COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

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

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

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Temperature

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solubility of $DyCl_3.6H_2O$ 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	33.46	33.38	33.64	33.24	33.43	1.332
30	32.60	32.40	32.27	32.26	32.38	1.270
40	31.91	31.98	32.36	32.12	32.09	1.254
50	33.67	33.39	33.58	33.10	33.43	1.332
60	35.04	34.66	34.87	35.10	34.91	1.423

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

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

[c] Molalities calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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

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

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:

$DyCl_3.6H_2O$ prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over $CaCl_2$, P_2O_5 and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 161.8 - 163.1°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr $CuSO_4$ followed by distn. Ethanol concn detd refractometrically and pycnometrically.

ESTIMATED ERROR:

Soly: results apparently precise to within ± 0.9 % (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(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			$DyCl_3$ solubility[a] mass %	mol kg^{-1}	nature of the solid phase
2-methoxyethanol;	$C_3H_8O_2$;	[109-86-4]	3.9	0.151	$DyCl_3.nC_3H_8O_2$ (n = 2-3)
2-ethoxyethanol;	$C_4H_{10}O_2$;	[110-80-5]	9.4	0.386	$DyCl_3.2C_4H_{10}O_2$

[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 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) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(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 $DyCl_3$ in diethyl ether at 20°C was reported to be

$$3.2 \times 10^{-2} \text{ mass } \%$$

The corresponding molality calculated by the compiler is

$$1.19 \times 10^{-3} \text{ mol kg}^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. Equilibrium was attained within 24 h and it was verified by constancy in the Dy concentration. The saturated solution and the equilibrated solid phase were analyzed. Dy determined by complexometric titration using urotropine buffuer and methyl-thymol blue indicator. Chloride determined by titration with $AgNO_3$ solution. The solid phase corresponded to $DyCl_3.Et_2O$ (the etherate was dried under vacuum at 40°C prior to analysis).

SOURCE AND PURITY OF MATERIALS:

Anhydrous $DyCl_3$ 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) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(2) 1,2-Diethoxyethane; $C_6H_{14}O_2$; [629-14-1]

ORIGINAL MEASUREMENTS:

Kirmse, E.M.; Zwietasch, K.J.

Z. Chem. 1967, 7, 281.

VARIABLES:

T/K = 298

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

The solubility of $DyCl_3$ in 1,2-diethoxyethane at 25°C was reported to be

0.25 mass %

The corresponding molality calculated by the compiler is

$0.0093 \text{ mol kg}^{-1}$

The composition of the solid phase was given in terms of the Dy:Cl:ether ratio as

1:2.91:1.70

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over P_2O_5.

Dy was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of C_2H_5I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:

No estimates possible.

REFERENCES:

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

COMPONENTS: (1) Dysprosium chloride; $DyCl_3$; [10025-74-8] (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			$DyCl_3$ solubility^{a,b} mass %	mol kg^{-1}
1-ethoxy-2-methoxyethane;	$C_5H_{12}O_2$;	[5137-45-1]	0.6	0.022
1-methoxypentane;	$C_6H_{14}O$;	[628-80-8]	0.08	0.003
1,4-dioxane;	$C_4H_8O_2$;	[123-91-1]	0.4	0.015

[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.

Dy 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) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(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 $DyCl_3$ in tetrahydrofuran at 20°C (room temperature) was reported to be

0.645 g per 100 ml of solution

(0.00240 mol dm^{-3}, compiler)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

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

Dysprosium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is $DyCl_3.3.45C_4H_8O$.

SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. $DyCl_3$ was prepared by conversion of the oxide by high temperature reaction with an excess of NH_4Cl followed by heating the product in a stream 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) Dysprosium chloride; $DyCl_3$; [10025-74-8]

(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 solutions

mass %	mol/kg sln	g dm^{-3}	mol dm^{-3}	mol kg^{-1} (compiler)	density/g cm^{-3}
39.2	1.45	534.4	1.98	2.40	1.36

The solid phase is $DyCl_3$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and precipitated with aq NH_3. The pptd $Dy(OH)_3$ was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous and only the anhydrous $DyCl_3$ 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 complexation between TBP and $DyCl_3$ in solution.

SOURCE AND PURITY OF MATERIALS:

Anhydrous $DyCl_3$ prepared by chlorination of the oxide with CCl_4 vapor (1,2). Source and purity of materials not given. Dy 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:	ORIGINAL MEASUREMENTS:
(1) Dysprosium chloride; $DyCl_3$; [10025-74-8] (2) Amines	Kirmse, E.M. *Tr. II Vses. Konf. po Teor. Rastvorov* 1971, 200-6.
VARIABLES:	**PREPARED BY:**
T/K = 298	T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

solvent			solubility[a] mass %	mol kg^{-1}
2-propanamine;	iso-C_3H_9N;	[75-31-0]	24.5	1.207
2-propen-1-amine;[b]	C_3H_7N;	[107-11-9]	18.0	0.816
1-butanamine;	n-$C_4H_{11}N$;	[109-73-9]	21.2	1.001
2-butanamine;	sec-$C_4H_{11}N$;	[13952-84-6]	15.4	0.677

[a] Molalities calculated by the compilers.

[b] The original paper 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) Dysprosium chloride; $DyCl_3$; [10025-74-8]

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

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Room temperature: T/K = 298 ± 3

PREPARED BY:

T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

Starting with the solvate $DyCl_3.3((CH_3)_2N)_3PO$, the solubility at 25 ± 3°C[a] was given as

$$0.109 \text{ mol dm}^{-3}$$

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

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 ($t_{½}$ = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be $DyCl_3 \cdot 3C_6H_{18}N_3OP$.

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

SOURCE AND PURITY OF MATERIALS:

$DyCl_3.3C_6H_{18}N_3OP$ prepared by dissolving the hydrate in the solvent and heating to 140-145°C for 5 m. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over P_2O_5 in a stream of dry nitrogen. Yield was about 90%.

The solvent was purified as described in (1).

ESTIMATED ERROR:

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

Temp: precision ± 3 K.

REFERENCES:

1. Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. *Elektrokhimiya* 1975, *11*, 163.

COMPONENTS:

(1) Dysprosium chloride; $DyCl_3$; [10025-74-8]

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

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

ORIGINAL MEASUREMENTS:

Lyubimov. E.I.; Batyaev, I.M.

Zh. Prikl. Khim. 1972, *45*, 1176-8.

VARIABLES:

T/K = 293

Concentration of $SnCl_4$

PREPARED BY:

T. Mioduski

EXPERIMENTAL VALUES:

$SnCl_4$:$POCl_3$ ratio (by volume)	$SnCl_4$ concentration mol dm^{-3}	Dy_2O_3 solubility[a,b] moles Dy dm^{-3}
1:100	0.085	0.022
1:50	0.17	0.023 (0.018)
1:25	0.33	0.034
1:15	0.59	0.023
1:10	0.78	0.032

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

[b] This is also the solubility of $DyCl_3$ in the $SnCl_4$-$POCl_3$ mixture because the oxide is quantitatively converted to the chloride according to

$$Dy_2O_3 + 6POCl_3 = 2DyCl_3 + 3P_2O_3Cl_4$$

Authors state that the solubility of $DyCl_3$ is enhanced by complex formation according to

$$2DyCl_3 + 3SnCl_4 = Dy_2(SnCl_6)_3$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Isothermal method used. $POCl_3$ + $SnCl_4$ solutions were prepared by volume in a dry box. The $SnCl_4$ content was verified by chemical analysis for Sn. This solution and Dy_2O_3 were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution and then rotated in an air thermostat at 20° C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Preheating to 220°C lowered the equilibration time at 20°C to 2 hours.

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

SOURCE AND PURITY OF MATERIALS:

Dy_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 ± 0.2 K (compiler).

REFERENCES: