COMPONENTS:

(1) Lutetium chloride; $LuCl_3$; [10099-66-8]

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

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

ORIGINAL MEASUREMENTS:

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

Zh. Neorg. Khim. 1976, *21*, 551-4; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1976, *21*, 296-8.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $LuCl_3.6H_2O$ 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	53.30	53.20	53.28	53.26	53.26	2.926
30	54.86	54.70	54.75	54.80	54.78	3.111
40	57.10	56.90	56.95	57.05	57.00	3.404
50	61.60	61.48	61.52	61.45	61.51	4.104
60	68.35	68.20	68.39	68.18	68.28	5.528

[a] It is not clearly stated whether the mixture is 96.8 mass % 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Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$LuCl_3.6H_2O$ 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 analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 148.0 - 150.5°C. 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 ± 0.9% (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Lutetium chloride; $LuCl_3$; [10099-66-8]

(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. Inorg. Chem. (Engl. Transl.)* 1977, *22*, 955-8.

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with the solvate $LuCl_3.3C((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.073 \text{ mol dm}^{-3}$$

[a] Table 3 in the English translation of the source paper states the temperature to be 23 ± 3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25 ± 3°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{1/2}$ = 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 analyzed and found to be $LuCl_3.3C_6H_{18}N_3OP$.

The solvate was analyzed for metal content by complexometric titration, 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 were also carried out by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $LuCl_3$ prepared by modification of Taylor & Carter's method (1) by subliming NH_4Cl from a mixture of $LuCl_3$ with 6 moles of NH_4Cl in a stream of inert gas at 200-400°C. The product contained less than 3% of LuOCl.

The solvent was purified as in (2).

ESTIMATED ERROR:

Soly: precision ± 0.001 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:

(1) Lutetium chloride; $LuCl_3$; [10099-66-8]

(2) Tetrachlorostannate; $SnCl_4$; [7646-78-8]

(3) Phosphorus oxychloride; $POCl_3$; [10025-87-3]

ORIGINAL MEASUREMENTS:

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}	Lu_2O_3 solubility[a,b] moles Lu dm^{-3}
0	0	0.1
1:100	0.085	0.3
1:50	0.17	0.7 (0.1)
1:25	0.33	0.9
1:15	0.59	0.7
1:10	0.78	0.8

[a] Solutions preheated to 220°C. Value in parenthesis corresponds to preheating at 120°C.

[b] This is also the solubility of $LuCl_3$ in the $SnCl_4$-$POCl_3$ mixture because the oxide is quantitatively converted to the chloride according to

$$Lu_2O_3 + 6POCl_3 = 2LuCl_3 + 3P_2O_3Cl_4$$

Authors state the the solubility of $LuCl_3$ is enhanced by complex formation according to

$$2LuCl_3 + 3SnCl_4 = Lu_2(SnCl_6)_3$$

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 Lu_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 220°C lowered the equilibration time at 20°C to 2 hours.

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

SOURCE AND PURITY OF MATERIALS:

Lu_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: