COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(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/°C	solubility[a] mass %	mol kg^{-1}	d/g cm^{-3}	η/P	n_D^{20}	nature of the solid phase
25	0.024	0.0093_5	1.6875	0.0386	1.5552	$SmCl_3.3.5H_2O$
50	0.035	0.00136	1.649	0.0311	1.5557	$SmCl_3.3.5H_2O$
75	0.054	0.00210	1.613	0.0249	1.5548	$SmCl_3.3.5H_2O$

[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 samarium detd gravimetrically by precipitating as the oxalate and igniting to the oxide. Samarium 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.

At 120°C, partial hydrolysis takes place with formation of SmOCl.

SOURCE AND PURITY OF MATERIALS:

$SmCl_3.6H_2O$ prepd by dissolving Sm_2O_3 in HCl, evaporating and cooling, and then recrystallized and dried in a desiccator. Sm_2O_3, 99.6% pure, contained oxide impurities of other rare earth metals, Fe (0.01%), Ca (0.01-0.05%), and Cu (0.01%). Analysis of the hexahydrate gave the following (in mass % units): Sm 41.51; Cl 28.90; H_2O 29.59.

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) Samarium chloride; $SmCl_3$; [10361-82-7]

(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 de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

	mean solubilities/mol kg^{-1}	
t/°C	a	b
25	3.33	3.37

a. Initial salt is the adduct $SmCl_3.4CH_3OH$. Equilibrated solid phase analyzed and found to be $SmCl_3.4CH_3OH$.

b. Solutions equilibrated with anhydrous $SmCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $SmCl_3.4CH_3OH$.

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. Samarium 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 $SmCl_3.4CH_3OH$ confirmed by ^1H NMR and X-ray diffraction.

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

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as described in (3). The adduct $SmCl_3.4CH_3OH$ prepared by dissolving the hydrate in a small excess of o-methylformate followed by distillation and crystallization from methanol.

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).

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.

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, 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) Samarium chloride; $SmCl_3$; [10361-82-7]

(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 de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

	mean solubilities/mol kg^{-1}	
t/°C	a	b
25	1.97	2.03

a. Initial salt is the adduct $SmCl_3.3C_2H_5OH$. Equilibrated solid phase analyzed and found to be $SmCl_3.3C_2H_5OH$.

b. Solutions equilibrated with anhydrous $SmCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $SmCl_3.3C_2H_5OH$.

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. Samarium 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 $SmCl_3.3C_2H_5O$ confirmed by ^1H NMR and X-ray diffraction.

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

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $SmCl_3.3C_2H_6O$ prepared by dissolving the hydrate in a small excess of o-ethylformate followed by distillation and crystallization from ethanol.

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

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.

ESTIMATED ERROR:

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

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

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, 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) Samarium chloride; $SmCl_3$; [10361-82-7]

(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 $SmCl_3.6H_2O$ 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^{-1c}
20	28.97	28.89	29.29	28.87	29.00	1.195
30	29.49	29.66	29.59	29.63	29.59	1.152
40	30.42	30.47	30.61	30.75	30.56	1.206
50	31.93	31.96	31.83	31.88	31.90	1.284
60	34.82	34.58	34.79	34.65	34.73	1.458

[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 were obtained after 3 h of equilibration, and the remaining two data points were 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 145.5 - 146°C.

SOURCE AND PURITY OF MATERIALS:

$SmCl_3.6H_2O$ 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. Found (%) for Sm: 41.38, 41.25 (calcd 41.21). Found (%) for Cl: 29.10, 29.15 (calcd 29.18). 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn determined refractometrically and pycnometrically.

ESTIMATED ERROR:

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

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(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 de Lausanne. 1971,

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubilities/mol kg^{-1}	
t/°C	a	b
25	0.23	0.23

a. Initial salt is the adduct $SmCl_3.3C_3H_7OH$. Equilibrated solid phase analyzed and found to be $SmCl_3.3C_3H_7OH$.

b. Solutions equilibrated with anhydrous $SmCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $SmCl_3.3C_3H_7OH$.

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. Samarium determined by titration with $(NH_4)_3(EDTA)$ 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 $SmCl_3.3C_3H_8O$ 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:

Sm_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $SmCl_3.3C_3H_8O$ 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.

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

ESTIMATED ERROR:

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

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

REFERENCES:

1. Brunisholz, F.; Quinche, J.P.; Kalo, A.M. *Helv. Chim. Acta* 1964, *47*, 14.
2. Flatt, 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) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Alcohols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$SmCl_3$ solubility[a] mass %	mol kg^{-1}	nature of the solid phase
1,2-ethanediol;	$C_2H_6O_2$;	[107-21-1]	32.9	1.91	$SmCl_3.3C_2H_6O_2$
1-propanol;	C_3H_8O;	[71-23-8]	30.5	1.71	$SmCl_3.C_3H_8O$
2-propen-1-ol[b];	C_3H_6O;	[107-18-6]	38.0	2.39	$SmCl_3.C_3H_6O$

[a] Molalities calculated by the compilers.

[b] In the original paper the solvent was specified simply as C_3H_5OH. Upon request, the author kindly identified the solvent as allyl alcohol.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Only the nature of the solid phase was reported. Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

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

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

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

ORIGINAL MEASUREMENTS:

McCarty, C.N.
Master of Science Thesis. The University of Illinois. Urbana, IL, USA. 1933: this work was also cited by

Hopkins, B.S.; Audrieth, L.F.
Trans. Electrochem. Soc. 1934, *66*, 134-42.

VARIABLES:

Temperature: T/K = 273 - 323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Sm_2O_3[a] g/25 cc	$SmCl_3$[b] g/dm^3	$SmCl_3$[b] mol/dm^3
0	0.2873	16.92	0.0659
10	0.5210	30.68	0.1195
20	0.7238	42.62	0.1660
30	1.0236	60.28	0.2348
40	1.2827	75.54	0.2942
50	1.4415	84.89	0.3306

[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 $SmCl_3$ from preparations 1 and 2.

[b] Recalculated by the compilers using 1977 IUPAC recommended atomic weights.

Equilibrated solid phase not analyzed.

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 were sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber bung was 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. Sm 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 of 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.

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

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

ORIGINAL MEASUREMENTS:

McCarty, C.N.
Master of Science Thesis. The University of Illinois. Urbana, IL, USA. 1933: this work is also cited by

Hopkins, B.S.; Audrieth, L.F.
Trans. Electrochem. Soc. 1934, *66*, 135-42.

VARIABLES:

Temperature: T/K = 273 - 323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Sm_2O_3[a] g/25 cc	$SmCl_3$[b] g/dm^3	$SmCl_3$[b] mol/dm^3
0	0.1556	9.16	0.0357
10	0.3307	19.47	0.0758
20	0.5666	33.37	0.1300
30	0.7720	45.46	0.1771
40	0.9873	58.14	0.2264
50	1.3540	79.74	0.3106

[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 $SmCl_3$ from preparations 1 and 2.

[b] Recalculated by the compilers using 1977 IUPAC recommended atomic weights.

Equilibrated solid phase not analyzed.

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 were sealed from the atmosphere by placing gum rubber tubing over the stoppers and necks of the bottles, and a rubber bung was 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. 134°C. Sm 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. Extracting 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.

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Alkoxy-ethanols

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	solubility[a] mol kg^{-1}	nature of the solid phase
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	5.7	0.24	$SmCl_3 \cdot nC_3H_8O_2$ (n=2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	26.3	1.39	$SmCl_3 \cdot 2C_4H_{10}O_2$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the authors the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

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

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Diethyl ether (ethyl ether); $C_4H_{10}O$; [60-29-7]

ORIGINAL MEASUREMENTS:

Dzhuraev, Kh. Sh.; Mirsaidov, U.; Kurbanbekov, A.; Rakhimova, A.

Dokl. Akad. Nauk Tadzh. SSR 1976, *19*, 32-4.

VARIABLES:

T/K = 293

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $SmCl_3$ in diethyl ether at 20°C was reported to be

$$7.4 \times 10^{-3} \text{ mass \%.}$$

The corresponding molality calculated by the compiler is

$$2.84 \times 10^{-4} \text{ mol kg}^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrum was attained within 24 h and it was verified by constancy in the Sm concentration. The saturated solution and the equilibrated solid phase were analyzed. Sm determined by complexometric titration using urotropine buffer and methyl-thymol blue indicator. Chloride determined by titration with $AgNO_3$. The solid phase corresponded to $SmCl_3 \cdot 0.5Et_2O$ (the etherate was dried under vacuum at 40°C prior to analysis).

SOURCE AND PURITY OF MATERIALS:

Anhydrous $SmCl_3$ prepared by the ethanol solvate method (no details given).

Ethyl ether was dried with Na and distilled from $LiAlH_4$ before use.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergesaess, U.
Z. Chem. 1968, *8*, 472-3.

Kirmse, E.M.
Tr. II Vses. Konf. po Teor. Rastvorov. 1971, 200-6.

VARIABLES:

Room temperature: T/K around 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$SmCl_3$ solubility[a,b] mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.45	0.018
1,3-dioxolane;	$C_3H_6O_2$;	[646-06-0]	2.6	0.104
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.07	0.0027

[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 solubility was within experimental error limits.

Sm was determined by complexometric titration.

No other details given.

SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared 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) Samarium chloride; $SmCl_3$; [10361-82-7] (2) Tetrahydrofuran; C_4H_8O; [109-99-9]	**ORIGINAL MEASUREMENTS:** Rossmanith, K.; Auer-Welsbach, C. *Monatsh. Chem.* 1965, *96*, 602-5.
VARIABLES: Room temperature: T/K about 293	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $SmCl_3$ in tetrahydrofuran at room temperature (about 20°C) was reported as

1.49 g/100 ml solution

The solid phase is

$SmCl_3 \cdot 1.98C_4H_8O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor for 60-80 hours at room temperature. Samarium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. For the solid phase analysis, the solvent was determined by difference.

Anhydrous substances were handled in a dry box through which was passed a current of dry and CO_2-free nitrogen.

SOURCE AND PURITY OF MATERIALS:

Sources and purities not specified. $SmCl_3$ prepared by reaction of the oxide at high temperatures with an excess of NH_4Cl followed by heating the product in a current of dry nitrogen, and then in vacuum to remove unreacted NH_4Cl.

Tetrahydrofuran was distilled from $LiAlH_4$.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]

ORIGINAL MEASUREMENTS:

Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N.

Zh. Neorg. Khim. 1975, *20*, 908-14; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) 1975, *20*, 508-11.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Composition of saturated solution

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg^{-1} (compiler)	density/g cm^{-3}
33.4	1.30	436.5	1.70	1.95	1.30

The solid phase is $NdCl_3$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd slns prepared isothermally with magnetic stirring. Equil was attained after 25-30 d. The sln was centrifuged and an aliquot for analysis taken and added to methanol and pptd with aq NH_3. The pptd $Sm(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous and only anhydr $SmCl_3$ was found. All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complextion between TBP and $SmCl_3$ in solution. Additional studies with unsaturated solutions (IR spectra, viscosity, molar conductivities) are discussed in the source paper.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $SmCl_3$ prepd by chlorination of Sm_2O_3 with CCl_4 vapor (1,2). Source and purity of materials not given. Sm was analyzed gravimetrically, and Cl by Volhard's method. Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. *Zh. Neorg. Khim.* 1964, *9*, 1427.
2. Novikov, G.I.; Tolmacheva, V.D. *Zh. Prikl. Khim.* 1965, *38*, 1160.

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(2) Amines

ORIGINAL MEASUREMENTS:

Kirmse, E.M.

Tr. II Vses. Konf. po Teor. Rastvorov 1971, 200-6.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			$SmCl_3$ solubility[a] mass %	$SmCl_3$ solubility[a] mol kg^{-1}
2-propanamine;	iso-C_3H_9N;	[75-31-0]	15.2	0.698
2-propen-1-amine[b];	C_3H_7N;	[107-11-9]	5.6	0.225

[a] Molalities calculated by the compilers.

[b] The original paper simply specifies the solvent as $C_3H_5NH_2$, and upon request the author kindly identified the solvent as allylamine.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

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

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(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.* <u>1977</u>, *22*, 1761-6; *Russ. J. Inorg. Chem. Engl. Transl.* <u>1977</u>, *22*, 955-8.

VARIABLES:

Room Temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

At room temperature, the solubility was reported as

$$0.126 \text{ mol dm}^{-3}$$

The solid phase is the solvate $SmCl_3$.3HMPT

It is apparent that the authors determined the solubility of $SmCl_3$ several times, but only the above single value was reported.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Anhydrous $SmCl_3$ placed in a test-tube with the solvent in a dry box and shaken at room temperature. Aliquots removed periodically to test for equilibrium. The analysis was carried out by complexometric titration (no details) and by radioassay using ^{170}Tm. "The results obtained by the two methods agreed." Analysis showed the solid phase to be $SmCl_3$.3HMPT. Analysis of the solid phase performed by complexometric titration for Sm and by the Volhard method for Cl: HMPT was obtained by difference.

SOURCE AND PURITY OF MATERIALS:

$SmCl_3$ prepared similar to (1): a 6:1 molar mixture of NH_4Cl + Sm_2O_3 was heated to 200 to 400°C in a stream of inert gas. Excess NH_4Cl sublimed, and the remaining $SmCl_3$ contained less than 3% oxide impurity.

Hexamethylphosphorotriamide (HMPT) was purified as in (2).

ESTIMATED ERROR:

Soly: ± 0.001 mol dm^{-3} at the 95% confidence level.

Temp: 25 ± 3°C.

REFERENCES:

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

COMPONENTS:

(1) Samarium chloride; $SmCl_3$; [10361-82-7]

(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}	Sm_2O_3 solubility[a] moles Sm dm^{-3}
1:250	0.035	0.10
1:100	0.085	0.18
1:50	0.17	0.28
1:25	0.33	0.25
1:15	0.59	0.11
1:10	0.78	0.10

[a] This is also the solubility of $SmCl_3$ in the $SnCl_4$-$POCl_3$ mixtures because the oxide is quantitatively converted to the chloride according to

$$Sm_2O_3 + 6POCl_3 = 2SmCl_3 + 3P_2O_3Cl_4$$

Thus the equilibrated solutions should actually be considered to be a four component system containing $SnCl_4$, $SmCl_3$, $P_2O_3Cl_4$ 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 Sm_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° to 2 hours.

Sm was determined by colorimetric analysis, and in some cases by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

SOURCE AND PURITY OF MATERIALS:

Sm_2O_3 of "the first sort" was heated 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: