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Abstract. Trivalent metal ions have a relevant impact on different kinds of industry; for example, indium(III) and europium(III) ions are of practical importance in the development of new semiconductors and luminescent probes, respectively. Transport properties of ions and salts in aqueous solutions are important physico-chemical parameters allowing a better understanding of the behaviour of these ions in the solution and so, helping to describe better the mechanism of processes taking place in their presence. However, the measurement of those transport properties is complicated due to the occurrence of hydrolysis; that may justify the scarcity of *e.g.* diffusion data for aqueous solutions of europium(III) and indium(III) chlorides. In this study mutual diffusion coefficients for aqueous solutions of InCl_3 and EuCl_3 in a concentration range from $0.002 \text{ mol}\cdot\text{dm}^{-3}$ to $0.01 \text{ mol}\cdot\text{dm}^{-3}$ at 298.15 K are reported. The open-ended conductometric capillary cell was used. The results are discussed on the basis of the Onsager-Fuoss and Pikal models.

Keywords: diffusion coefficients, europium(III), indium(III), aqueous solutions.

1. Introduction

Europium(III) is a trivalent lanthanide ion with attractive and versatile spectroscopic and magnetic properties [1, 2], which are an advantage for applications in different fields such as biochemistry or materials. Eu(III) can be used as a luminescent probe of bioactive species including metal ions, oxyanions and acidity of biological environments [3-5]. Eu(III) can also be applied for the study of surfactant association in the solution [6]. The luminescent properties of Eu(III) have also been used for the development of light-emitting diodes (LED) with an improved red emission [7, 8]. Recently, europium-quantum dots and europium-fluorescein composite nanoparticles for the metal ion detection have been developed [9]. Eu(III) spectroscopy has also been used to characterize the

complicated structural evolution that takes place during the gelation and densification of materials prepared by the sol-gel process [10].

Indium(III) chloride is an efficient catalyst for inducing various types of organic reactions, such as synthesis of saccharides [11], Mukaiyama aldol reactions [12], Diels-Alder reactions [13, 14], aza-Michael reactions [15], and also in microwave irradiation assisted synthesis [16]; other practical applications of indium(III) chloride includes, for example, its use as a constituent of a photosensitizer used as a photodynamic therapy agent for ocular diseases [17]. However, one of the broadest application of In(III) is as indium tin oxide (ITO), a solid solution with excellent electrical and optical properties [18]. In the last few years, the combined use of these metals has been attempted in order to improve the properties of LEDs, by using Eu(III) as a buffer layer on ITO [19] or introduce luminescent properties to ITO films or nanoparticles [20, 21].

However, the use of Eu(III) and In(III) in solids and solutions requires an understanding of the factors affecting the properties of the ion. In this paper we report about diffusion coefficients of aqueous solutions of Eu(III) and In(III) chlorides at 298.15 K contributing for better knowledge of the behavior of those ions in the solution.

2. Experimental

2.1. Reagents

The solutes used in this study were indium chloride and europium chloride (Aldrich, *pro analysi* > 97 % and *pro analysi* > 99.9 %, respectively) without further purification. Aqueous solutions were prepared using bi-distilled water. All solutions were freshly prepared just before each experiment.

2.2. Diffusion Measurements

The open-ended capillary cell used was constructed in this laboratory and is essentially the same as that

previously reported [22]. The cell has two vertical capillaries, each closed at one end by a platinum electrode and positioned one above the other with the open ends separated by *ca.* 14 mm.

The upper (top) and lower (bottom) tubes, initially filled with solutions of $0.75c$ and $1.25c$ concentrations, respectively, were surrounded with a solution of concentration c . This ambient solution was contained in a $200 \times 140 \times 60$ mm glass tank, which was immersed in a bath thermostatted at 298.15 K. The tank was divided internally by Perspex sheets, while a glass stirrer created a slow lateral flow of ambient solution across the open ends of the capillaries. Experimental conditions were such that the concentration at each of the open ends was equal to the ambient solution value c , that is the physical length of the capillary tube coincided with the diffusion path. This means that the required boundary conditions described in literature [22] to solve Fick's second law of diffusion are applicable. Therefore, the so-called Δl -effect [22] is reduced to negligible proportions. In contrast to a manual apparatus, where diffusion is followed by measuring the ratio of resistances of the top and bottom tubes, $w = R_t/R_b$ by an alternating current transformer bridge, in our automatic apparatus w was measured by a Solartron digital voltmeter (DVM) 7061 with 6 1/2 digits. A Bradley Electronics Model 232 power source supplied 30 V (stable to ± 0.1 mV) to a potential divider that applied a 250 mV signal to the platinum electrodes in the top and bottom capillaries. By rapidly (< 1 s) measuring the V and V' voltages from the top and bottom electrodes relative to the central electrode at ground potential the w was then calculated from the DVM readings.

To measure the differential diffusion coefficient D at a given concentration c , 2 dm³ each of a "top" solution of concentration $0.75c$ and a "bottom" solution $1.25c$ were prepared. The "bulk" solution of concentration c was produced by mixing accurately the measured volumes of 1 dm³ of the "top" solution with 1 dm³ of the "bottom"

solution. The glass tank and the two capillaries were filled with solution c immersed in the thermostat, and were allowed to come to thermal equilibrium. The quantity $TR_{inf} = 10^4/(1 + w)$ was now measured very accurately (where $w = R_t/R_b$ is the electrical resistance ratio for solutions of concentration c of the top (t) and bottom (b) diffusion capillaries at infinite time). $TR = 10^4/(1 + w)$ is the equivalent at any time t .

The capillaries were then filled with "top" and "bottom" solutions, which were allowed to diffuse into the "bulk" solution. Resistance ratio readings were taken at various times, beginning 1000 min after the start of an experiment. The diffusion coefficient was evaluated using a linear least-squares procedure to fit the data, followed by an iterative process which uses 20 terms of the expansion series of the solution of the Fick's second law for the present boundary conditions. The theory developed for this cell has been described previously [22].

3. Results and Discussion

Tables 1 and 2 show the experimental diffusion coefficients D of EuCl_3 and InCl_3 in aqueous solutions at 298.15 K. These results are the average of 3 experiments performed on consecutive days. The experimental procedure shows good reproducibility, as shown by the small standard deviations, $S_{D_{\text{av}}}$. The accuracy of the systems (uncertainty 1–2 %) has been demonstrated by measurements on other solutions of different electrolytes (*e.g.* [2-16]).

The following polynomial in c was fitted to the data by a least squares procedure,

$$D = a_0 + a_1 c + a_2 c^2 \quad (1)$$

where the coefficients a_0 , a_1 , and a_2 are adjustable parameters. Table 3 shows the coefficients a_0 to a_2 of Eq. (1).

Table 1

Diffusion coefficients \bar{D} of EuCl_3 in aqueous solutions at different concentrations c at 298.15 K

c , mol·dm ⁻³	\bar{D} ^{a)} , 10 ⁻⁹ m ² ·s ⁻¹	$S_{\bar{D}}$ ^{b)} , 10 ⁻⁹ m ² ·s ⁻¹	D_{OF} ^{c)} , 10 ⁻⁹ m ² ·s ⁻¹ ($a = 5.6 \cdot 10^{-10}$ m ^{d)})	D_{Pik} ^{c)} , 10 ⁻⁹ m ² ·s ⁻¹ ($a = 5.6 \cdot 10^{-10}$ m ^{d)})	$\Delta D/D_{\text{OF}}$ ^{e)} , %	$\Delta D/D_{\text{Pik}}$ ^{e)} , %
$2 \cdot 10^{-3}$	1.216	0.010	1.180	1.519	3.0	-19.9
$3 \cdot 10^{-3}$	1.200	0.013	1.169	1.795	2.6	-33.1
$5 \cdot 10^{-3}$	1.179	0.020	1.158	2.205	1.8	-46.5
$8 \cdot 10^{-3}$	1.160	0.011	1.153	3.009	0.6	-61.4
$1 \cdot 10^{-2}$	1.151	0.010	1.152	3.994	-0.1	-71.2

Notes: ^{a)} \bar{D} is the mean diffusion coefficient of 3 experiments; ^{b)} $S_{\bar{D}}$ is the standard deviation; ^{c)} D_{OF} and D_{Pik} represent the calculated diffusion coefficients from Onsager-Fuoss and Pikal equations, respectively; ^{d)} Sum of hydrated ionic radii (diffraction methods) [23] and ^{e)} $\Delta D/D_{\text{OF}}$ and $\Delta D/D_{\text{Pik}}$ represent the relative deviations between \bar{D} and D_{OF} and D_{Pikal} values, respectively.

Table 2

Diffusion coefficients \bar{D} of InCl_3 in aqueous solutions at different concentrations c at 298.15 K

c , $\text{mol}\cdot\text{dm}^{-3}$	\bar{D} ^{a)} , $10^{-9} \text{ m}^2\cdot\text{s}^{-1}$	$S_{\bar{D}}$ ^{b)} , $10^{-9} \text{ m}^2\cdot\text{s}^{-1}$	D_{OF} ^{c)} , $10^{-9} \text{ m}^2\cdot\text{s}^{-1}$ ($a = 5.3\cdot 10^{-10} \text{ m}^{\text{d}}$)	D_{Pik} ^{c)} , $10^{-9} \text{ m}^2\cdot\text{s}^{-1}$ ($a = 5.3\cdot 10^{-10} \text{ m}^{\text{d}}$)	$\Delta D/D_{\text{OF}}$ ^{e)} , %	$\Delta D/D_{\text{Pik}}$ ^{e)} , %
$2\cdot 10^{-3}$	0.945	0.021	1.081	0.921	-12.6	2.6
$3\cdot 10^{-3}$	0.867	0.026	1.077	0.844	-19.5	2.7
$5\cdot 10^{-3}$	0.686	0.023	1.072	0.698	-36.0	-1.7
$8\cdot 10^{-3}$	0.622	0.020	1.067	0.477	-41.2	30.3
$1\cdot 10^{-2}$	0.619	0.011	1.067	0.370	-42.0	67.3

Notes: ^{a)} \bar{D} is the mean diffusion coefficient of 3 experiments; ^{b)} $S_{\bar{D}}$ is the standard deviation; ^{c)} D_{OF} and D_{Pik} represent the calculated diffusion coefficients from Onsager-Fuoss and Pikal equations, respectively; ^{d)} Sum of hydrated ionic radii (diffraction methods) [23] and ^{e)} $\Delta D/D_{\text{OF}}$ and $\Delta D/D_{\text{Pik}}$ represent the relative deviations between \bar{D} and D_{OF} and D_{Pikal} values, respectively.

They may be used to calculate values of diffusion coefficients at specified concentrations within the range of the experimental data shown in Tables 1 and 2. The goodness of the fit (obtained with a confidence interval of 98 %) can be assessed by the correlation coefficient, R^2 .

Table 3

Fitting coefficients (a_0 - a_2) of a polynomial equation $[D/(10^{-9} \text{ m}^2\cdot\text{s}^{-1}) = a_0 + a_1 (c/\text{mol}\cdot\text{dm}^{-3}) + a_2 (c/\text{mol}\cdot\text{dm}^{-3})^2]$ to the mutual differential diffusion coefficients of europium chloride and indium chloride in aqueous solutions at 298.15 K

Electrolyte	a_0	a_1	a_2	R^2
EuCl_3	1.246	-17.06	766.7	0.997
InCl_3	1.196	-139.4	8228	0.990

3.1 Limiting Diffusion Coefficients

Extrapolation of the fit of these equations to infinitesimal concentration gives the estimated diffusion coefficients obtained (*i.e.* $D^0 = a_0$ in Table 3), which account for the diffusion of both the cation and the anion under these conditions. As can be seen in Table 4 the agreement between these values and those obtained by Nernst equation [24, 25] (Eq. (2)) using different values for equivalent conductance of europium and indium at infinitesimal concentration is reasonable.

$$D^0 = \frac{RT}{F^2} \frac{|z_1| + |z_2|}{|z_1 z_2|} \frac{I_1^0 I_2^0}{I_1^0 + I_2^0} \quad (2)$$

λ_1^0 and λ_2^0 represent the equivalent conductance of the cation and anion at infinitesimal concentration, respectively, and z_1 represents the algebraic valency of a cation and z_2 is the algebraic valency of an anion.

Table 4

Limiting diffusion coefficients D^0 for the systems $\text{EuCl}_3/\text{H}_2\text{O}$ and $\text{InCl}_3/\text{H}_2\text{O}$

Electrolyte	D_{exp}° ^{a)} , $10^{-9} \text{ m}^2\cdot\text{s}^{-1}$	$D_{\text{Nernst}}^{\circ}$, $10^{-9} \text{ m}^2\cdot\text{s}^{-1}$	$\Delta D^{\circ}/D$, % ^{d)}
EuCl_3	1.246	1.235 ^{b)} 1.274 ^{b)}	0.9 -2.2
InCl_3	1.196	1.150 ^{c)}	4.0

Notes: ^{a)} Limiting D_{exp}° values were calculated by extrapolating our experimental data, D_{exp} (Tables 1 and 2) to $c \rightarrow 0$ at 298.15 K; ^{b)} Diffusion coefficients estimated by Nernst' equation (Eq. (2)), using $I(\text{Eu}^{3+}) = 192\cdot 10^{-4} \text{ S}\cdot\text{m}^2\cdot\text{mol}^{-1}$ and $I(\text{Eu}^{3+}) = 203.4\cdot 10^{-4} \text{ S}\cdot\text{m}^2\cdot\text{mol}^{-1}$, respectively. These values were obtained by using a Stokes-Einstein equation and experimental values, respectively [26]. ^{c)} Diffusion coefficients estimated by Nernst' equation (Eq. (2)), using the value $I(\text{In}^{3+}) = 168.9\cdot 10^{-4} \text{ S}\cdot\text{m}^2\cdot\text{mol}^{-1}$ obtained by Campbell *et al.* [27].

3.2 Inter Ionic Effects on Diffusion

Having in mind to understand the transport process of this electrolyte in aqueous solutions, the experimental mutual diffusion coefficients at 298.15 K were compared as a first approach with those estimated by the Onsager-Fuoss and Pikal equations (Eqs. (5) and (10) [24, 28, 29]) (Tables 1 and 2). The first equation is expressed by:

$$D = \left(1 + c \frac{\partial \ln y_{\pm}}{\partial c} \right) (D^0 + \sum D_n) \quad (3)$$

where D is the mutual diffusion coefficient of the electrolyte, the first term in parenthesis is the activity factor, y_{\pm} is the mean molar activity coefficient, c is the concentration in mol dm^{-3} , D^0 is the Nernst limiting value

of the diffusion coefficient (Eq. (2)), and D_n are the electrophoretic terms given by:

$$D_n = k_B T A_n \frac{\left(z_1^n t_2^0 + z_2^n t_1^0 \right)^2}{|z_1 z_2|^n a^n} \quad (4)$$

where k_B is the Boltzmann's constant; T is the absolute temperature; A_n are functions of the dielectric constant, of the solvent viscosity, of the temperature, and of the dimensionless concentration-dependent quantity (ka), k being the reciprocal of average radius of the ionic atmosphere; t_1^0 and t_2^0 are the limiting transport numbers of the cation and anion, respectively.

Since the expression for the electrophoretic effect has been derived on the basis of the expansion of the exponential Boltzmann function, because that function is consistent with the Poisson equation, we only would have to take into account the electrophoretic term of the first and the second order ($n = 1$ and $n = 2$). Thus, the experimental data D_{exp} can be compared with the calculated D_{OF} on the basis of Eq. (5)

$$D = \left(1 + c \frac{\partial \ln y_{\pm}}{\partial c} \right) (D^0 + D_1 + D_2) \quad (5)$$

The theory of mutual diffusion in binary electrolytes, developed by Pikal [28], includes the Onsager-Fuoss equation, but it has new terms resulting from the application of the Boltzmann exponential function for the study of diffusion. In other words, instead of approximating the Boltzmann exponential by a truncated power series, the calculations are performed retaining the full Boltzmann exponential. As a result of this procedure, a term representing the effect of ion-pair formation appears in the theory as a natural consequence of the electrostatic interactions. The electrophoretic correction appears now as the sum of two terms

$$Dv_j = Dv_j^L + Dv_j^S \quad (6)$$

where Dv_j^L represents the effect of long-range electrostatic interactions, and Dv_j^S represents them as short-range ones.

Designated by $M = 10^{12} L/c$ is the solute thermodynamic mobility, where L is the thermodynamic diffusion coefficient, ΔM can be represented by the equation

$$\frac{1}{M} = \frac{1}{M^0} \left(1 - \frac{\Delta M}{M^0} \right) \quad (7)$$

where M^0 is the value of M for infinitesimal concentration, and

$$\Delta M = \Delta M^{\text{OF}} + \Delta M_1 + \Delta M_2 + \Delta M_A + \Delta M_{\text{H1}} + \Delta M_{\text{H2}} + \Delta M_{\text{H3}} \quad (8)$$

The first term on the right hand in the above equation ΔM^{OF} represents the Onsager-Fuoss term for the effect of the concentration in the solute thermodynamic mobility M ; the second term ΔM_1 is a consequence of the approximation applied on the ionic thermodynamic force; the other terms result from the Boltzmann exponential function.

The relation between the solute thermodynamic mobility and the mutual diffusion coefficient is given by:

$$D = \frac{L}{c} 10^3 RTv \left(1 + c \frac{\partial \ln y_{\pm}}{\partial c} \right) \quad (9)$$

where R is the gas constant, and v is the number of ions formed upon complete ionization of one solute "molecule". From Eqs. (7) and (9) we obtain a version of the Pikal's equation more useful for estimating the mutual diffusion coefficients of electrolytes D_{Pikal} . That is,

$$D_{\text{Pikal}} = \frac{10^3 RTv}{\frac{1}{M^0} \left(1 - \frac{\Delta M}{M^0} \right)} \left(1 + c \frac{\partial \ln y_{\pm}}{\partial c} \right) \quad (10)$$

Both Onsager-Fuoss and Pikal's theories introduce the ion size parameter a , the distance of the closest approach from the Debye-Huckel and it is well known that there is no direct method for measuring this parameter. In this work, the values for a were estimated from Marcus data (Table XIII of Ref. [23]) using two approximations (Table 5). Firstly, the a -values were estimated as the sum of the ionic radii (R_{ion}) reported by Marcus [23]. The R_{ion} values were obtained as the difference between the mean internuclear distance of a monoatomic ion or the central atoms of polyatomic ions, and the oxygen atom of a water molecule in its first hydration shells ($d_{\text{ion-water}}$), and the half of the mean intermolecular distance between two water molecules in a liquid water (R_{water}). Briefly,

$$R_{\text{ion}} = d_{\text{ion-water}} - R_{\text{water}} \text{ and } a = R_{\text{cation}} + R_{\text{anion}}$$

In order to account for the effect of the ion hydration shell on the a -values, a second approximation considers the sum of the $d_{\text{ion-water}}$ values reported by Markus [23]. In other words, in this approach the a -values are determined as $a = R_{\text{cation-water}} + R_{\text{anion-water}}$ (Table 5).

Table 5

Values of mean distances of the closest approach ($a/10^{-10}$ m) of the systems $\text{EuCl}_3/\text{H}_2\text{O}$ and $\text{InCl}_3/\text{H}_2\text{O}$ calculated by two methods

Electrolyte	Sum of ionic radii in solutions ^{a)} $a = R_{\text{cation}} + R_{\text{anion}}$	Sum of mean ion-water internuclear distances ^{a)} $a = d_{\text{cation-water}} + d_{\text{anion-water}}$
EuCl_3	2.86	5.64
InCl_3	2.56	5.34

Note: ^{a)} see [23]

For EuCl_3 , we see that the agreement between experimental data and Pikal calculations is not good (Table 1), eventually because of the full use of Boltzmann's exponential in Pikal's development. However, Onsager-Fuoss theory leads to calculated values close to the experimental data (deviations $\leq 3\%$, Table 1). In these estimations, the choice of the parameter a was irrelevant, within reasonable limits. We may use any value because slight variations in this parameter a have little effect on the final results of D_{OF} and D_{Pikal} . For InCl_3 (Table 2), Pikal's treatment gives better agreement with D_{exp} than Onsager-Fuoss for dilute solutions. However, in this case, the final result D_{Pikal} is strongly affected by the choice of this parameter a . In fact, there is a better agreement with D_{Pikal} if we use a value of parameter a equal to the sum of the hydrated ionic radii. Despite the limitations of this theory when applied to non symmetrical electrolytes, this good applicability in this case can lead us to admit that the effects of short range interactions on the diffusion of this electrolyte at those concentrations are relevant, contrary in EuCl_3 . The hydration of this salt, its hydrolysis [30] and the eventual formation of ion pairs, increasing with concentration, can be responsible for those effects.

For $c > 0.008\text{ M}$ the results predicted from the above model differ markedly from experimental observation (*i.e.* 30–67 %). This is not surprising if we take into account the change with concentration of parameters such as viscosity [25, 31, 32], dielectric constant [25] and hydration [25, 31–33], which are not taken into account in these models.

4. Conclusions

Experimental values for mutual diffusion coefficients for InCl_3 and EuCl_3 in a concentration range from $0.002\text{ mol}\cdot\text{dm}^{-3}$ to $0.01\text{ mol}\cdot\text{dm}^{-3}$ at 298.15 K were determined using a conductimetric cell. The results were discussed on the basis of the Onsager-Fuoss and Pikal models. For EuCl_3 , the reasonable agreement between the two sets of D values thus found suggests that the ionic species europium(III) and chloride are the main ones contributing to the transport properties and, therefore, the hydrolysis effect can be neglected. However, we see that the agreement between experimental data and Pikal calculations was not good, eventually because of the full use of Boltzmann's exponential in Pikal's development.

In contrast, for InCl_3 , we can conclude that the behaviour of the diffusion of this electrolyte depends strongly on the viscosity change and the hydration factor in the solution.

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ДИФУЗІЙНА ПОВЕДІНКА ТРИВАЛЕНТНИХ ІОНІВ МЕТАЛІВ У ВОДНИХ РОЗЧИНАХ

Анотація. Йони тривалентного металу мають суттєвий вплив на різні галузі промисловості, наприклад, йони індію(III) та європію(III) дуже важливі при розробленні нових напівпровідників і люмінесцентних зондів, відповідно. Транспортні властивості йонів та солей у водних розчинах є важливими фізико-хімічними параметрами, які дають можливість краще зрозуміти поведінку цих йонів у розчині і таким чином допомагають описувати механізм процесів, що відбуваються у їх присутності. Проте, вимірювання цих транспортних властивостей утруднене із-за появи гідролізу, який може

бути пояснений нестачею, наприклад, дифузійних даних для водних розчинів хлоридів європію(III) та індію(III). В роботі визначено, що коефіцієнти взаємної дифузії водних розчинів InCl_3 і EuCl_3 знаходяться в діапазоні концентрацій від $0,002$ до $0,01$ моль·дм⁻³ при $298,15$ К. Для досліджень використано наскрізну

кондуктомеричну капілярну комірку. Одержані результати оброблено на основі моделей Онзагера-Фюоса і Пікаля.

Ключові слова: коефіцієнти дифузії, європій(III), індій(III), водні розчини.