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The Extraction of Yb(III) and Lu(III) with Chloroform from Aqueous Solutions in the Presence of Cupferron

Danuta M. Czakis-Sulikowska and **Natalia Pustelnik***

Institute of General Chemistry, Technical University,
PL-90924 Łódź, Poland

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The equilibrium of distribution of Yb(III) and Lu(III) between chloroform and the aqueous phase in the presence of cupferron (the ammonium salt of N-nitrosophenylhydroxylamine) were studied as a *pH* function of the aqueous phase and the concentration of N-nitrosophenylhydroxylamine (H*L*).

The stability constants for the $LnL_n^{(3-n)}$ complexes ($n = 1 \div 3$) being formed in the aqueous phase were established, as well as the equilibrium constants of the extraction reaction

$$Ln(H_2O)_m^{3+} + 3\,HL_{(o)} \overset{K_{ex}}{\rightleftharpoons} LnL_{3(o)} + 3\,H^+ + m\,H_2O\,(Ln^{3+} = \mathrm{Yb, Lu}),$$

two-phase stability constants for the LnL_3 complexes, $pH_{0.5}$ and the separation factor Lu(III) from Yb(III).

(Keywords: Cupferron; Extraction; Rare earth complexes)

Die Extraktion von Yb(III) *und* Lu(III) *aus wäßrigen Lösungen mittels Chloroform in Anwesenheit von Cupferron*

Das Gleichgewicht der Verteilung von Yb(III) und Lu(III) zwischen Chloroform und Wasser in Anwesenheit von Cupferron (Ammoniumsalz des N-Nitrosophenylhydroxylamins) als Funktion des *pH*-Wertes der Wasserphase und der Konzentration des N-Nitrosophenylhydroxylamins (H*L*) wurde untersucht.

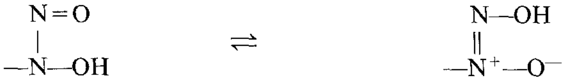
Die Beständigkeits-Konstanten der in der Wasserphase entstehenden Komplexe $LnL_n^{(3-n)}$ ($n = 1 \div 3$), die Gleichgewichtskonstante der Extraktions-Reaktion:

$$Ln(H_2O)_m^{3+} + 3\,HL_{(o)} \overset{K_{ex}}{\rightleftharpoons} LnL_{3(o)} + 3\,H^+ + m\,H_2O\,(Ln^{3+} = \mathrm{Yb, Lu}),$$

die zweiphasigen Beständigkeits-Konstanten der Komplexe LnL_3, $pH_{0.5}$ und der Trennungskoeffizient des Lu(III) von Yb(III) wurden bestimmt.

Introduction

Cupferron (the ammonium salt of N-nitrosophenylhydroxylamine) is used for both precipitatory and extractive eduction and separation of ions of several metals [*e.g.* 1–6]. Free N-nitrosophenylhydroxylamine (HL), showing acidic properties in aqueous solutions [7], may occur in two tautomeric variations [4, 8, 9]



$$\begin{array}{c} N{=}O \\ | \\ -N-OH \end{array} \quad \rightleftharpoons \quad \begin{array}{c} N-OH \\ \| \\ -N^{+}-O^{-} \end{array}$$

I II

Together with ions of several metals, it forms stable, five-membered chelate rings [1, 3, 8]. According to some authors [8, 9] they originate from form II through the substitution of the hydrogen atom by metal and a coordinative bond with oxygen.

Popov and *Wendlandt* [10] found out that in an aqueous solution containing trivalent rare earth metal ions, soluble complex MeL^{2+} ions are formed, along with deposits consisting of MeL_3 (Me = La, Ce, Pr, Nd, Sm, Gd). Other sources [11] also report sparingly soluble cupferrates Ce(III) and Nd(III) to have the MeL_3 composition. According to *Vasilienko*'s data [12], the sparingly soluble compound of cupferron and Y(III) has the composition YL_3 NH_4L.

As it can be seen from *Cheng*'s [13], *Philipienko*'s [14], and others [1] research, all the rare earth elements can, under suitable conditions, form sparingly water-soluble cupferron compounds. They can be dissolved in numerous organic solvents among in 4-methyl-2-pentanone [13], methyl isobutyl ketone [1], chloroform [with the exception of Pr(III) and Er(III)] [14].

The cupferrates of the rare earth elements—the cerium and yttrium subgroups—differ markedly with regard to their dissolubility and stability in organic solvents containing oxygen (cyclohexanol, cyclohexanone, isoamyl alcohol, ethyl acetate) [15]. It was suggested that these differences may be utilized for approximate separation of mixture of these elements. Ce(III) can be eliminated from the aqueous phase completely, in the form of a cupferrate, by means of triple extraction with chloroform [16]. La(III), Sm(III), and Y(III) in the presence of cupferron are extracted both by chloroform and methyl isobutyl ketone, in the form of MeL_3 compounds [17–20]. It follows from *Dyrssen*'s research that methyl isobutyl ketone is a more effective extraction solvent of the cupferrates of these elements than chloroform [17, 19]. Y(III) in the presence of considerable cupferron surplus can be quantitavely extracted with isoamyl alcohol [21].

Distinct differences in the dissolubility of the rare earth metal cupferrates in various organic solvents justify the possibility of using cupferron for their separation by an extraction method. The values of equilibrium constants of the extraction (K_{ex}) from the aqueous phase with chloroform [$\lg K_{ex} = -6.22$ (La), -4.74 (Y) [20], -5.8 (Sm) [22]] determined for some of them, seem to confirm such a possibility.

This paper presents the distribution equilibrium data for the extraction of Yb(III) and Lu(III) in tracer concentration with chloroform from aqueous solutions in the presence of cupferron and interprets the results on the basis of a probable extraction reaction.

Experimental

Cupferron (Reachim p. a.) was purified (due to its instability). A saturated aqueous solution of cupferron was decolourized with active carbon; then N-nitrosophenylhydroxylamine was precipitated with hydrochloric acid. After being dissolved in ethanol, it was later neutralized with ammonia [23]. The obtained crystals were repeatedly rinsed with ethanol and then recrystallized from ethanol, rinsed with ether and dried in a vacuum desiccator over ammonium carbonate. They were stored without exposure to light, in the presence of ammonium carbonate [24]. The melting point of the purified compound was 163 °C. It was the lowest temperature in which the compound would melt without external signs of decomposition (browning). The capillary tubes containing the compound were introduced into an oil bath of established temperature. The cupferron solutions were prepared by means of dissolving a given weighed portion of the compound in water just before it was used.

The basic solutions of $Yb(ClO_4)_3$ or $Lu(ClO_4)_3$ were prepared by dissolving Yb_2O_3 (99.9% pure—Fluka) or Lu_2O_3 (99.99% pure—Koch-Light Lab.) in $HClO_4$ (3 mol/dm^3) and then diluting it with water. The total lanthanide concentration in the solution was determined by *EDTA* titration with xylenol orange as indicator [25]. Chloroform (POCh—Gliwice p. a.) was purified by destillation. A 0.05% aqueous solution of the sodium salt of bis-2,7-(2-arseno-benzo-1-azo)—chromotropic acid (Arsenazo III, Reachim p. a.) was used. Perchloric acid was obtained from Hopkin Williams (p. a.). All other reagents, from POCh—Gliwice, were analytically pure. Redestillated water was used for the preparation of solutions, from which gases had been removed by boiling.

Extraction Procedure

20 cm^3 of an aqueous phase containing 1 000 μg Yb(III) od Lu(III) and varying quantities of cupferron (0.020–0.200 mmoles) was extracted with 20 cm^3 $CHCl_3$. The *pH* of the aqueous phase was adjusted with a small amount of diluted solutions of perchloric acid or sodium hydroxide. The ionic strength was kept constant ($\mu = 0.1$ mol/dm^3) with the addition of sodium perchlorate. The extraction was conducted at the temperature of 21 ± 1 °C, by mechanical mixing (laboratory shaker of the WV-3 type). Because of the instability of cupferron in acid environment [9, 26], the extraction time did not exceed 5 minutes. In initial tests it was found that this time was sufficient to achieve the equilibrium state of the system. After the separation of the phases, the *pH* of the aqueous phase was measured (*pH*-meter Radelkis of OP-206 type, measuring accuracy ± 0.02 *pH*), as well as the lanthanide(III) concentration in both phases. The lanthanide content in the organic phase was determined by means of the re-extraction method. It was found that a single re-extraction of the organic phase with an equal volume of

perchloric acid of 2.0 mol/dm^3 concentration for 5 minutes allows for total elimination of Yb(III) or Lu(III) from the organic phase.

The concentration of lanthanide in the aqueous phase and in the re-extract was determined by using the colorimetric method from Arsenazo-III [27] in the medium of $HClO_4$ (0.10 mol/dm^3 concentration). The absorbance was measured at $\lambda = 650$ nm, on a spectral colorimeter in a 5 cm cell.

The balance of the determined Yb(III) or Lu(III) content in both phases corresponded to the amount introduced (measuring accuracy ± 5%).

The distribution coefficient (D) of Ln(III) was calculated as:

$$D = \frac{[Ln(\mathrm{III})]_{tot(\mathrm{o})}}{[Ln(\mathrm{III})]_{tot(aq)}} \tag{1}$$

where $[Ln(\mathrm{III})]_{tot(\mathrm{o})}$ and $[Ln(\mathrm{III})]_{tot(aq)}$ refer to the Ln(III) concentration in the equilibrium state in the organic and aqueous phase, respectively.

The changes of the Yb(III) and Lu(III) distribution ceoefficient D were determined in two series of measurements:

(i) with constant initial cupferron concentration in the aqueous phase and variable pH,

(ii) with constant *pH* and variable cupferron concentration.

Results and Discussion

N-nitrosophenylhydroxylamine (HL) in chloroform in the concentration range 10^{-5}–10^{-2} mol/cm^3 does not form dimers [26]. In order to calculate the concentration of the $[L^-]_{(\mathrm{aq})}$ anion in the aqueous phase in equilibrium state the following equation may be written on the basis of balance of ligand:

$$C_{\mathrm{NH_4}L_{in}} \cdot V_{aq,in} = V_{(\mathrm{o})}([\mathrm{H}L]_{(\mathrm{o})} + N[LnL_N]_{(\mathrm{o})}) + V_{(aq)}([L^-]_{(aq)}$$

$$+ [\mathrm{H}L]_{(aq)} + \sum_{n=1}^{S} n[LnL_n]_{(aq)}) \tag{2}$$

where $C_{\mathrm{NH_4}L_{in}}$ is the initial concentration of cupferron, $[\mathrm{H}L]_{(\mathrm{o})}$ and $[\mathrm{H}L]_{(aq)}$ are the equilibrium concentrations of free HL in the organic and aqueous phases, $[LnL_N]_{(\mathrm{o})}$ and $[LnL_n]_{(aq)}$ are the equilibrium concentrations of complexes Ln(III) with L^- in the organic and the aqueous phases, $V_{aq,in}$ and $V_{(aq)}$, $V_{(\mathrm{o})}$ are the initial and the equilibrium volumes of phases.

When the dissociation constant ($K_{\mathrm{H}L}$) and distribution constant ($P_{\mathrm{H}L}$) of HL are introduced

$$K_{\mathrm{H}L} = \frac{[\mathrm{H}^+][L^-]}{[\mathrm{H}L]} \tag{3}$$

$$P_{\mathrm{H}L} = \frac{[\mathrm{H}L]_{(\mathrm{o})}}{[\mathrm{H}L]_{(aq)}} \tag{4}$$

with equal volumes ($V_{aq,in} = V_{(o)} = V_{(aq)}$) from Eq. (2) we get

$$[L^{-}]_{(aq)} = (C_{\mathrm{NH_4}L_{in}} - N[LnL_N]_{(o)} - \sum_{n=1}^{S} n[LnL_n]_{(aq)}) \cdot \alpha$$

where

$$\alpha = 1 \Bigg/ \left[\frac{[\mathrm{H}^{+}]}{K_{\mathrm{H}L}} (1 + p_{\mathrm{H}L}, + 1 \right] \tag{5}$$

The preliminary curves showing the dependence of the determined distribution coefficients Yb(III) and Lu(III) on $[L^{-}]_{(aq)}$ were obtained for the approximate $[L^{-}]_{(aq)}$ values calculated from equation

$$[L^{-}]_{(aq)} = C_{\mathrm{NH_4}L_{in}} \cdot \alpha \tag{6}$$

The dependencies of $\lg D$ on $\lg [L^{-}]_{(aq)}$ obtained for different initial cupferron concentrations in the aqueous phase within the *pH* range 2–6, both for Yb(III) and Lu(III), can be described by one curve. The tangent obliquity to this curve in the range of low $[L^{-}]_{(aq)}$ values is ≈ 2.7. This obliquity decreases gradually, together with an increase of $[L^{-}]_{(aq)}$ up to that of 0, $[L^{-}]_{(aq)}$ values being high. Such type of $\lg D$ dependency on $\lg [L^{-}]_{(aq)}$ indicates that a LnL_3 complex is extracted into the organic phase [28, 29] and the highest complex appearing in the system within the investigated *pH* range is LnL_3 [in Eq. (5) N and S values are equal 3.0].

The influence of a reaction of Yb(III) and Lu(III) hydroxo complexes formation in the aqueous phase may be disregarded in the measurement conditions [30]. The complexes of lanthanide with ammonia are thermodynamically unstable in aqueous solution [31]. In the applied conditions no complexes of *Ln*(III) with $\mathrm{ClO_4^-}$ ions are formed either [32]. The initial experiments have shown that Yb(III) and Lu(III) are not extracted with chloroform in the absence of cupferron.

Figures 1 and 2 present the dependency of the determined distribution coefficients Yb(III) (Fig. 1) and Lu(III) (Fig. 2) on free $[L^{-}]_{(aq)}$ anion concentration within the *pH* range 2–6 of the aqueous phase at $pK_{\mathrm{H}L} = 4.16$ and $\lg p_{\mathrm{H}L} = 2.18$ [7]. The free $[L^{-}]_{(aq)}$ values were found from Eq. (5) by successive approximation.

Preliminary values of $\sum_{n=1}^{3} n[LnL_n]_{(aq)}$ were calculated from the relationship

$$\sum_{n=1}^{3} n[LnL_n]_{(aq)} = \frac{[Ln(\mathrm{III})]_{tot(aq)} \sum_{n=1}^{3} n\beta_n [L^{-}]^{n}_{(aq)}}{(1 + \sum_{n=1}^{3} \beta_n [L^{-}]^{n}_{(aq)})} \tag{7}$$

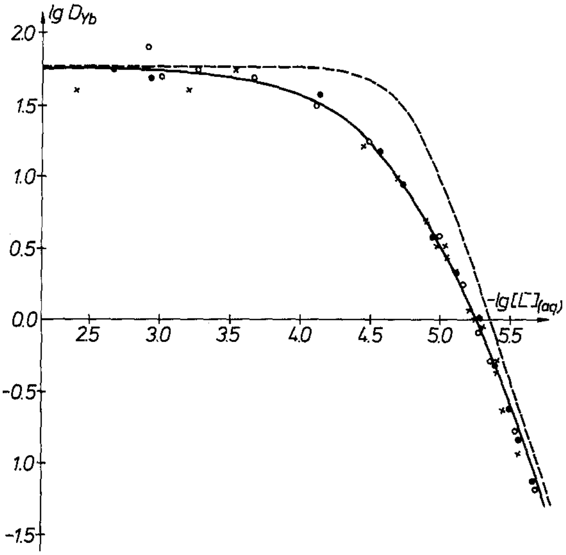


Fig. 1. The distribution coefficient D of Yb(III) as a function of $[L^-]_{(aq)}$ in the aqueous phase. $C_{\mathrm{Yb(III)},in} = 50\ \mu\mathrm{g/cm^3}$; $C_{\mathrm{NH_4}L,in} = 3.00 \cdot 10^{-3}$ (●), $5.00 \cdot 10^{-3}$ (○), $1.00 \cdot 10^{-2}$ mol/dm^3 (×). The full-drawn curve is determined by the parameters $\lg p_{\mathrm{Yb}L_3} = 1.76$, $1/3 \lg \beta_{\mathrm{Yb}L_3} = 4.77$, $1/2 \lg \beta_n^2/\beta_{n-1} \cdot \beta_{n+1} = 0.21$ (the broken limiting curve corresponds to $1/2 \lg \beta_n^2/\beta_{n-1} \cdot \beta_{n+1} = -\sim$)

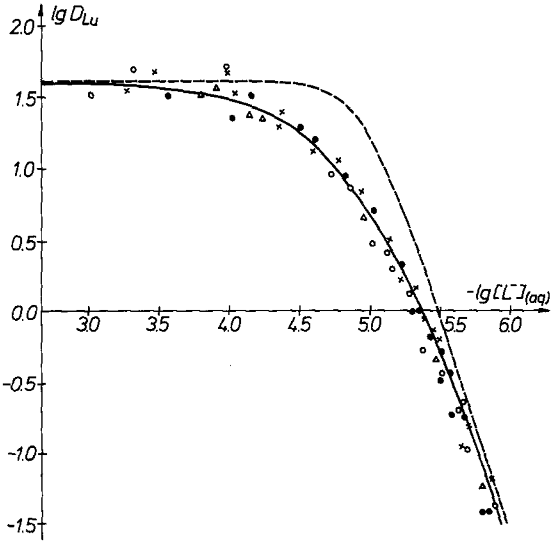


Fig. 2. The distribution coefficient D of Lu(III) as a function of $[L^-]_{(aq)}$ in the aqueous phase. $C_{\mathrm{Lu(III)},in} = 50\ \mu\mathrm{g/cm^3}$; $C_{\mathrm{NH_4}L,in} = 1.00 \cdot 10^{-3}$ (△), $3.00 \cdot 10^{-3}$ (●), $5.00 \cdot 10^{-3}$ (○), $1.00 \cdot 10^{-2}$ mol/dm^3 (×). The full-drawn curve is determined by the parameters $\lg p_{\mathrm{Lu}L_3} = 1.60$, $1/3 \lg \beta_{\mathrm{Lu}L_3} = 4.95$, $1/2 \lg \beta_n^2/\beta_{n-1} \cdot \beta_{n+1} = 0.21$ (the broken limiting curve corresponds to $1/2 \lg \beta_n^2/\beta_{n-1} \cdot \beta_{n+1} = -\sim$)

where β_n is the stability constant of a given complex, and together with the value of $[LnL_3]_{(o)}$ substituted into Eq. (5) to give better values of $[L^-]_{(aq)}$.

The total Ln(III) concentration in the aqueous phase may be illustrated by the following equation:

$$[Ln(\mathrm{III})]_{tot(aq)} = [Ln^{3+}]_{(aq)} + \sum_{n=1}^{3} [LnL_n^{(3-n)}]_{(aq)}$$

$$= [Ln^{3+}]_{(aq)}(1 + \sum_{n=1}^{3} \beta_n [L^-]^n_{(aq)}) \qquad (8)$$

The distribution constant (p_{LnL_3}) of the extracted complex LnL_3 and its stability constant (β_{LnL_3}) are equal to:

$$p_{LnL_3} = \frac{[LnL_3]_{(o)}}{[LnL_3]_{(aq)}} \qquad (9)$$

$$\beta_{LnL_3} = \frac{[LnL_3]}{[Ln^{3+}][L^-]^3} \qquad (10)$$

Equation (1), after Eqs. (8)–(10) have been considered, will assume the following form:

$$D = p_{LnL_3} \frac{\beta_{LnL_3} \cdot [L^-]^3_{(aq)}}{(1 + \sum_{n=1}^{3} \beta_n [L^-]^n_{(aq)})} \qquad (11)$$

On the basis of Eq. (11), stability constants β_n were estimated for $LnL_n^{(3-n)}$ complexes being formed in the system, and distribution constants p_{LnL_3} of the extracted chelates were determined by means of the two-parameter *Dyrssen* and *Sillen* method [33]. Table 1 lists the determined values.

The two-phase stability constant of the extracted LnL_3 complexes (β_3^x) may be calculated from the relationship

$$\lg \beta_3^x = \lg \beta_{LnL_3} + \lg p_{LnL_3} \qquad (12)$$

In Table 1 β_3^x values have been given as well, calculated from Eq. (11) for the obtained values of stability constants β_{LnL_n} of the complexes, and for the values $[L^-]_{(aq)}$ determined graphically for $D = 1$.

On the basis of the results presented above, the process of Yb(III) and Lu(III) extraction in the studied system can be described by the following summary equation:

$$Ln^{3+}_{(aq)} + 3\,\mathrm{H}L_{(o)} \rightleftharpoons LnL_{3(o)} + 3\,\mathrm{H}^+_{(aq)} \qquad (13)$$

Table 1. *The values of the stability constants* (β_{LnL_n}) *for* $LnL_n^{(3-n)}$ *complexes, distribution constants* (p_{LnL_3}) *and two-phase stability constants* (β_3^x) *for the* LnL_3 *complexes (at 21 ± 1 °C, ionic strength 0.1)*

Constant	Yb(III)	Lu(III)
$\lg \beta_{LnL^{2+}}$ (± 0.04)	5.19	5.37
$\lg \beta_{LnL_2^+}$ (± 0.04)	9.96	10.32
$\lg \beta_{LnL_3}$ (± 0.04)	14.31	14.85
$\lg p_{LnL_3}$ (± 0.05)	1.76	1.60
$\lg \beta_3^x$ (± 0.04)	16.07	16.45

Table 2. *The equilibrium constants* ($\lg K_{ex}$) *of* Yb(III) *and* Lu(III) *extraction reaction with* $CHCl_3$ *from aqueous solutions in the presence of cupferron (at 21 ± 1 °C, ionic strength 0.1)*

Conditions of measurements		$pH_{0.5}$	$\lg K_{ex}$
Variable	Constant		
	Extraction of Yb(III)		
pH	$C_{NH_4L_{in}}$ (mol/dm^3)		
2.60 ÷ 6.40	$3.00 \cdot 10^{-3}$	3.69	− 2.93
	$5.00 \cdot 10^{-3}$	3.44	− 2.97
	$1.00 \cdot 10^{-2}$	3.12	− 2.98
$C_{NH_4L_{in}}$ (mol/dm^3)	*pH*		
$1.00 \cdot 10^{-3}$ ÷	3.40		− 2.95
÷ $1.00 \cdot 10^{-2}$	3.60		− 2.96
	Average $\lg K_{ex}$(Yb)	± 0.04	− 2.96
	Extraction of Lu(III)		
pH	$C_{NH_4L_{in}}$ (mol/dm^3)		
2.10 ÷ 6.50	$3.00 \cdot 10^{-3}$	3.58	− 2.68
	$5.00 \cdot 10^{-3}$	3.33	− 2.62
	$1.00 \cdot 10^{-2}$	3.00	− 2.60
$C_{NH_4L_{in}}$ (mol/dm^3)	*pH*		
$1.00 \cdot 10^{-3}$ ÷	3.00		− 2.60
÷ $1.00 \cdot 10^{-2}$	3.60		− 2.65
	Average $\lg K_{ex}$(Lu)	± 0.05	− 2.63

The equilibrium constant of this process (K_{ex}) will be

$$K_{ex} = \frac{[LnL_3]_{(o)}[\mathrm{H}^+]^3_{(aq)}}{[Ln^{3+}]_{(aq)}[\mathrm{H}L]^3_{(o)}} \tag{14}$$

Taking into account Eqs. (3), (4), and (11) it may be shown from Eq. (14) that:

$$\lg K_{ex} = \lg D + \lg\left(1 + \sum_{n=1}^{3} \beta_n [L^-]^n_{(aq)}\right) + 3\lg\frac{K_{\mathrm{H}L}}{p_{\mathrm{H}L}} - 3\lg[L^-]_{(aq)} \qquad (15)$$

The values $\lg K_{ex}$, calculated on the basis of Eq. (15) for the obtained values of D in different series of measurements, have been compiled in Table 2. The values $[L^-]_{(aq)}$ were calculated from Eq. (5).

The separation factor between Lu(III) and Yb(III) (obtained from the ratio of K_{ex} values) is 2.1.

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