COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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.

	solubility[a]					nature of the
t/°C	mass %	mol kg^{-1}	d/g cm^{-3}	η/p	n_D^{20}	solid phase
25	0.029	0.00116	1.679	0.0389	1.5553	$NdCl_3.3.5H_2O$
50	0.037	0.00148	1.648	0.0311	1.5553	"
75	0.055	0.00220	1.612	0.0249	1.5550	$NdCl_3.3H_2O$

[a] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Equilibrium attained after 12 d at 25°C, 10 d at 50°C, and 7 d at 75°C.

Initial salt, liquid phases and solid phases analysed for Nd by the oxalate method or by titration with Trilon B using Xylene Orange indicator, and for chloride by the Volhard method. Presumably water was found by difference. Solid phase compositions confirmed by X-ray analysis.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3.6H_2O$ prepd by dissolving 99.8% Nd_2O_3 in HCl, evaporating and cooling, recrystallizing, and drying in a desiccator. The oxide contained oxide impurities of other rare earths and Fe (0.01%), Ca((0.01-0.05%), and Cu (0.01%). The product was analysed for metal and halide (mass %): Nd 40.25%, Cl 26.30%, H_2O 33.45%.

Purified solvent (method not specified) had the following properties: d_4^{20} = 1.6807 g cm^{-3}, and n_D^{20} = 1.5543.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: accuracy ± 0.1 K (authors).

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Neodymium chloride; $NdCl_3$; [10024-93-8] (2) Methanol; CH_4O; [67-56-1]	Mark Salomon USA ET & DL Ft. Monmouth, NJ, U.S.A. and Tomasz Mioduski Institute of Nuclear Research Warsaw, Poland

CRITICAL EVALUATION:

The solubility of $NdCl_3$ in methanol has been reported in three publications (1-3). West (1) studied the solubility from 283-313 K and reported his results in volume units. The results in (2) for 273-323 K and in (3) for 298.15 K are both in mass units, but there is serious disagreement between these results. For example at 298.2 K Grigorovich (2) reports a solubility of 4.466 mol kg^{-1} (solid phase is $NdCl_3 \cdot 3CH_3OH$) while Merbach et al. (3) report a solubility of 2.75 mol kg^{-1} (solid phase is $NdCl_3 \cdot 4CH_3OH$).

While we cannot directly compare West's solubility result of ~ 2.25 mol dm^{-3} at 298.2 K with those in (2,3), it certainly is more consistent with the result of Merbach et al. This does not necessarily imply that there is some systematic error in the results of Grigorovich, but rather that a metastable phase is involved.

Merbach et al. report two values for the solubility of $NdCl_3$ in methanol at 298.15 K. The first value of 2.75 mol kg^{-1} was obtained when the solution was equilibrated with the tetrasolvate. Starting with the anhydrous salt, Merbach et al. found a solubility of 2.84 mol kg^{-1}, and the difference in these two results (3 %) is much greater than the experimental precision of 0.5 % estimated by the compilers. It would thus appear that the higher solubility result of Merbach et al. represents a solution in "quasi" equilibrium with both the stable $NdCl_3 \cdot 4CH_3OH$ and metastable $NdCl_3 \cdot 3CH_3OH$ solid phases.

At this time we designate both results of Grigorovich (2) and Merbach et al. (3) as *tentative* solubility data. For 298.15 K the tentative solubility in the stable tetrasolvate system is 2.75 mol kg^{-1} (3), and at 273.2 K, 298.2 K and 323.2 K the tentative solubilities for the metastable trisolvate system are 3.349 mol kg^{-1}, 4.466 mol kg^{-1} and 5.133 mol kg^{-1}, respectively (2).

REFERENCES

1. West, D.H. *Masters Thesis*. University of Illinois. Urbana, IL. 1932. Some of West's data have also been published in reference 4 below.

2. Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986.

3. (a) Merbach, A.; Pitteloud, M.N.; Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44.
   (b) Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.

4. Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283 - 313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

$t/^{\circ}C$	g Nd_2O_3 in 10 cc of saturated sln sample 1	sample 2	average[a]	solubility of $NdCl_3$ [a,b] mol dm^{-3}
10	3.5696	3.5737	3.5717	2.1230
20	3.7587	3.7268	3.7428	2.2247
30	3.838	3.8162	3.8271[c]	2.2748
40	3.9727	3.9955	3.9841	2.3681

[a] Calculated by compilers.

[b] Calculated by compilers based on average mass of Nd_2O_3.

[c] Author gives average value of 3.8221 g/10 cc.

The solid phase was not analyzed.

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 stopper 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 to the oxide and weighed.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_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 was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

ORIGINAL MEASUREMENTS:

Grigorovich, Z.I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
t/°C	mass %	mol kg^{-1}
0	45.63	3.349
25	52.81	4.466
50	56.26	5.133

[a] Molalities calculated by the compiler. At 25°C the solid phase is $NdCl_3.3CH_3OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05 %.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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 de Lausanne. 1971.

VARIABLES:

T/K = 298.2

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

t/°C	mean solubilities/mol kg^{-1} a	 b
25	2.75	2.84

a. Initial salt is the adduct $NdCl_3.4CH_3OH$. Equilibrated solid phase analyzed and found to be $NdCl_3.4CH_3OH$.

b. Solutions equilibrated with anhydrous $NdCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $NdCl_3.4CH_3OH$.

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. Neodymium 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 $NdCl_3.4CH_3OH$ 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:

Nd_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 $NdCl_3.4CH_3OH$ prepared by dissolving the hydrate in a small excess of o-methlyformate 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).

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) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

EVALUATOR:

Mark Salomon
USA ET & DL
Ft. Monmouth, NJ, U.S.A.
and
T. Mioduski
Institute of Nuclear Research
Warsaw, Poland

CRITICAL EVALUATION:

The solubility of $NdCl_3$ in ethanol has been reported in three publications (1-3). King (1) studied the solubility from 273-323 K and reported his results in volume units. The results in (2) for 273-323 K and in (3) for for 298.15 K are both in mass units, but there is serious disagreement between these results. For example at 298.2 K Grigorovich (2) reports a solubility of 3.080 mol kg^{-1} (solid phase is $NdCl_3 \cdot 2C_2H_5OH$) while Merbach et al. (3) report a solubility of 1.35 mol kg^{-1} (solid phase is $NdCl_3 \cdot 3C_2H_5OH$).

While we cannot directly compare King's solubility result of 1.2858 mol dm^{-3} at 298 K with those in (2,3), it certainly is more consistent with the result of Merbach et al. This does not necessarily imply that there is some systematic error in the results of Grigorovich, but rather that a metastable phase is involved.

Merbach et al. report two values for the solubility of $NdCl_3$ in ethanol at 298.15 K. The first value of 1.35 mol kg^{-1} was obtained when the solution was equilibrated with the trisolvate. Starting with the anhydrous salt, Merbach et al. found a solubility of 1.52 mol kg^{-1}, and the difference in these two results (11 %) is much greater than the experimental precision of 0.5 % estimated by the compilers. It would thus appear that the higher solubility result of Merbach et al. represents a solution in "quasi" equilibrium with both the stable $NdCl_3 \cdot 3C_2H_5OH$ and $NdCl_3 \cdot 2C_2H_5OH$ solid phases.

At this time we designate both results of Grigorovich (2) and Merbach et al. (3) as *tentative* solubility data. For 298.15 K the tentative solubility in the stable trisolvate system is 1.35 mol kg^{-1}, and at 273.2 K, 298.2 K and 323.2 K, the tentative solubilities for the metastable disolvate system are 2.420 mol kg^{-1}, 3.080 mol kg^{-1} and 2.333 mol kg^{-1}, respectively. At this time we cannot explain the decrease in the solubility at 323.K in the metastable system: either an error exists or the system has reverted to the stable trisolvate system.

REFERENCES

1. King, F.E. *Masters Thesis*. University of Illinois. Urbana, IL. 1932. Some of King's data have also been published in reference 4 below.

2. Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986.

3. (a) Merbach, A.; Pitteloud, M.N.: Jaccard, P. *Helv. Chim. Acta* 1972, 55, 44.
   (b) Pitteloud, M.N. *These*. Faculte des Sciences de l'Universite de Lausanne. 1971.

4. Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

ORIGINAL MEASUREMENTS:

King, F.E.

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

VARIABLES:

T/K = 273 - 323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

t/°C	g Nd_2O_3 in 10 cc satd sln experimental	average[a]	density/g cm^{-3} exptl	av[a]	solubility of $NdCl_3$[a,b] mol dm^{-3}	mol kg^{-1}
0	1.5598	1.5673	1.0102	1.0105	0.9316	0.9219
0	1.5748		1.0108			
10	1.6053	1.6122	1.0370	1.0374	0.9582	0.9237
10	1.6190		1.0378			
15	1.6338	1.6330	1.0502	1.0505	0.9706	0.9240
15	1.6322		1.0507			
20	2.0129	2.0082	1.0613	1.0614	1.1937	1.1246
20	2.0035		1.0615			
25	2.4274	2.4194	1.1183	1.1184	1.4380	1.2858
25	2.4113		1.1185			
30	1.9223	1.9242	1.0524	1.0524	1.1437	1.0868
30	1.9260		1.0524			
40	2.2178	2.2256	1.0832	1.0832	1.3228	1.2213
40	2.2333		1.0831			
40[c]	3.0182	3.0173	1.207	-----	1.7935	1.486
40[c]	3.0164		-----			
50	2.9742	2.9705	1.1740	1.1745	1.7656	1.5033
50	2.9667		1.1750			

[a] Calculated by compilers.

[b] Calculated by compilers using average values for mass Nd_2O_3 and density of satd slns.

[c] These points detd after two points at 50°C detd: i.e. after cooling from 50°C to 40°C. These data points probably represent metastable equilibria.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Salt and alcohol placed in 250 cc stoppered bottle and mechanically agitated in a thermostat for at least 24 h. Bottle sealed by placing rubber tubing over stopper and neck of 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 alcohol, addn of water, and pptn of the rare earth with oxalic acid. The oxalate was filtered and ignited to const weight 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 solvent 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 given*. These samples were dried in vac over P_2O_5, weighed, converted to the oxalate and ignited to the oxide. Two analyses gave 2.59 and 1.65 molecules of crystallization.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepd from spectro-pure Nd_2O_3 by treatment with aq HCl, and evapn to the point of crystn. Crystals dried in atm of dry HCl for 24 h followed by slow heating in dry HCl until the anhydr salt was obtained. The salt was stored in a vac desiccator over P_2O_5. The salt was analyzed for presence of H_2O by gravimetric analysis (oxlalate-oxide method), but results not given: presumably little or no water was found. Ethanol obtained from the stock room (i.e. source and purity unknown) 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 was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-0]

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

ORIGINAL MEASUREMENTS:

Grigorovich, Z.I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

t/°C	solubility[a] mass %	solubility[a] mol kg^{-1}
0	37.75	2.420
25	43.56	3.080
50	36.89	2.333

[a] Molalities calculated by the compiler. At 25°C the solid phase is $NdCl_3.2C_2H_5OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05%.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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 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	1.35	1.52

a. Initial salt is the adduct $NdCl_3.3C_2H_5OH$. Equilibrated solid phase analyzed and found to be $NdCl_3.3C_2H_5OH$.

b. Solutions equilibrated with anhydrous $NdCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $NdCl_3.3C_2H_5OH$.

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. Neodymium 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 $NdCl_3.3C_2H_5O$ confirmed by ^1H NMR and X-ray diffraction.

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

SOURCE AND PURITY OF MATERIALS:

Nd_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $NdCl_3.3C_2H_6O$ 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.

ESTIMATED ERROR:

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

Temp: precision probably at least ± 0.05K 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) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

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

ORIGINAL MEASUREMENTS:

Sakharova, N.N.; Sakharova, Yu.Gl; 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 $NdCl_3.6H_2O$ in 96.8% C_2H_5OH[a]

t/°C	sample 1 g/100 g[b]	sample 2 g/100 g	sample 3 g/100 g	sample 4 g/100 g	mean solubilities g/100 g	mean solubilities mol kg^{-1}[c]
20	35.38	35.54	35.44	35.50	35.47	0.989
30	36.75	36.80	36.87	36.70	36.80	1.026
40	38.92	38.86	39.39	39.03	39.05	1.089
50	42.98	43.19	48.64[d]	42.78	42.89	1.196
60	47.89	47.60	47.68	48.18	47.84	1.334

[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.

[d] This value appears to be a typographical error in the English translation. To obtain the reported mean solubility of 42.89 g/100 g, the correct value for this data point should be 42.64 g/100 g (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 126.9-128.1°C.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3.6H_2O$ 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 Nd:40.30, 40.10 (calcd 40.20). Found (%) for Cl; 29.59, 29.45 (calcd 29.69). 98.8% 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) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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:

T/K = 283 - 333

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of satd sln		density/g cm^{-3}		solubility $NdCl_3$[a,b]	
t/°C	experimental	average[a]	exptl	av[a]	mol dm^{-3}	mol kg^{-1}
10	1.5788	1.5996	1.3123	-----	0.9508	0.7245
10	1.6205		-----			
15	2.2830	2.2824	1.4029	-----	1.3566	0.9670
15	2.2818		-----			
20	2.7850	2.8059	1.4087	1.4087	1.6678	1.1839
20	2.8268		1.4087			
25	2.7790	2.7843	1.4550	1.4555	1.6549	1.1370
25	2.7895		1.4560			
30	2.3184	2.3214[c]	1.3490	1.3490	1.3798	1.0229
30	2.3244		1.3489			
40	2.9200	2.9539[d]	1.4666	1.4666	1.7554	1.1972
40	2.9878		1.4666			
50	3.3283	3.3517	1.5050	1.5060	1.9922	1.3228
50	3.3750		1.5070			
60	3.0925	3.0814	-----	-----	1.8316	-----
60	3.0703		-----			

[a] Calculated by compilers.

[b] Calculated by compilers from average mass Nd_2O_3 and average density of satd sln.

[c] Author gives av value of 2.3218 g Nd_2O_3: this appears to be a typographical error.

[d] Author gives av value of 2.9589 g Nd_2O_3: this appears to be a typographical error.

The solid phase was not analyzed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

$NdCl_3$ 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 turbidity was significant and appeared different leading author to speculate possible reaction between solute and solvent. Results at 50° 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:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_2O_3, and evapn of solvent until crystn. Crystals dehydrated by method of Kremers (2). Salt analyzed 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% (compilers).

Temp: not specified.

REFERENCES:

1. Some data from Racster's Thesis was 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:	EVALUATOR:
(1) Neodymium chloride; $NdCl_3$; [10024-93-8] (2) 1-Propanol; C_3H_8O; [71-23-8]	Mark Salomon USA ET & DL Ft. Monmouth, NJ, U.S.A. and Tomasz Mioduski Institute of Nuclear Research Warsaw, Poland

CRITICAL EVALUATION:

The solubility of $NdCl_3$ in 1-propanol has been reported in three publications (1-3). West (1) reported solubility data over the temperature range of 283-313 K, and his data are in volume units. The results for 273-323 K (2) and 298 K (3) are both in mass units, but there is serious disagreement between these two publications. For example at 298.2 K Grigorovich (2) reports a solubility of 1.587 mol kg^{-1} (solid phase is $NdCl_3 \cdot 2C_3H_7OH$), and Kirmse (3) reports a solubility of 1.295 % (the nature of the solid phase was not specified).

Since the difference in the results of (2,3) is much greater than the experimental precision in either study, a probable explaination is that the results of Grigorovich are for metastable equilibria (see the critical evaluations for the $NdCl_3$ - CH_3OH and $NdCl_3$ - C_2H_5OH systems).

Kirmse's data probably represent the stable system at 298 K, and for which we assign a *tentative* solubility of 1.295 mol kg^{-1} and a solid phase of $NdCl_3 \cdot nC_3H_7OH$ where $n \geq 3$.

Grigorovich's data probably represent metastable equilibria involving the solid phase $NdCl_3 \cdot 2C_3H_7OH$, and for which the *tentative* solubilities at 273.2 K, 298.2 K and 323.2 K are 1.474 mol kg^{-1}, 1.547 mol kg^{-1} and 1.483 mol kg^{-1}, respectively. Again the low solubility at 323.2 K either represents experimental error or (partial) reversion to the stable higher solvate system.

REFERENCES

1. West, D.H. *Thesis*. University of Illinois. Urbana, IL. 1932. Some data from West's thesis were published in graphical form in reference 4 below.
2. Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986.
3. Kirmse, E.M. *Tr. II Vses Konf. po Teor. Rastvorov* 1971, 200.
4. Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283-313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of saturated sln			solubility of $NdCl_3$[b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	1.4392	1.4694	1.4543	0.8644
20	1.8311	1.8353	1.8332	1.0896
30	2.0328	2.0293	2.0311	1.2072
40	2.5593	2.5753	2.5673	1.5260

[a] Calculated by compilers.

[b] Calculated by compilers using average mass of Nd_2O_3.

The solid phase was not analyzed.

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 stopper 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:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_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 wk and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was neg.

ESTIMATED ERROR:

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

Temp: precision ± 0.2 K (author).

REFERENCES:

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

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

ORIGINAL MEASUREMENTS:

Grigorovich, Z.I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

t/°C	solubility[a] mass %	solubility[a] mol kg^{-1}
0	26.98	1.474
25	28.46	1.587
50	27.10	1.483

[a] Molalities calculated by the compiler. At 25°C the solid phase is $NdCl_3.2C_3H_7OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05 %.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, 7, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$;
[10024-93-8]

(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			$NdCl_3$ solubility^{a,b} mass %	mol kg^{-1}
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	16.8	0.806
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	18.3	0.894
1,2-ethanediol;	$C_2H_6O_2$;	[107-21-1]	31.0	1.793
1-propanol;	C_3H_8O;	[71-23-8]	24.5	1.295

[a] Molalities calculated by the compilers.

[b] Nature of the solid phases not specified.

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) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932[1]

VARIABLES:

T/K = 283 - 313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

t/°C	g Nd_2O_3 in 10 cc of saturated sln			solubility of $NdCl_3$[b]
	sample 1	sample 2	average[a]	mol dm^{-3}
10	0.0152	0.0149	0.0151	0.0089_5
20	0.0175	0.0187	0.0181	0.0108
30	0.0300	0.0295	0.0298	0.0177
40	0.0743	0.0710	0.0727	0.0432

[a] Calculated by compilers.

[b] Calculated by compilers using average mass of Nd_2O_3.

The solid phase was not analyzed.

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 stopper 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:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_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 was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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 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	0.04	0.06

a. Initial salt is the adduct $NdCl_3.3C_3H_7OH$. Equilibrated solid phase analyzed and found to be $NdCl_3.3C_3H_7OH$.

b. Solutions equilibrated with anhydrous $NdCl_3$. Equilibrated solid phases not analyzed, but assumed by the compilers to be $NdCl_3.3C_3H_7OH$.

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. Neodymium 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 $NdCl_3.3C_3H_8O$ 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:

Nd_2O_3 of at least 99.9% purity dissolved in HCl to produce the hexahydrate. The salt was dehydrated as in (3). The adduct $NdCl_3.3C_3H_8O$ 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) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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:

T/K = 283 - 333

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 25 cc satd sln		density/g cm^{-3}		soly $NdCl_3$[a,b]	
t/°C	experimental	average[a]	exptl	ave[c]	mol dm^{-3}	mol kg^{-1}
10	0.4982	0.4971	1.2690	1.2737	0.1182	0.0928
10	0.4959		1.2784			
20	2.7237	2.7264	1.3593	1.3595	0.6482	0.4768
20	2.7290		1.3597			
25	2.1932	2.1892	1.3386	1.3395	0.5205	0.3886
25	2.1851		1.3403			
30	1.3407	1.3418	1.3135	1.3141	0.3190	0.2428
30	1.3429		1.3147			
40	2.2771	2.2782	1.330	1.330	0.5417	0.407
40	2.2793		1.329			
50	3.0694	3.0785	1.3727	1.3723	0.7319	0.5334
50	3.0875		1.3718			
60	2.8782	2.8824	-----	-----	0.6853	-----
60	2.8865					

[a] Calculated by compilers.

[b] Based on average mass of Nd_2O_3.

The solid phase was not analyzed.

[c] Recalculated by compilers.

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, then permitted to stand for 15 h after which slns were still turbid. For analyses, duplicate 25 cc aliquots of turbid slns were taken from each bottle and the rare earth pptd as the oxalate. The ppt was filtered, ignited, and weighed as the oxide. Author states the presence of turbidity had a small effect on the overall accuracy of the soly determinations. Densities of satd slns determined by withdrawing samples from the bottles, placing them into a pycnometer, and weighing "as quickly as possible." The pycnometer was calibrated for each temp.

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:

$NdCl_3$ prepd by adding HCl to spectro-pure Nd_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 for 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% (compilers). Error in accuracy due to turbidity is unknown.

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

REFERENCES:

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

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1-Butanol; $C_4H_{10}O$; [71-36-3]

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283 - 313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of saturated sln			Solubility of $NdCl_3$[b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	1.9230	1.9047	1.9139	1.1376
20	2.1378	2.1323	2.1351	1.2691
30	2.2974	2.3028	2.3001	1.3672
40	2.9250	2.8954	2.9102	1.7298

[a] Calculated by compilers

[b] Calculated by compilers using average mass of Nd_2O_3.

The solid phase was not analyzed.

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 stopper 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:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_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 was published in graphical form by Hopkins, B.S.; Quill, L.L. *Proc. Natl. Acad. Sci. U.S.A.* 1933, *19*, 64.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1-Butanol; $C_4H_{10}O$; [71-36-3]

ORIGINAL MEASUREMENTS:

Grigorovich, Z.I.

Zh. Neorg. Khim. 1963, *8*, 986-9.

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

	solubility[a]	
t/°C	mass %	mol kg^{-1}
0	16.01	0.761
25	17.64	0.855
50	25.00	1.330

[a] Molalities calculated by the compiler. At 25°C the solid phase is $2NdCl_3.3C_4H_9OH$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermographically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05%.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) 1-Pentanol (amyl alcohol); $C_5H_{12}O$; [71-41-0]

ORIGINAL MEASUREMENTS:

West, D.H.

Masters Thesis. The University of Illinois. Urbana, IL. 1932.[1]

VARIABLES:

T/K = 283 - 313

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

	g Nd_2O_3 in 10 cc of saturated sln			solubility of $NdCl_3$[a,b]
t/°C	sample 1	sample 2	average[a]	mol dm^{-3}
10	2.5211	2.4910	2.5061	1.4896
20	2.5312	2.5403	2.5358	1.5072
30	2.5545	2.5825	2.5685	1.5267
40	2.8731	2.8873	2.8802	1.7120

[a] Calculated by compilers.

[b] Calculated by compilers from average mass Nd_2O_3.

The solid phase was not analyzed.

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 stopper 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, 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:

$NdCl_3$ prepd by addn of HCl to spectro-pure Nd_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 wk and then distilled: the first and last 15-20 cc were discarded. $CuSO_4$ test for H_2O was neg.

ESTIMATED ERROR:

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

Temp: precision ± 0.2 K (author).

REFERENCES:

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

COMPONENTS: (1) Neodymium chloride; $NdCl_3$; [10024-93-8] (2) 1-Pentanol; $C_5H_{12}O$; [71-41-0]	**ORIGINAL MEASUREMENTS:** Grigorovich, Z.I. *Zh. Neorg. Khim.* 1963, *8*, 986-9.
VARIABLES: Temperature	**PREPARED BY:** T. Mioduski

EXPERIMENTAL VALUES:

t/°C	solubility[a] mass %	solubility[a] mol kg^{-1}
0	15.96	0.758
25	15.30	0.721
50	14.23	0.662

[a] Molalities calculated by the compiler. At 25°C the solid phase is $NdCl_3 \cdot 1.0C_5H_{12}O$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solutions were thermostated and equilibrated for 3 days. Both the saturated solutions and the solid phase were analyzed for $NdCl_3$ (no details were given).

The alcohol adduct was studied thermo=graphically.

SOURCE AND PURITY OF MATERIALS:

$NdCl_3$ prepared by dissolving "experimental" grade oxide in distilled HCl. The resulting chloride was dehydrated by treatment with thionyl chloride (1).

The alcohol was purified and dried by "standard methods."

ESTIMATED ERROR:

Soly: author states accuracy to be about 0.05%.

Temp: nothing specified.

REFERENCES:

1. Freeman, I.H. *J. Inorg. Nucl. Chem.* 1958, *7*, 286.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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.

VARIABLES:

T/K = 273 - 323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Nd_2O_3[a] g/25 cc	$NdCl_3$[b] g/dm^3	$NdCl_3$[b] mol/dm^3
0	0.3470	20.67	0.0825
10	0.5709	34.02	0.1357
20	0.7465	44.48	0.1775
30	0.9132	54.41	0.2171
40	1.1691	69.66	0.2780
50	1.2654	75.39	0.3009

[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 $NdCl_3$ from preparations 1 and 2.

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

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 stopper was fitted into the open 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 wk and then distilled. A middle portion (fraction not specified) was retained and stored in a stoppered flask: b.p. 123°C. Nd 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.

ESTIMATED ERROR:

Soly: precision probably within 3% (compilers).

Temp: precision ± 0.2 K (author).

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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.

VARIABLES:

T/K = 273 - 323

PREPARED BY:

M. Salomon and T. Mioduski

EXPERIMENTAL VALUES:

Composition of Saturated Solutions

t/°C	Nd_2O_3 [a] g/25 cc	$NdCl_3$ [b] g/dm^3	$NdCl_3$ [b] mol/dm^3
0	0.2656	15.82	0.0631
10	0.4642	27.66	0.1104
20	0.6396	37.95	0.1514
30	0.7627	45.44	0.1813
40	0.9410	56.07	0.2237
50	1.1581	69.00	0.2753

[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 $NdCl_3$ from preparations 1 and 2.

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

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 stopper was fitted into the open 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. Nd 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) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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 $NdCl_3$ in diethyl ether at 20°C was reported to be

$$5.8 \times 10^{-3} \text{ mass \%}$$

The corresponding molality calculated by the compiler is

$$2.31 \times 10^{-4} \text{ mol kg}^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrium was attained within 24 h and it was verified by constancy in the Nd concentration. Both the saturated solution and the equilibrated solid phase were analyzed. Nd determined by complexometric titration in the presence of urotropine buffer and methyl-thymol blue indicator. Chloride determined by titration with $AgNO_3$. The solid phase corresponded to $NdCl_3 \cdot 0.3Et_2O$ (the etherate was dried under vacuum at 40°C prior to analysis).

SOURCE AND PURITY OF MATERIALS:

Anhydrous $NdCl_3$ prepared by the ethanol solvate method (no details given).

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

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Ethers

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, 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			solubility[a,b]	
			mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.04	1.6×10^{-3}
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	0.07	2.8×10^{-3}
1,3-dioxolane;	$C_3H_6O_2$;	[646-06-0]	0.8	3.2×10^{-2}
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.1	4.0×10^{-2}

[a] Molalities calculated by the compilers.

[b] Nature of the 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.

Nd 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) Neodymium chloride; $NdCl_3$; [10024-93-8] (2) Alkyl ethers	**ORIGINAL MEASUREMENTS:** Kirmse, E.M.; Dressler, H. *Z. Chem.* 1975, *15*, 239-40.
VARIABLES: Room Temperature (293-298 K)	**PREPARED BY:** T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	mol kg^{-1}
1-methoxyheptane;	$C_8H_{18}O$;	[629-32-3]	0.4[b]	0.016
1-methoxynonane;	$C_{10}H_{22}O$;	[7289-51-2]	0.02[c]	8×10^{-4}

[a] Molalities calculated by the compilers.

[b] Solid phase $NdCl_3$:$C_8H_{18}O$ found to be 1: > 2.

[c] Solid phase not specified.

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}. Nd 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:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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 $NdCl_3$ in tetrahydrofuran at room temperature (about 20°C) was reported as

1.16 g/100 ml solution (0.046 mol dm^{-3}, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor for 60-80 hours at room temperature. Neodymium 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.

Analysis of the solid phase yielded a $NdCl_3$:C_4H_8O ratio of 1:1.99.

SOURCE AND PURITY OF MATERIALS:

Sources and purities not specified. $NdCl_3$ 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:	ORIGINAL MEASUREMENTS:
(1) Neodymium chloride; $NdCl_3$; [10024-93-8] (2) Tributylphosphate; $C_{12}H_{27}O_4P$; [126-73-8]	Korovin, S.S.; Galaktionova, O.V.; Lebedeva, E.N.; Voronskaya, G.N. *Zh. Neorg. Khim.* <u>1975</u>, *20*, 908-14; *Russ. J. Inorg. Chem.* (*Engl. Transl.*) <u>1975</u>, *20*, 508-11.
VARIABLES:	PREPARED BY:
One temperature	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}
31.8	1.27	408.0	1.63	1.86	1.28

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 $Pr(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analysed (no details given) for phosphorous and only anhydr $PrCl_3$ 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 $PrCl_3$ in solution. Additional studies with unsaturated solutions (IR spectra, viscosity, molar conductivities) are discussed in the source paper.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $NdCl_3$ prepd by chlorination of Nd_2O_3 with CCl_4 vapor (1,2). Source and purity of materials not given. Nd 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.* <u>1964</u>, *9*, 1427.
2. Novikov, G.I., Tolmacheva, V.D. *Zh. Prikl Khim.* <u>1965</u>, *38*, 1160.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Alkyl 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			solubility[a] mass %	mol kg^{-1}
1-propanamine;	C_3H_9N;	[107-10-8]	25.7	1.380
2-propanamine;	iso-C_3H_9N;	[75-31-0]	0.1	0.004
2-propen-1-amine[b];	C_3H_7N;	[107-11-9]	0.05	0.002

[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) Neodymium chloride; NdCl$_3$; [10024-93-8]

(2) Hexamethylphosphorotriamide; C$_6$H$_{18}$N$_3$OP; [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

EXPERIMENTAL VALUES:

The solubility of the anhydrous salt at 25 ± 3°C was given as

$$0.119 \pm 0.005 \text{ mol dm}^{-3}$$

Starting with the solvate NdCl$_3$.3((CH$_3$)$_2$N)$_3$PO, the solubility at 25 ± 3°C[a] was given as

$$0.125 \pm 0.001 \text{ mol dm}^{-3}$$

[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 room temperature (25 ± 3°C).

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 equilibrium 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 NdCl$_3$.3C$_6$H$_{18}$N$_3$OP.

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 NdCl$_3$ prepd similarly to that in (1) by subliming NH$_4$Cl from a mixt of NdCl$_3$ and 6 moles of NN$_4$Cl in a stream of inert gas at 200-400°C (NdOCl content less than 3%). The solvent was purified as in (2).

NdCl$_3$.3C$_6$H$_{18}$N$_3$OP prepd by dissolving the hydrate in C$_3$H$_{18}$N$_3$OP 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$_2$O$_5$ in a stream of dry nitrogen. Yield was about 90 %.

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) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Gallium chloride; $GaCl_3$; [13450-90-3]

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

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Zh. Neorg. Khim. 1976, *21*, 2556-7; *Russ. J. Inorg. Chem. (Engl. Transl.)* 1976, *21*, 1406.
Zh. Fiz. Khim. 1979, *53*, 1588-90; *Russ. J. Phys. Chem. (Engl. Transl.)* 1979, *53*, 897-9.

VARIABLES:

Concentration of $GaCl_3$ at 298 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$GaCl_3/NdCl_3$ molar ratio	$GaCl_3$ concentration mmol dm^{-3}	$NdCl_3$ solubility mmol dm^{-3}	equiv conductivity S cm^2 $equiv^{-1}$
pure $POCl_3$	——	0.01	----
1:22.4	1.0	23	4.6
1:17.8	2.3	39	3.9
1:11.9	3.0	36	5.6
1:10.0	4.1	41	5.2
1:7.6	5.5	42	5.1
1:7.5	6.1	46	4.8
1:5.9	7.1	42	5.4
1:6.3	8.1	51	4.6
1:5.6	9.1	51	5.7
1:5.3	10.1	54	5.8

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Anhydrous $GaCl_3$ and $NdCl_3$ were added to $POCl_3$ in an argon filled dry box. The specified amounts of $GaCl_3$ were dissolved first in $POCl_3$. Solubilities were determined at 25°C.

No other information given.

COMMENTS AND/OR ADDITIONAL DATA:

The authors state that the increase in the solubility of $NdCl_3$ upon increasing $GaCl_3$ concentration is due to complex formation.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $NdCl_3$ and $GaCl_3$ were prepared as described in (1).

$POCl_3$ was dehydrated with P_2O_5 and distilled twice under vacuum.

Solutions were prepared in an argon filled dry box.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Puzankova, N.L.; Slastenova, N.M.; Solov'ev, M.A.; Batyaev, I.M. *Sintez i Issledovanie Nizkotemperaturnykh Khloridnykh Matrits*. XXVI Herzen Lecture. Scientific Papers. 1973.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(2) Gallium chloride; $GaCl_3$; [13450-89-0]

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

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Zh. Fiz. Khim. 1979, *53*, 1588-90; *Russ. J. Phys. Chem. (Engl. Transl.)* 1979, *53*, 897-9. *Zh. Neorg. Khim.* 1976, *21*, 2556-7; *Russ. J. Inorg. Chem. (Engl. Transl)* 1976, *21*, 1406.

VARIABLES:

Concentration of $GaCl_3$

Temp not specified, but probably 298 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$GaCl_3$ concentration mol dm^{-3}	$NdCl_3$ solubility mol dm^{-3}
0	0.01
1.0	22
2.3	39
3.0	36
4.1	41
5.5	42
6.1	46
7.1	42
9.1	51
9.1	51
10.1	54

Authors state that the increase in the solubility of $NdCl_3$ with increasing concentration is due to the formation of $NdCl_3(POCl_3)_x(GaCl_3)_y$ complexes. Stepwise formation constants for these complexes are given below.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The $NdCl_3$-$POCl_3$-$GaCl_3$ systems were synthesized in sealed tubes by the solvolthermal method at 120°C as described in (1). No other details given. The compilers assume that after treating the solutions at 120°C for about 2 h, they were then isothermally equilibrated at 25°C (see the compilations of references 1 and 2 in this volume.)

COMMENTS AND/OR ADDITIONAL DATA:

For the equilibria

$$NdCl_3 + yGaCl_3 = NdCl_3.yGaCl_3$$

the following stepwise formation constants constants, βy, were given:

$$\beta_1 = (2.7 \pm 0.1) \times 10^6$$

$$\beta_2 = (3.6 \pm 0.2) \times 10^9$$

$$\beta_3 = (3.8 \pm 0.2) \times 10^8$$

$$\beta_4 = (3.6 \pm 0.2) \times 10^{14}$$

SOURCE AND PURITY OF MATERIALS:

Anhydrous metal chlorides were prepared as described in (3).

$POCl_3$ was purified by the usual method.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, *45*, 1176.
2. Batyaev, I.M.; Solov'ev, M.A. *Izv. Akad. Nauk SSSR, Neorg. Mater.* 1977, *13*, 104.
3. Slastenova, N.M.; Batyaev, I.M.; Bel'kova, N.L.; Kuz'menko, A.S.; Ryabov, E.N. *Zh. Prikl. Khim.* 1975, *48*, 1953.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

(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:

$SnCl_4$ concentration

T/K = 293 and 333

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4:POCl_3$ ratio	$SnCl_4$ concn	solubility of Nd_2O_3/mol dm^{-3} [a]		
(by volume)	mol dm^{-3}	20°C [b]	20°C [c]	60°C [d]
0	0	0.007	-----	0.003
1:250	0.035	0.11	0.093	0.092
1:100	0.085	0.27	0.23	0.20
1:50	0.17	0.30	0.30	0.26
1:25	0.33	0.26	0.20	0.31
1:15	0.59	0.15	0.13	0.22
1:10	0.78	0.11	0.10	0.22
1:5	1.4	-----	-----	0.20
1:1.5	3.0	-----	-----	0.19
1:1	4.6	-----	-----	0.090
4:1	6.8	-----	0.010	0.010
pure $SnCl_4$	8.5	2×10^{-4}	-----	-----

[a] This is also the solubility of $NdCl_3$ since the oxide is quantitatively converted to the chloride according to

$$Nd_2O_3 + 6POCl_3 = 2NdCl_3 + 3P_2O_3Cl_4$$

Assuming $P_2O_3Cl_4$ to be soluble, the equilibrated solutions would then constitute a four component mixture.

[b] Preheated at 120°C for 2 hours.

[d] No pretreatment.

[c] Preheated at 60°C (time not specified).

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 Nd_2O_3 were placed in sealed ampoules, heated to 120°C for 2 hours to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2 hours. Without preheating equilibrium was established after 200 hours. Preheating to 120°C lowered the equilibration time at 20°C to 2 hours.

Nd 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.

The solubility of $NdCl_3$ in pure $POCl_3$ is small, but in the presence of $SnCl_4$ the solubility increases due to complexation:

$$2NdCl_3 + 3SnCl_4 = Nd_2(SnCl_6)_3$$

SOURCE AND PURITY OF MATERIALS:

Nd_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:

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

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

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Izv. Akad. Nauk SSSR, Neorg. Mater. 1977, *13*, 104-8.

VARIABLES:

$SnCl_4$ concentration

T/K = 293

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$SnCl_4/POCl_3$ volume ratio	$SnCl_4$ concn 10^2c_2/mol dm^{-3}	$SnCl_4/NdCl_3$ mol ratio	solubility of $NdCl_3$ 10^3c_1/mol dm^{-3}
0.25:29.7	5.0	10:1	5.0
0.50:29.5	10.0	19.2:1	5.2
1.0:29.0	15.0	27.7:1	5.4
1.25:28.7	20.0	35.7:1	5.6
1.50:28.5	25.0	43.9:1	5.7
1.75:28.2	30.0	50.91:1	5.9
2.0:28.0	35.0	57.4:1	6.1
2.25:27.7	40.0	63.5:1	6.3
2.50:27.5	45.0	69.2:1	6.5
2.75:27.2	50.0	74.6:1	6.7
3.0:27.0	55.0	79.7:1	6.9

The solubility of $NdCl_3$ in pure $POCl_3$ was reported to be 10^{-5} mol dm^{-3} (solubility product = 2.7 x 10^{-19} mol^4 dm^{-12}). The increasing solubility of $NdCl_3$ observed upon increasing the $SnCl_4$ concentration from 0.05-0.55 mol dm^{-3} is attributed to outer sphere coordination of $SnCl_4$ by $POCl_3$. Stability constants, β_i, for the reactions

$$NdCl_3 + nPOCl_3 = NdCl_3.nPOCl_3$$

were calculated for i = 1-4.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Solvent-solute mixtures were sealed in glass ampoules and rotated in an air thermostat at 120°C for 24 h. The ampoules were then rotated in a thermostat at 20°C for 24 h. After equilibrium was reached, aliquots were withdrawn in a dry argon atmosphere and evaporated in vacuum. The dry residues were hydrolyzed, the hydrolyzed products filtered, and Nd determined spectrophotometrically. The solutions were analyzed at pH 3 (phormic buffer) using 0.05% arsenazo III solution. A calibration curve was used for these analyses.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

COMMENTS AND/OR ADDITIONAL DATA:

Conflicting data exist between the source paper and earlier work (1). In the source paper the solubility of $NdCl_3$ in pure $POCl_3$ is given as 10^{-5} mol dm^{-3} compared to 7 x 10^{-3} mol dm^{-3} reported in (1). In (1), the enhancement of the solubility of $NdCl_3$ is attributed to the formation of $Nd_2(SnCl_6)_3$, and in the present work the enhancement is attributed to coordination of $SnCl_4$ by $POCl_3$.

REFERENCES:

1. Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, *45*, 1176. (see previous page for the compilation of this paper).

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$; [10024-93-8]

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

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

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Zh. Fiz. Khim. 1979, *53*, 1588-90;
Russ. J. Phys. Chem. (Engl. Transl.) 1979, *53*, 897-9.

VARIABLES:

Concentration of $SnCl_4$

Temp not specified, but probably 298 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$SnCl_4$ concentration mol dm^{-3}	$NdCl_3$ solubility mol dm^{-3}
0	0.01
50	5.0
100	5.2
150	5.4
200	5.6
250	5.7
300	5.9
350	6.1
400	6.3
450	6.5
500	6.7

Authors state that the increase in the solubility of $NdCl_3$ with increasing concentration is due to the formation of $NdCl_3(POCl_3)_x(SnCl_4)_y$ complexes. Stepwise formation constants for these complexes are given below.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The $NdCl_3$-$POCl_3$-$SnCl_4$ systems were synthesized in sealed tubes by the solvolthermal method at 120°C as described in (1). No other details given. The compilers assume that after treating the solutions at 120°C for about 2 h, they were then isothermally equilibrated at 25°C as in (2): see the compilations of references 1 and 2 in this volume

COMMENTS AND/OR ADDITIONAL DATA:

For the equilibria

$$NdCl_3 + ySnCl_4 = NdCl_3.ySnCl_4$$

the following stepwise formation constants constants, βy, were given:

$$\beta_1 = (1.5 \pm 0.1) \times 10^4$$
$$\beta_2 = (3.8 \pm 0.2) \times 10^6$$
$$\beta_3 = (3.6 \pm 0.2) \times 10^5$$
$$\beta_4 = (1.2 \pm 0.1) \times 10^8$$

SOURCE AND PURITY OF MATERIALS:

Anhydrous metal chlorides were prepared as described in (3).

$POCl_3$ and $SnCl_4$ were purified by the usual methods.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, *45*, 1176.
2. Batyaev, I.M.; Solov'ev, M.A. *Zh. Neorg. Khim.* 1976, *21*, 2556.
3. Slastenova, N.M.; Batyaev, I.M.; Bel'kova, N.L.; Kuz'menko, A.S.; Ryabov, E.N. *Zh. Prikl. Khim.* 1975, *48*, 1953.

COMPONENTS:

(1) Neodymium chloride; $NdCl_3$;
[10024-93-8]

(2) Zinc chloride; $ZnCl_2$;
[7846-85-7]

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

ORIGINAL MEASUREMENTS:

Batyaev, I.M.; Solov'ev, M.A.

Zh. Fiz. Khim. 1979, 53, 1588-90;
Russ. J. Phys. Chem. (Engl. Transl.) 1979, 53, 897-9.

VARIABLES:

Concentration of $ZnCl_2$

Temp not specified, but probably 298 K

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

$ZnCl_2$ concentration mol dm^{-3}	$NdCl_3$ solubility mol dm^{-3}
0	0.01
17	2.0
16	1.9
14	1.8
13	1.7
12	1.6
12	1.5
11	1.4
10	1.3
9	1.2
9	1.1

Authors state that the increase in the solubility of $NdCl_3$ with increasing concentration is due to the formation of $NdCl_3(POCl_3)_x(ZnCl_2)_y$ complexes. Stepwise formation constants for these complexes are given below.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The $NdCl_3$-$POCl_3$-$ZnCl_2$ systems were synthesized in sealed tubes by the solvothermal method at 120°C as described in (1). No other details given. The compilers assume that after treating the solutions at 120°C for about 2 h, they were then isothermally equilibrated at 25°C as in (2): see the compilations of references 1 and 2 in this volume.

COMMENTS and/OR ADDITIONAL DATA:

For the equilibria

$$NdCl_3 + yZnCl_2 = NdCl_3 \cdot yZnCl_2$$

the following stepwise formation constants constants, β_y, were given:

$$\beta_1 = (6.7 \pm 0.3) \times 10^4$$
$$\beta_2 = (2.5 \pm 0.1) \times 10^5$$
$$\beta_3 = (7.4 \pm 0.3) \times 10^7$$
$$\beta_4 = (5.4 \pm 0.2) \times 10^9$$

SOURCE AND PURITY OF MATERIALS:

Anhydrous metal chlorides were prepared as described in (3).

$POCl_3$ was purified by the usual method.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Lyubimov, E.I.; Batyaev, I.M. *Zh. Prikl. Khim.* 1972, 45, 1176.
2. Batyaev, I.M.; Solov'ev, M.A. *Zh. Neorg. Khim.* 1976, 21, 2556.
3. Slastenova, N.M.; Batyaev, I.M.; Bel'kova, N.L.; Kuz'menko, A.S.; Ryabov, E.N. *Zh. Prikl. Khim.* 1975, 48, 1953.