COMPONENTS:
(1) Holmium chloride; HoCl₃; [10138-62-2]
(2) Ethanol; C₂H₆O; [64-17-5]
(3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:
Sakharova, Yu.G; Ezhova, T.A.

VARIABLES:
Temperature

PREPARED BY:
T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

<table>
<thead>
<tr>
<th>t/°C</th>
<th>sample 1</th>
<th>sample 2</th>
<th>sample 3</th>
<th>sample 4</th>
<th>mean solubilities</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>g/100 g</td>
<td>g/100 g</td>
<td>g/100 g</td>
<td>g/100 g</td>
<td>g/100 g</td>
</tr>
<tr>
<td>20</td>
<td>34.62</td>
<td>34.81</td>
<td>34.86</td>
<td>34.87</td>
<td>34.79</td>
</tr>
<tr>
<td>30</td>
<td>33.72</td>
<td>33.85</td>
<td>34.04</td>
<td>33.89</td>
<td>33.87</td>
</tr>
<tr>
<td>40</td>
<td>33.88</td>
<td>34.02</td>
<td>33.83</td>
<td>34.16</td>
<td>33.97</td>
</tr>
<tr>
<td>50</td>
<td>34.25</td>
<td>34.68</td>
<td>34.47</td>
<td>34.56</td>
<td>34.49</td>
</tr>
<tr>
<td>60</td>
<td>36.02</td>
<td>36.11</td>
<td>35.77</td>
<td>35.83</td>
<td>35.93</td>
</tr>
</tbody>
</table>

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

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

c Molalities calculated by the compilers.

AUXILIARY INFORMATION

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

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

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS:
HoCl₃.6H₂O prep'd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over CaCl₂, P₂O₅ and NaOH. The crystals analyzed for the metal by titm with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 163.4 - 164.5°C. 96.8% ethanol prep'd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr CuSO₄ 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. Holmium chloride; \(\text{HoCl}_3\);
   [10138-62-2]
2. Alkoxy-ethanols

**ORIGINAL MEASUREMENTS:**

Kirmse, E.M.


**VARIABLES:**

\(T/K = 298\)

**PREPARED BY:**

T. Mioduski and M. Salomon

**EXPERIMENTAL VALUES:**

<table>
<thead>
<tr>
<th>Solvent</th>
<th>(\text{mol kg}^{-1})</th>
<th>Nature of the solid phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-methoxyethanol; (\text{C}_3\text{H}_8\text{O}_2); [109-86-4]</td>
<td>3.2, 0.122</td>
<td>(\text{HoCl}_3\cdot n\text{C}_3\text{H}_8\text{O}_2) ((n = 2-3))</td>
</tr>
<tr>
<td>2-ethoxyethanol; (\text{C}<em>6\text{H}</em>{10}\text{O}_2); [110-80-5]</td>
<td>8.4, 0.338</td>
<td>(\text{HoCl}_3\cdot 2\text{C}<em>6\text{H}</em>{10}\text{O}_2)</td>
</tr>
</tbody>
</table>

\(^{a}\)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.
COMPONENTS:
(1) Holmium chloride; HoCl₃; [10138-62-2]
(2) 1,2-Diethoxyethane; C₆H₁₄O₂; [629-14-1]

VARIABLES:
T/K = 298

EXPERIMENTAL VALUES:
The solubility of HoCl₃ in 1,2-diethoxyethane at 25°C was reported to be 0.37 mass %
The corresponding molality calculated by the compiler is 0.0137 mol kg⁻¹
The composition of the solid phase was given in terms of the Ho:Cl:ether ratio as 1:2.97:1.82

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 P₂O₅.
Ho 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 C₂H₅I with the monoethylether of ethylene glycol.

ESTIMATED ERROR:
No estimates possible.

REFERENCES:
COMPONENTS:
(1) Holmium chloride; HoCl$_3$; [10138-62-2]
(2) Alkyl ethers

VARIABLES:
Room temperature: T/K = 293-298

EXPERIMENTAL VALUES:

<table>
<thead>
<tr>
<th>Solvent</th>
<th>HoCl$_3$ solubility$^a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-methoxypentane; $^b$</td>
<td>C$<em>6$H$</em>{14}$O; [628-80-8]</td>
</tr>
<tr>
<td>1-methoxyheptane;</td>
<td>C$<em>6$H$</em>{18}$O; [629-32-3]</td>
</tr>
<tr>
<td>1-methoxyoctane;</td>
<td>C$<em>9$H$</em>{20}$O; [929-56-6]</td>
</tr>
<tr>
<td>1-methoxynonane;</td>
<td>C$<em>{10}$H$</em>{22}$O; [7289-51-2]</td>
</tr>
<tr>
<td>1-methoxydecane;</td>
<td>C$<em>{11}$H$</em>{24}$O; [7289-52-3]</td>
</tr>
</tbody>
</table>

$^a$Molarities calculated by the compilers.

$^b$Identical result for this system reported in (1)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:
The solute-solvent mixtures were agitated at room temperature until the solutions were saturated. The anhydrous reagents were handled in a dry box containing P$_2$O$_5$.

Holmium was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations for each system.

SOURCE AND PURITY OF MATERIALS:
No information given.

ESTIMATED ERROR:
No information given.

REFERENCES:
**COMPONENTS:**

1. Holmium chloride; HoCl₃; [10138-62-2]
2. Ethers

**ORIGINAL MEASUREMENTS:**

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


**VARIABLES:**

Room temperature: T/K around 298

**EXPERIMENTAL VALUES:**

<table>
<thead>
<tr>
<th>solvent</th>
<th>mass %</th>
<th>mol kg⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-ethoxy-2-methoxyethane; C₅H₁₂O₂; [5137-45-1]</td>
<td>0.65</td>
<td>0.024</td>
</tr>
<tr>
<td>di-n-propyl ether; C₆H₁₄O; [111-43-3]</td>
<td>0.1</td>
<td>0.004</td>
</tr>
<tr>
<td>1-ethoxybutane; C₆H₁₄O; [628-81-9]</td>
<td>0.01</td>
<td>0.0004</td>
</tr>
<tr>
<td>1-methoxypentane; C₆H₁₄O; [628-80-8]</td>
<td>2.4</td>
<td>0.091</td>
</tr>
<tr>
<td>1,4-dioxane; C₄H₈O₂; [123-91-1]</td>
<td>0.55</td>
<td>0.020</td>
</tr>
</tbody>
</table>

aMolalities calculated by the compilers.

bNature 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.

Ho 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:**

**COMPONENTS:**

1. Holmium chloride; HoCl₃; [10138-62-2]
2. Tetrahydrofuran; C₄H₆O; [109-99-9]

**ORIGINAL MEASUREMENTS:**


**VARIABLES:**

Room Temperature: T/K about 293

**PREPARED BY:**

T. Mioduski

**EXPERIMENTAL VALUES:**

The solubility of HoCl₃ in tetrahydrofuran at 20°C (room temperature) was reported to be

\[
\begin{align*}
0.698 \text{ g per 100 ml of solution} \\
(0.0257 \text{ mol dm}^{-3}, \text{ compiler}).
\end{align*}
\]

**AUXILIARY INFORMATION**

**METHOD/APPARATUS/PROCEDURE:**

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

Holmium 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 HoCl₃·3.33C₄H₆O.

**SOURCE AND PURITY OF MATERIALS:**

Sources and purities of initial materials not specified. HoCl₃ was prepared by conversion of the oxide by high temperature reaction with an excess of NH₄Cl followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH₄Cl.

Tetrahydrofuran was distilled from LiAlH₄.

**ESTIMATED ERROR:**

Nothing specified.

**REFERENCES:**
COMPONENTS:
(1) Holmium chloride; HoCl₃; [10138-62-2]
(2) Tributylphosphate; C₁₂H₂₇O₄P; [126-73-8]

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

VARIABLES:
T/K = 298

EXPERIMENTAL VALUES:

<table>
<thead>
<tr>
<th>Component</th>
<th>Mass %</th>
<th>mol/kg sln</th>
<th>g dm⁻³</th>
<th>mol dm⁻³</th>
<th>mol kg⁻¹</th>
<th>Density/g cm⁻³</th>
</tr>
</thead>
<tbody>
<tr>
<td>HoCl₃</td>
<td>41.0</td>
<td>1.52</td>
<td>573.1</td>
<td>2.12</td>
<td>2.56</td>
<td>1.39</td>
</tr>
</tbody>
</table>

The solid phase is HoCl₃

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 NH₃. The pptd Ho(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 HoCl₃ 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 HoCl₃ in solution.

SOURCE AND PURITY OF MATERIALS:
Anhydrous HoCl₃ prepared by chlorination of the oxide with CCl₄ vapor (1,2). Source and purity of materials not given. Ho was analyzed gravimetrically, and Cl by Volhard's method.

Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

ESTIMATED ERROR:
No estimate possible.

REFERENCES:
1. Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N.
2. Novikov, G.I.; Tolmacheva, V.D.
   Zh. Prikl. Khim. 1965, 38, 1160
COMPONENTS:
(1) Holmium chloride; HoCl₃; [10138-62-2]
(2) Alkyl amines

ORIGINAL MEASUREMENTS:
Kirmse, E.M.

VARIABLES:
T/K = 298

PREPARED BY:
T. Mioduski and M. Salomon

EXPERIMENTAL VALUES:

<table>
<thead>
<tr>
<th>Solvent</th>
<th>HoCl₃ solubility$^a$</th>
<th>mass %</th>
<th>mol kg$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-propanamine; n-C₃H₇N</td>
<td>[107-10-8]</td>
<td>33.4</td>
<td>1.849</td>
</tr>
<tr>
<td>di-2-butylamine; (sec-C₃H₇N)₂NH; [626-23-3]</td>
<td>1.2</td>
<td>0.045</td>
<td></td>
</tr>
</tbody>
</table>

$^a$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.
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.
<table>
<thead>
<tr>
<th>COMPONENTS:</th>
<th>ORIGINAL MEASUREMENTS:</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>VARIABLES:</th>
<th>PREPARED BY:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Room temperature: T/K = 298 ± 3</td>
<td>T. Mioduski and M. Salomon</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>EXPERIMENTAL VALUES:</th>
</tr>
</thead>
</table>

Starting with the solvate HoCl₃.3C((CH₃)₂N)₃PO, the solubility at 25 ± 3°C was given as 0.108 mol dm⁻³.

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.

<table>
<thead>
<tr>
<th>AUXILIARY INFORMATION</th>
</tr>
</thead>
</table>

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 (Tm = 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 HoCl₃.3C₆H₁₈N₃O₃P. 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:
HoCl₃.3C₆H₁₈N₃O₃P prepared by dissolving the hydrate in the solvent and heating to 140-145°C for 5 min. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over P₂O₅ in a stream of dry nitrogen. Yield was about 90%.

The solvent was purified as described in (1).

ESTIMATED ERROR:
Soly: precision ± 0.001 mol dm⁻³ at a 95% level of confidence (authors).
Temp: precision ± 3 K.

REFERENCES:
COMPONENTS:
(1) Holmium chloride; HoCl₃; [10138-62-2]
(2) Tetrachlorostannate; SnCl₄; [7646-78-8]
(3) Phosphorus oxychloride; POC₁₃; [10025-87-3]

ORIGINAL MEASUREMENTS:
Lyubimov, E.I.; Batyaev, I.M.

VARIABLES:
T/K = 293
Concentration of SnCl₄

PREPARED BY:
T. Mioduski

EXPERIMENTAL VALUES:

<table>
<thead>
<tr>
<th>SnCl₄:POCl₃ ratio (by volume)</th>
<th>SnCl₄ concentration mol dm⁻³</th>
<th>solubility₃,₅ moles Ho dm⁻³</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0.003</td>
</tr>
<tr>
<td>1:100</td>
<td>0.085</td>
<td>0.009</td>
</tr>
<tr>
<td>1:50</td>
<td>0.17</td>
<td>0.026 (0.015)</td>
</tr>
<tr>
<td>1:25</td>
<td>0.33</td>
<td>0.041</td>
</tr>
<tr>
<td>1:15</td>
<td>0.59</td>
<td>0.031</td>
</tr>
<tr>
<td>1:10</td>
<td>0.78</td>
<td>0.044</td>
</tr>
</tbody>
</table>

₃Solutions preheated to 220°C. Value in parenthesis corresponds to preheating at 120°C.

₅This is also the solubility of HoCl₃ in the SnCl₄-POCl₃ mixture because the oxide is quantitatively converted to the chloride according to

$$\text{Ho}_2\text{O}_3 + 6\text{POCl}_3 = 2\text{HoCl}_3 + 3\text{P}_2\text{O}_5\text{Cl}_4$$

Authors state that the solubility of HoCl₃ is enhanced by complex formation according to

$$2\text{HoCl}_3 + 3\text{SnCl}_4 = \text{Ho}_2(\text{SnCl}_6)_3$$

METHOD/APPARATUS/PROCEDURE:
Isothermal method used. POCl₃ + SnCl₄ solutions were prepared by volume in a dry box. The SnCl₄ content was verified by chemical analysis for Sn. This solution and H₂O₂ 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 equilibrium time at 20°C to 2 hours.

Ho 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:
H₂O₂ of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade SnCl₄ and POCl₃ were dehydrated with P₂O₅ 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: