547	$LaCl_3.2H_2O$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Depending on temp, equil was established after 12 d at 25°C, 10 d at 50°C, and 7 d at 75°C.

Chloride was detd by the Volhard method, and lanthanum detd gravimetrically by precipitating as the oxalate and igniting to the oxide. Lanthanum was also detd by titration with Trilon B with Xylene Orange indicator.

The composition of the solid phase was established by chemical analysis, and confirmed by X-ray analysis.

Samples of the solid phases were also studied thermographically after removal of excess solvent by washing with absolute ethyl ether which is claimed not to change the composition of hydrate.

Authors state that at 110°C the equil solid phase is the monohydrate, but no solubility data are given. At 120°C partial hydrolysis takes place with formation of LaOCl.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3.7H_2O$ prepd by dissolving La_2O_3 in HCl, evaporating and cooling, and then recrystallized and dried in a desiccator. La_2O_3, 99.9% pure, contained oxide impurities of other rare earth metals, Fe (0.01%), Ca (0.01-0.05 %), and Cu (0.01%). Analysis of the heptahydrate gave the following (in mass % units): La 37.42; Cl 28.67; H_2O 33.91.

Purified solvent (method not specified) had the following properties: d_4^{20} = 1.6807 g/ml, and n_D^{20} = 1.5543.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: accuracy ± 0.1 K (authors).

REFERENCES:

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

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

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Concentration of CH_3OH
T/K = 295

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

Initial Concn Methanol mol dm^{-3}	$LaCl_3$ solubility[a] mol dm^{-3}
1.0	0.00225
1.5	0.00580
2.0	0.01017
2.5	0.01600
3.0	0.02350
3.5	0.03155
4.0	0.04169

[a] Solid phase is $LaCl_3.CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous $LaCl_3$ at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

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

SOURCE AND PURITY OF MATERIALS:

Source and purity of $LaCl_3$ not specified. Anhydrous $LaCl_3$ prepared by method described in (2).

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

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K.

REFERENCES:

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

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Alcohols; ROH

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

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Concentration of ROH
T/K = 295

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Numerical data were given only for the $LaCl_3$ - CH_3OH - C_6H_6 system (see the compilation for this system). The remaining data were presented graphically and in the form of the equation

$$K = [LaCl_3.nROH] / [ROH]^n \qquad [1]$$

In this equation $[LaCl_3.nROH]$ is the solubility in units of mol dm^{-3}, [ROH] is the total alcohol concentration in units of mol dm^{-3}, and n is the solvate number in solution (see ref. 1). According to this equation, n is calculated from the slope of a plot of the logarithm of the solubility, log $[LaCl_3.nROH]$, against log [ROH] . Thus the solubility of $LaCl_3$ can be calculated as a function of ROH concentration using the reported values of n and K (see table below). The alcohol concentrations were varied from 1-5 mol dm^{-3}.

alcohol	n	-log K	nature of the solid phase
methanol; CH_4O; [67-56-1]	2	2.58	$LaCl_3.CH_3OH$
ethanol; C_2H_6O; [64-17-5]	1	2.05	$LaCl_3.3C_2H_5OH$
	3	2.57	
1-propanol; C_3H_8O; [71-23-8]	1	2.10	$LaCl_3.3C_3H_7OH$
	3	3.29	

For those systems where two values of n and K are reported, the overal solubility of $LaCl_3$ is obtained by using the values for n-K in eq. [1] which give the greater solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous $LaCl_3$ at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

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

SOURCE AND PURITY OF MATERIALS:

Source and purity of $LaCl_3$ not specified. Anhydrous $LaCl_3$ prepared by method described in (2).

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

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K

REFERENCES:

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

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

T/K = 273.2, 298.2, 323.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

	solubility $LaCl_3$/mol kg^{-1}	
t/°C	a	b
0	---	2.23
25	2.45	2.44
50	---	2.98

[a] Initial solid was $LaCl_3.4CH_3OH$. Equilibrated solid phase analysed and found to contain 4.0 moles methanol per mole of salt.

[b] Initial solid was anhydrous $LaCl_3$. Equilibrated solid phase not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Lanthanum determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $LaCl_3.4CH_3OH$ confirmed by ^1H NMR and x-ray diffraction.

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

COMMENTS AND/OR ADDITIONAL DATA:

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

SOURCE AND PURITY OF MATERIALS:

La_2O_3 of at least 99.9% purity dissolved in HCl to produce the heptahydrate. The adduct $LaCl_3.4CH_3OH$ prepared by dissolving the heptahydrate in a small excess of o-methylformate followed by distillation and crystallization from methanol. The anhydrous salt prepared by dehydration as described in (3).

Methanol was purified and dried by the Vogel method.

ESTIMATED ERROR:

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

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

REFERENCES:

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

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

ORIGINAL MEASUREMENTS:

King, F. E.

Masters Thesis.

University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

t/°C	g La_2O_3 in 10 cc satd sln experimental	average[a]	density/g cm^{-3} exptl	av[a]	solubility of $LaCl_3$[a,b] mol dm^{-3}	mol kg^{-1}
0	1.8753	1.8525	1.0487	------	1.1372	1.0844
0	1.8297		------			
10	2.0314	2.0431	1.0774	-----	1.2541	1.1640
10	2.0547					
15	2.1682	2.1350	------	-----	1.3106	------
15	2.1017					
20	2.8246	2.8292	1.1751	1.1749	1.7367	1.4782
20	2.8337		1.1747			
25	2.7874	2.7704	1.1697	1.1693	1.7006	1.4544
25	2.7534		1.1689			
25[c]	2.9355	2.9304	1.1839	1.1837	1.7989	1.5196
25[c]	2.9252		1.1835			
30	2.7338	2.7361	1.1510	1.1510	1.6796	1.4593
30	2.7384		1.1509			
40	2.8743	2.8725	1.1719	1.1719	1.7633	1.5047
40	2.8707		1.1718			
40[c]	3.6374	------	------	------	2.2328	------
50	4.4695	4.4721	1.4100	1.4101	2.7452	1.9468
50	4.4747		1.4102			

a,b Calculated by compilers. b. From average values of mass La_2O_3 and satd sln densities.

c. Repeat analyses after lowering temp: compilers presume that after reaching 50°C, the temp was lowered first to 40°C, then to 25°C. These points probably represent metastable equilibria.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Salt and ethanol placed in 250 cc stoppered bottles and mechanically agitated in a thermostat for at least 24h. Bottles were sealed by placing rubber tubing over the stopper and neck of each bottle. Slns allowed to settle for at least 12 h and duplicate 10 cc aliquots removed with pipet previously rinsed with the sln. Analyses performed by evapn of ethanol, addn of water, and pptn of lanthanum with oxalic acid. The oxalate was filtered and ignited to const mass as the oxide. Densities measured with a pycnometer, but author states loss by evapn resulted in slightly low values. Soly detns using single bottle by (1) starting at 0°C and raising the temp for the next detn, and (2) by cooling the bottle to a lower temp for a second analysis. Salt and ethanol added to the bottle as needed. The results of the second duplicate analysis (i.e. by cooling) resulted in higher soly values (see table). Several samples of the solid were taken for analyses, but *temperature not specified*. These samples were dried in vac over P_2O_5, weighed, converted to the oxalate and ignited to the oxide. Two analyses gave 2.77 and 1.39 molecules of crystallization.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd from spectro-pure La_2O_3 by addn of aq HCl, and evapn to the point of crystn. Crystals dried in atm of dry HCl for 24 h at room temp followed by slow heating in dry HCl until anhydr salt obtained. The salt was stored in a vac desiccator over P_2O_5. The salt was analysed for H_2O by gravimetric analysis (oxalate-oxide method), but results not given: presumably little or no water found. Ethanol obtained from the stock room (i.e. source and purity unknown) was dried with anhydr Na_2SO_4.

ESTIMATED ERROR:

Soly: precision no better than ± 5% (compilers)

Temp: precision ± 1 K.

REFERENCES:

1. Some of the data from King's Thesis were published in graphical form by Hopkins, B. S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]
(2) Ethanol; C_2H_6O; [64-17-5]

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

T/K = 273.2, 298.2, 323.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	Solubility $LaCl_3$/mol kg^{-1} a	b
0	---	1.14
25	1.26	1.34
50	---	1.97

a. Initial solid was $LaCl_3.3C_2H_6O$. Equilibrated solid phase analysed and found to contain 3.6 moles ethanol per mole of $LaCl_3$.

b. Initial solid was anhydrous $LaCl_3$. The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Lanthanum determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $LaCl_3 \cdot 3C_2H_6O$ confirmed by ^1H NMR and x-ray diffraction.

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

COMMENTS AND/OR ADDITIONAL DATA:

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

SOURCE AND PURITY OF MATERIALS:

La_2O_3 of at least 99.9% purity dissolved in HCl to produce the heptahydrate. The adduct $LaCl_3 \cdot 3C_2H_6O$ prepared by dissolving the hydrate in a small excess of o-ethylformate followed by distillation and crystallization from ethanol. The anhydrous salt was prepared by dehydration as described in (3).

Ethanol (Fluka) was used as received. Purity and absence of water was confirmed by NMR method.

ESTIMATED ERROR:

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

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

REFERENCES:

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

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

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

ORIGINAL MEASUREMENTS:

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

Zh. Neorg. Khim. 1975, *20*, 1479-83; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1975, *20*, 830-2.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $LaCl_3.6H_2O$ in 96.8 % C_2H_5OH[a]

	sample 1	sample 2	sample 3	sample 4	mean solubilities	
t/°C	g/100 g[b]	g/100 g	g/100 g	g/100 g	g/100 g	mol kg^{-1}[c]
20	40.05	40.23	39.82	40.23	40.08	1.134
30	44.30	44.25	44.18	44.21	44.23	1.252
40	48.70	48.73	48.99	48.66	48.69	1.378
50	56.42	56.38	56.59	56.61	56.50	1.599
60	71.25	71.14	71.14	71.36	71.23	2.016

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

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

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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

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

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

The hexahydrate melted at 93.9 - 94.5°C.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analysed for the metal by titrn with Trilon B, and for Cl by the Volhard method. Found (%) for La: 39.20, 39.39 (calcd 39.33). Found (%) for Cl: 30.10, 30.08 (calcd 30.16). 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.8% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

ORIGINAL MEASUREMENTS:

Racster, L.V.
Masters Thesis.
University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salamon and T. Mioduski

EXPERIMENTAL VALUES:

t/°C	g La_2O_3 in 10 cc of satd sln experimental	average[a]	density/g cm^{-3} exptl	av[a]	solubility of $LaCl_3$[a,b] mol dm^{-3}	mol kg^{-1}
10	1.9275	1.8843	1.3580	------	1.1567	0.8518
10	1.8410		------			
15	2.1165	2.1058	1.3856	------	1.2926	0.9329
15	2.0950		------			
20	1.1443	1.1466	1.2597	1.2597	0.7038	0.5587
20	1.1488		1.2597			
30	2.7345	2.7578	1.4357	1.4356	1.6929	1.1792
30	2.7810		1.4355			
40	3.1325	3.0523	1.4535	1.4570	1.8736	1.2860
40	2.9720		1.4605			
50	3.0810	3.0188	1.4410	1.4413	1.8531	1.2858
50	2.9565		1.4415			
60	2.5923	2.6082	------	------	1.6010	------
60	2.6240		------			

a. Calculated by compilers.
b. Calculated by compilers from average mass La_2O_3 and average density of satd sln.

The solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

$LaCl_3$ and solvent placed in 250 cc glass stoppered bottle and mechanically agitated for 24 h. Rubber tubing placed over stopper and neck of bottle and end of tubing sealed with a rubber stopper to prevent water from entering the bottle. Slns allowed to settle for 12-18 h, but slight turbidity persisted, particularly at the lowest and highest temps. At 60°C sln turbidity was significant and appeared different leading author to speculate possible reaction between solute and solvent. Results at 50°C and 60°C said to be approximate. Duplicate 10 cc aliquots pipetted from the bottle for each temp. Each aliquot diluted with 25 cc H_2O and the rare earth pptd as the oxalate with oxalic acid. The oxalate was filtered, ignited and weighed as the oxide. Densities of satd slns detd pycnometrically using pycnometer calibrated at each temp. Densities at 10°C and 15°C may be high due to the condensation of atm water on the surface of the pycnometer.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd by addn of HCl to spectro-pure La_2O_3, and evapn of solvent until crystn. Crystals dehydrated by method of Kremers (2). Salt analysed for presence of H_2O gravimetrically by conversion to oxalate and ignition to the oxide. No water of crystn was found. Ethylene glycol (source and purity not specified) was distilled and initial 5% of distillate discarded. The distilled solvent was stored in a flask sealed with paraffin.

ESTIMATED ERROR:

Soly: Precision no better than ± 3%, and accuracy is probably poor (compilers).

Temp: Not specified.

REFERENCES:

1. Some data from Racster's Thesis were published in graphical form by Hopkins, B.S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

2. Kremers, H.C. *J. Am. Chem. Soc.* 1925 *17*, 298.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

ORIGINAL MEASUREMENTS:

West, D.H.
Masters Thesis.
University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g La_2O_3 in 10 cc of saturated sln			solubility of $LaCl_3$[a,b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	1.8057	1.8263	1.8160[c]	1.1148
20	2.0097	2.0251	2.0174	1.2384
30	2.3641	2.3763	2.3702	1.4550
40	2.8013	2.7879	2.7946	1.7115

a. Calculated by compilers.

b. Calculated by compilers from average mass La_2O_3.

c. Author gives 1.8146 g for this average.

The solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 cc of alcohol and excess salt placed in 250 cc glass stoppered bottle, and rubber tubing placed over the stopper and neck of the bottle and a rubber bung fitted into the open end of the tubing to prevent leakage of water into the bottle. The bottle was immersed in a thermostat and mechanically agitated for at least 12 h. The saturated solutions were then permitted to settle for a minimum of 12 h, and duplicate 10 cc aliquots removed with a pipet Water was added to the aliquots and the sln heated and oxalic acid added to precipitate the rare earth oxalate. The precipitate was filtered, washed with distilled water, and ignited and weighed as the oxide.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd by addn of HCl to spectro-pure La_2O_3, and evaporating the sln to a paste which crystallized upon cooling. The hydrate was dried in a stream of dry HCl by slowly increasing the temp. The anhyd salt was stored in cork-stoppered bottles in a desiccator over P_2O_5. Analysis by conversion to the oxalate and ignition to the oxide showed the salt to be anhydr. Commercial alcohol placed over CaO for 1 week and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was negative.

ESTIMATED ERROR:

Soly: precision probably within ± 3 % (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. Some data from West's Thesis were published in graphical form by Hopkins, B.S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

ORIGINAL MEASUREMENTS:

West, D.H.
Masters Thesis.
University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g La_2O_3 in 10 cc of saturated sln			solubility of $LaCl_3$ [a,b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	0.0283	0.0269	0.0276	0.0169
20	0.0335	0.0319	0.0327	0.0201
30	0.0656	0.0642	0.0649	0.0398
40	0.0884	0.0906	0.0895	0.0549

a. Calculated by compilers.

b. Calculated by compilers from average mass La_2O_3.

The solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 100 cc of alcohol and excess salt placed in 250 cc glass stoppered bottle, and rubber tubing placed over the stopper and neck of the bottle and a rubber bung fitted into the open end of the tubing to prevent leakage of water into the bottle. The bottle was immersed in a thermostat and mechanically agitated for at least 12 h. The saturated solutions were then permitted to settle for a minimum of 12 h, and duplicate 10 cc aliquots removed with a pipet. Water was added to the aliquots and the sln heated and oxalic acid added to precipitate the rare earth oxalate. The precipitate was filtered, washed with distilled water, and ignited and weighed as the oxide.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd by addn of HCl to spectro-pure La_2O_3, and evaporating the sln to a paste which crystallized upon cooling. The hydrate was dried in a stream of dry HCl by slowly increasing the temp. The anhyd salt was stored in cork-stoppered bottles in a desiccator over P_2O_5. Analysis by conversion to the oxalate and ignition to the oxide showed the salt to be anhydr. Commercial alcohol placed over CaO for 1 week and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was negative.

ESTIMATED ERROR:

Soly: precision probably within ± 3 % (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. Some data from West's Thesis were published in graphical form by Hopkins, B.S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Two results were reported for 25°C.

1. Starting with $LaCl_3.3C_3H_8O$, the solubility was reported to be 0.004 mol kg^{-1}. The equilibrated solid phase was analysed and found to contain a 4.5 - 5.1 moles of iso-propanol per mole of $LaCl_3$.

2. Starting with the anhydrous salt, the solubility was reported to be 0.016 mol kg^{-1}. The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method as in (1,2). Mixtures were equilibrated for at least 4 days. Prolonged operations were performed in a dry box. Lanthanum determined by titration with $(NH_4)_3H(EDTA)$ using a small amount of urotropine buffer and Xylenol Orange indicator. Chloride was determined by potentiometric titration with $AgNO_3$ solution. Composition of the adduct $LaCl_3.3C_3H_8O$ confirmed by ^1H NMR and x-ray diffraction.

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

COMMENTS AND/OR ADDITIONAL DATA:

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

SOURCE AND PURITY OF MATERIALS:

La_2O_3 of at least 99.9% purity dissolved in HCl to produce the hepthahydrate. The adduct $LaCl_3.3C_3H_8O$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and trans-solvation of the methanol complex with 2-propanol. The anhydrous salt was prepared by dehydration as described in (3).

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

ESTIMATED ERROR:

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

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

REFERENCES:

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

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) 1,2,3-propanetriol (glycerol); $C_3H_8O_3$; [56-81-5]

ORIGINAL MEASUREMENTS:

Dawson, L. R.
Masters Thesis.
University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g La_2O_3 in 25 cc satd sln		density/g cm^{-3}		solubility of $LaCl_3$ [a,b]	
t/°C	experimental	average	exptl	ave [c]	mol dm^{-3}	mol kg^{-1}
10	0.2423	0.2495	1.2732	1.2681	0.0613	0.0483
10	0.2566		1.2630			
20	2.6962	2.6918	1.3592	1.3595	0.6609	0.4862
20	2.6873		1.3598			
25	2.0927	2.0891	1.3330	1.3332	0.5130	0.3848
25	2.0855		1.3334[d]			
30	1.3266	1.3277	1.3114	1.3123	0.3260	0.2484
30	1.3287		1.3131			
40	2.3058	2.3049	1.3639	1.3642	0.5659	0.4149
40	2.3039		1.3645			
50	2.4841	2.4812	1.3514	1.3494	0.6092	0.4515
50	2.4782		1.3474			
60	2.5302	2.5213	------	------	0.6191	------
60	2.5123					

a. Calculated by compilers.
b. Based on average mass of La_2O_3.
c. Recalculated by compilers.
d. Value tabulated in Thesis is 1.334, but this is obviously a typographical error.

The solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

About 175 cc glycerol placed in 250 cc glass stoppered bottles and "liberal amounts" of salt added. Mixtures mechanically agitated in thermostat for 24 h, and even after 15 h of standing turbidity was present. All analyses carried out on turbid slns. For analyses, duplicate 25 cc aliquots (from each bottle) were taken and the rare earth pptd as the oxalate. The ppt was filtered, ignited, and weighed as the oxide. Author states the presence of turbidity has small effect on the overall accuracy of the soly detns. Densities of satd slns determined by withdrawing samples from the bottles, placing them into a pycnometer, and weighing "as quickly as possible."

COMMENTS AND/OR ADDITIONAL DATA:

Since there is a sharp rise in soly from 10°C to 20°C followed by a sharp decrease to 30°C at which point the soly begins to rise again, it is evident that the solid phase in equil with the satd slns is changing. Unsuccessful attempts were made to isolate and identify the solid phases.

SOURCE AND PURITY OF MATERIALS:

$LaCl_3$ prepd by adding HCl to spectro-pure La_2O_3, and evaporating the solvent to the point of crystallization. Dehydration was carried out in a stream of dry HCl first at room temp 24 h, then at 100°C for ~ 12 h, 110°C for ~ 6 h, and 200°C for 3-4 h. HCl prepd from NaCl + H_2SO_4 and passed through H_2SO_4 drying towers. Glycerol (presumably c.p. or A.R. grade: compilers) distilled at reduced pressure and the "first portion" rejected (no other details given).

ESTIMATED ERROR:

Soly: based upon precision in analyses and temp control, overall precision in soly around ± 3%. Error in accuracy due to turbidity is unknown.

Temp: precision ± 0.5K except for the 10°C run where precision was ± 1.5 K.

REFERENCES:

1. Some of the data from Dawson's Thesis were published in graphical form by Hopkins, B. S.; Quill, L. L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

ORIGINAL MEASUREMENTS:

McCarty, C.N.

Masters Thesis. University of Illinois. Urbana, IL. 1933.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	La_2O_3[a] g/25 cc	$LaCl_3$[b] g/dm^3	$LaCl_3$[b] mol/dm^3
0	0.5793	34.89	0.1422
10	0.9068	54.61	0.2227
20	1.1026	66.40	0.2707
30	1.2228	73.64	0.3002
40	1.3077	78.75	0.3211
50	1.5886	95.67	0.3901

a. Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $LaCl_3$ from preparations 1 and 2.

b. Recalculated by the compilers using 1977 IUPAC recommended atomic masses (1).

The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in bottles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber bung fitted into the upper end of the tubing. After equilibration, the solutions were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

ESTIMATED ERROR:

Soly: precision probably within 3 % (compilers).

Temp: precision ± 0.2 K (author).

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 week and then distilled. A middle portion (fraction not specified) was retained and stored in a stoppered flask: b.p. 123°C. La salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide (no details) and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystallized salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extraction the chloride was carried out with HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 and by passing the resulting HCl through H_2SO_4 drying towers.

REFERENCES:

1. IUPAC Commission on Atomic Weights, *Pure Appl. Chem.* 1979, *51*, 405.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

ORIGINAL MEASUREMENTS:

McCarty, C.N.

Masters Thesis. University of Illinois. Urbana, IL. 1933.[1]

VARIABLES:

Temperature

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	La_2O_3 [a] g/25 cc	$LaCl_3$ [b] g/dm^3	$LaCl_3$ [b] mol/dm^3
0	0.2746	16.54	0.0674
10	0.4968	29.92	0.1220
20	0.7331	44.15	0.1800
30	0.9991	60.17	0.2453
40	1.1125	67.00	0.2732
50	1.3391	80.64	0.3288

a. Apparently these are average values of at least two analyses from a given bottle. The author did not indicate whether there were any differences in results using $LaCl_3$ from preparations 1 and 2.

b. Recalculated by the compilers using 1977 IUPAC recommended atomic masses (1).

The equilibrated solid phase was not analysed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. About 75-100 cc of solvent + excess salt were placed in bottles and agitated in a thermostat for at least 12 h. Ice + water was used for the 0°C measurements. The bottles were fitted with ground glass stoppers and sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber bung fitted into the upper end of the tubing. After equilibration, the solutions were allowed to settle for at least 12 h, and using a calibrated 25 cc pipet, two samples were removed for analysis. The samples were evaporated to dryness and dissolved in aq HCl and pptd as the oxalate by addn of oxalic acid. The samples were filtered, washed with dist water and ignited to constant weight as the oxide. The oxide was found to be insoluble in the organic solvent.

SOURCE AND PURITY OF MATERIALS:

Commercial solvent was permitted to stand over CaO for at least 1 week and distilled. A middle portion (fraction not specified) was retained and stored in a stoppered flask; b.p. 134°C. La salts prepd in 1925 as double ammonium nitrates were of "spectroscopic purity" and converted to the oxide (no details) and the anhydr chloride prepd by two methods. 1. The oxide was dissolved in aq HCl and the excess HCl evapd. The crystallized salt was dehydrated by heating in the presence of dry HCl first at 100°C for several h, then at 200°C. 2. The rare earth benzoate was pptd from the aq chloride or nitrate with sodium benzoate, and the benzoate dehydrated by heating to 110°C for at least 24 h. Extraction the chloride was carried out with HCl satd ether, and the resulting chloride heated at 60°C first in a stream of dry HCl and then in dry air. The salt was stored in a desiccator over P_2O_5. Dry HCl was prepd from NaCl + H_2SO_4 and by passing the resulting HCl through H_2SO_4 drying towers

ESTIMATED ERROR:

Soly: precision probably within 3 % (compilers).

Temp: precision ± 0.2 K (author).

REFERENCES:

1. IUPAC Commission on Atomic Weights, *Pure Appl. Chem.* 1979, *51*, 405.

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Dressler, H.

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

VARIABLES:

Room temperature: T/K = 293-298

PREPARED BY:

Mark Salomon and Tomasz Mioduski

EXPERIMENTAL VALUES:

solvent			solubility[a,b] mass %	solubility[a,b] mol kg^{-1}
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	0.6	0.02_5
1-methoxyoctane;	$C_9H_{20}O$;	[929-56-6]	0.1	0.004

[a] Molalities calculated by the compilers.

[b] Solid phase ratios La:ether found to be 1: > 2.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at room temperature until equilibrium was attained. The anhydrous reagents were handled in a dry box containing P_4O_{10}.

La was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum chloride; $LaCl_3$; [10099-58-8] (2) Ethers	Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U. *Z. Chem.* 1968, *8*, 472-3.
VARIABLES:	**PREPARED BY:**
Room temperature: T/K around 298	Mark Salomon and Tomasz Mioduski

EXPERIMENTAL VALUES:

solvent			$LaCl_3$ solubility[a,b] mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O$;	[5137-45-1]	0.4	0.016
1,3-dioxolane;	$C_3H_6O_2$;	[646-06-0]	0.5	0.020
1,4-dioxane;	$C_4H_8O_2$;	[123-81-1]	0.02	0.0008

[a] Molalities calculated by the compilers.

[b] Nature of solid phases not specified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isothermally agitated at 25°C or at room temperature. Authors state that the difference found for the solubilities was within experimental error limits.

La determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was preapred by the method of Taylor and Carter (1).

No other information given.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

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

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

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

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

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Concentration of CH_3OH
T/K = 295

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

Initial Concn Methanol mol dm^{-3}	$LaCl_3$ solubility[a] mol dm^{-3}
1.5	0.01320
2.0	0.02972
2.5	0.05482
3.0	0.09300
3.5	0.13804
4.0	0.21380

[a] Solid phase is $LaCl_3.CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous $LaCl_3$ at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

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

SOURCE AND PURITY OF MATERIALS:

Source and purity of $LaCl_3$ not specified. Anhydrous $LaCl_3$ prepared by method described in (2).

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

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K.

REFERENCES:

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

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$; [10099-58-8]

(2) Alcohols; ROH

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

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Concentration of ROH

T/K - 295

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Numerical data were given only for the $LaCl_3$ - CH_3OH - $C_4H_8O_2$ system (see the compilation for this system). The remaining data were presented graphically and in the form of the equation

$$K = [LaCl_3.nROH] / [ROH]^n \qquad [1]$$

In this equation $[LaCl_3.nROH]$ is the solubility in units of mol dm^{-3}, [ROH] is the total alcohol concentration in units of mol dm^{-3}, and n is the solvate number in solution (see ref. 1). According to this equation, n is calculated from the slope of a plot of the logarithm of the solubility, log $[LaCl_3.nROH]$, against log [ROH]. Thus the solubility of $LaCl_3$ can be calculated as a function of ROH concentration using the reported values of n and K (see table below). The alcohol concentrations were varied from 1-5 mol dm^{-3}.

alcohol	n	-log K	nature of the solid phase
methanol; CH_4O; [67-56-1]	3	2.36	$LaCl_3.CH_3OH$
ethanol; C_2H_6O; [64-17-5]	3	3.10	$LaCl_3.3C_2H_5OH$
1-propanol; C_3H_8O; [71-23-8]	1 2	2.90 3.45	$LaCl_3.3C_3H_7OH$

For the last system where two values of n and K are reported, the overall solubility of $LaCl_3$ is obtained by using the values for n-K in eq. [1] which give the greater solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used as described in (1). Solvent mixtures of known alcohol concentration were saturated with anhydrous $LaCl_3$ at 22 ± 1°C. Equilibrium was confirmed from constancy of the rare earth metal concentration upon repeated analyses.

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

SOURCE AND PURITY OF MATERIALS:

Source and purity of $LaCl_3$ not specified. Anhydrous $LaCl_3$ prepared by method described in (2).

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

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision ± 1 K

REFERENCES:

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

COMPONENTS:

(1) Lanthanum chloride; $LaCl_3$;
[10099-58-8]

(2) Hexamethylphosphorotriamide;
$C_6H_{18}N_3OP$;
[680-31-9]

ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. 1977, *22*, 1761-6;
Russ, J. Inrog. Chem. (Engl. Transl.) 1977, *22*, 955-8.

VARIABLES:

Room temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

Starting with anhydrous $LaCl_3$, the solubility at 25 ± 3°C was given as

$$0.106 \pm 0.002 \text{ mol dm}^{-3}$$

Starting with the solvate $LaCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C was given as

$$0.107 \pm 0.002 \text{ mol dm}^{-3}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature (25 ± 3°C) until eqilibrium was reached. Aliquots were withdrawn periodically and analysed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{\frac{1}{2}}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analysed and found to be $LaCl_3.3C_6H_{18}N_3OP$.

The solvate was analysed for metal content by complexometric titrn, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate also carried out by x-ray analysis.

SOURCE AND PURITY OF MATERIALS:

Anhyd $LaCl_3$ prepd similar to that in (1) by subliming NH_4Cl from a mixt of $LaCl_3$ and 6 moles of NH_4Cl in a stream of inert gas at 200-400°C (LaOCl content less than 3%). The solvent was purified as in (2).

$LaCl_3.3C_6H_{18}N_3OP$ prepd by dissolving the hydrate in $C_3H_{18}N_3OP$ and heating to 140-145° C for 5 m. The solvate was pptd by addition of abs ether, washing 7 times with ether, and drying over P_2O_5 in a stream of dry nitrogen. Yield was about 90%.

ESTIMATED ERROR:

Soly: precision ± 0.002 mol dm^{-3} at a 95% level of confidence (authors).

Temp: precision ± 3 K.

REFERENCES:

1. Taylor, M.D.; Carter, C.P. *J. Inorg. Nucl. Chem.* 1962, *24*, 387.
2. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lanthanum chloride; $LaCl_3$; [10099-58-8] (2) Tetrachlorostannate; $SnCl_4$; [7646-78-8] (3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]	Lyubimov, E. I.; Batyaev, I. M. *Zh. Prikl. Khim.* 1972, *45*, 1176-8.
VARIABLES: T/K = 293 Concentration of $SnCl_4$	PREPARED BY: T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	La_2O_3 solubility[a] moles La dm^{-3}
0	0	0.012
1:250	0.035	0.14
1:100	0.085	0.26
1:50	0.17	0.30
1:25	0.33	0.27
1:15	0.59	0.22
1:10	0.78	0.21

[a] This is also the solubility of $LaCl_3$ in the $SnCl_4$-$POCl_3$ mixtures because the oxide is quantitatively converted to the chloride according to

$$La_2O_3 + 6POCl_3 = 2LaCl_3 + 3P_2O_3Cl_4$$

Thus the equilibrated solutions should actually be considered to be a four component system containing $SnCl_4$, $LaCl_3$, $P_2O_3Cl_4$ and $POCl_3$ (the compiler assumes $P_2O_3Cl_4$ is soluble).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and La_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20°C to 2 hours.

La was determined by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

La_2O_3 of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade $SnCl_4$ and $POCl_3$ were dehydrated with P_2O_5 and distilled under vacuum.

ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably ± 0.2K (compiler).

REFERENCES: