# Complexation of Europium(III) and Terbium(III) Ions with Terephthalic Acid in Aqueous Solutions

Ya. E. Saitov<sup>a</sup>, P. B. Guseva<sup>a</sup>, Yu. N. Toikka<sup>a</sup>, N. A. Bogachev<sup>a</sup>, M. Yu. Skripkin<sup>a</sup>, and A. S. Mereshchenko<sup>a,\*</sup>

<sup>a</sup> St. Petersburg State University, St. Petersburg, 199034 Russia \*e-mail: a.mereshchenko@spbu.ru

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**Abstract**—The stability constants of the complexes of europium(III) and terbium(III) ions with terephthalic acid anions were determined by potentiometric titration over a wide pH range. The luminescent properties of these complexes were studied. It was shown that the complexes are most stable in slightly acidic solutions. Analysis of the photoluminescence emission spectra confirmed the adequacy of the proposed model of complexation.

**Keywords:** lanthanide complexes, rare earth elements, terephthalates, stability constants, MOFs, europium, terbium **DOI:** 10.1134/S1070363224030101

## INTRODUCTION

Luminescentg materials based on triply charged lanthanide ions have been widely discussed in the recent scientific literature due to their broad applications, ranging from LEDs and various sensors to materials for bioimaging and medicine [1–4]. A low probability of *f*-*f* transitions makes it difficult to use solvated lanthanide cations as luminescent probes. One approach to solve this problem involves the use of special ligands, that effectively absorb light and transfer energy to the lanthanide ions via the so-called antenna effect [5, 6]. Typical ligands used in such antenna complexes are aromatic and unsaturated molecules: calixarenes, dipicolinic acid, bipyridines, and carboxylates, including terephthalates [7–11]. Of particular interest in this context are luminescent metal-organic frameworks (MOFs) based on lanthanide ions, demonstrating an antenna effect from organic ligands [12, 13]. The photophysical properties of MOFs are determined by their structure and composition which, in turn, depend on many parameters, in particular, on the nature of the metal ion and the linker, synthesis conditions, including the solvent and temperature chosen. For rational design of MOFs with targeted photophysical properties, it is essential to understand the mechanism of MOF formation for controlling the synthesis. With the predominantly empirical approach taken at present to the synthesis of MOFs, the mechanism of MOF

formation is, however, not well understood. In this study, we examined the first stage of the formation of europium terephthalate and terbium ones of the most the most common luminescent MOFs.

#### **RESULTS AND DISCUSSION**

Using potentiometric titration over a wide pH range, the stability constants of the complexes of europium(III) and terbium(III) ions with terephthalic acid (H<sub>2</sub>bdc) anions were determined, and their luminescent properties were investigated in the systems Eu(NO<sub>3</sub>)<sub>3</sub>-H<sub>2</sub>bdc-NaOH and Tb(NO<sub>3</sub>)<sub>3</sub>-H<sub>2</sub>bdc-NaOH. Equilibrium modeling under the assumption of the coexistence of certain probable complex species in solution and calculation of their stability constants were carried out using the ReactLab pH PRO program [14]. The initial data were the experimentally obtained as dependences of pH of the solution of a 1 : 8 mixture of europium or terbium nitrate with terephthalic acid on the degree of titration f, i.e., on the number of added equivalents of the alkali per proton of terephthalic acid (Fig. 1). The initial calculations neglected complexation between the lanthanide ions and nitrate ions in the studied systems, because the stability constants of the terephthalate complexes of lanthanides are at least five orders of magnitude higher than those of the nitrate ones (log  $\beta_1$  from -0.85 to 0.3) [15].

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**Fig. 1.** (a, b) Titration curves of (a) Ln = Eu and (b) Tb with10 mM NaOH solution: (black lines) experimental and (red lines) calculated (on the basis of the complexation model for the solution containing 0.194 mM H<sub>2</sub>bdc, 0.024 mM Ln(NO<sub>3</sub>). (c) Experimental titration curve of 0.194 mM H<sub>2</sub>bdc with 10 mM NaOH solution. All solutions contained 100 mM KNO<sub>3</sub> background electrolyte.



**Fig. 2.** (1-4) Experimental curves of titration for the solutions, containing 0.194 mM H<sub>2</sub>bdc and Ln(NO<sub>3</sub>)<sub>3</sub>, with 10 mM NaOH solution for Ln = (a) Eu and (b) Tb at the concentration ratio of terephthalic acid to lanthanide nitrate in the original solution Ln(NO<sub>3</sub>)<sub>3</sub> : H<sub>2</sub>bdc of (1) 1 : 2, (2) 1 : 3, (3) 1 : 4, and (4) 1 : 8. (5) Titration curve of 0.194 mM H<sub>2</sub>bdc with 10 mM NaOH solution is presented for comparison. All solutions contained 100 mM KNO<sub>3</sub> background electrolyte.

The titration curves of the solutions of terephthalic acid and of the mixtures of terephthalic acid with europium or terbium nitrate exhibit significant differences (Fig. 2). Specifically, the titration curve of terephthalic acid contains one titration jump at the degree of titration f=2, associated with the proceeding of its neutralization in two steps, because the dissociation constants K<sub>1A</sub> and K<sub>2A</sub> are close: log K<sub>A1</sub> = -3.54, and log K<sub>A2</sub> = -4.46 [16]. The titration curves of the mixtures of terephthalic acid with europium or terbium nitrate exhibit a titration jump at a titration degree of ~3. This jump is attributable to the formation of hydroxo complexes of europium or terbium or to deprotonation of the complex of the lanthanide ion with the acidic anion Hbdc<sup>-</sup>. To elucidate the nature of the second jump in the titration curves of the mixtures of terephthalic acid with europium or terbium nitrate, we recorded the titration curves for different concentration ratios of terephthalic acid to the lanthanide nitrate in the initial solution:  $Ln(NO_3)_3$ :  $H_2bdc = 1 : 2, 1 : 3, 1 : 4$ , and 1 : 8 (Fig. 2) at a constant concentration of terephthalic acid of 0.194 mM. It is seen that the position of the jump in the titration curves and pH of the first jump are practically independent of the concentration ratio of the terephthalic acid to the lanthanide nitrate in the original solution.

## COMPLEXATION OF EUROPIUM(III) AND TERBIUM(III) IONS

Complex	log β			
Complex	Eu	Tb		
$Ln(1,4-bdc)^+$	8.2±0.1	7.5±0.1		
$\operatorname{Ln}(1,4\text{-bdc})_2^-$	14.0±0.1	13.2±0.1		

Table 1. Logarithms of the overall stability constants of the europium(III) and terbium(III) terephthalate complexes

At the same time, for the second jump, the degree of titration, as calculated relative to terephthalic acid, tends to increase significantly with increasing lanthanide nitrate concentration, which allows attributing the second jump to the formation of hydroxo complexes of the lanthanides. Therefore, for approximating the experimental titration curves, a model of complexation involving the formation of the OH<sup>-</sup>, H<sub>2</sub>bdc, Hbdc<sup>-</sup>, Lnbdc<sup>+</sup>, Ln(bdc)<sup>-</sup><sub>2</sub>, Ln(OH)<sup>2+</sup>,  $Ln(OH)_{2}^{+}$ , and  $Ln(OH)_{3}$  species was proposed. The stoichiometric matrix was built on the basis of three species: H<sup>+</sup>, Ln<sup>3+</sup>, and bdc<sup>2-</sup>. This model affords the best match with the experimental data at a minimum number of complex species. The calculations employed the values of two dissociation constants of terephthalic acid log  $K_{A1} = -3.54$ , log  $K_{A2} = -4.46$  from [16] and three stability constants of hydroxo complexes of europium  $\log K_{1Eu} = 6.2$ ,  $\log K_{2Eu} = 11.6$ ,  $\log K_{2Eu} = 16.8$  and of terbium log  $K_{1Tb} = 6.1$ , log  $K_{2Tb} = 11.7$ , log  $K_{2Tb} = 16.9$ from [17, 18].

Table 1 lists the stability constants for the complexes of europium(III) and terbium(III) ions with terephthalate

ion, obtained via approximation of the titration curves using ReactLab pH PRO program. The tabulated data show that the complexes formed by the terephthalate ion with europium slightly exceed in stability those formed with terbium.

Through approximation of the titration curves using ReactLab pH PRO program, the concentrations of various complex species at each point were calculated. For a better visual analysis of the evolution of the distribution of the complex species during acid-base titration, the fractional distribution diagrams of the europium(III) and terbium(III) species versus pH were plotted (Fig. 3). Figure 3 reveals a change in the dominant complex species with an increase in pH, and, consequently, in the degree of titration, in the series  $Ln^{3+}-Ln(1,4-bdc)^+$ ,  $Ln(1,4-bdc)_{2}^{-}-Ln(OH)^{2+}-Ln(OH)_{2}^{+}-Ln(OH)_{3}$ . Thus, we can conclude that the slightly acidic medium at pH 4-6.5 is preferred for the formation of terephthalate complexes of europium(III) and terbium(III) and that with increasing pH the terephthalate complexes undergo destruction to form stable hydroxo complexes of the lanthanides.



**Fig. 3.** Fractional distribution diagrams X of (a) europium(III) and (b) terbium(III) complexes during titration of the solution containing 0.194 mM H<sub>2</sub>bdc and 0.024 mM Ln(NO<sub>3</sub>)<sub>3</sub> with 10 mM NaOH solution: (1) Ln<sup>3+</sup>, (2) Lnbdc<sup>+</sup>, (3) Ln(bdc)<sub>2</sub><sup>-</sup>, (4) Ln(OH)<sup>2+</sup>, (5) Ln(OH)<sub>2</sub><sup>+</sup>, and (6) Ln(OH)<sub>3</sub>.

Solution no.	Ln	рН	c, mM			
			H <sub>2</sub> [1,4-bdc]	$Ln(NO_3)_3$	NaOH	KNO3
E1	Eu	3.94	0.194	0.024	0	100
E2		4.21	0.194	0.024	0.1	100
E3		4.63	0.194	0.024	0.2	100
E4		4.92	0.194	0.024	0.24	100
E5		5.64	0.194	0.024	0.3	100
E6		6.74	0.194	0.024	0.4	100
E7		7.28	0.194	0.024	0.5	100
E8		8.26	0.194	0.024	0.6	100
T1		3.93	0.194	0.024	0	100
T2	Tb	4.20	0.194	0.024	0.1	100
Т3		4.68	0.194	0.024	0.2	100
T4		6.00	0.194	0.024	0.3	100
T5		7.03	0.194	0.024	0.4	100
T6		7.35	0.194	0.024	0.45	100
Τ7		7.74	0.194	0.024	0.5	100
Τ8		9.53	0.194	0.024	0.6	100

Table 2. Concentrations of the components in the solutions used for absorption and luminescence spectra measurement

According to published data [19–21], compounds of europium(III) and terbium(III) with terephthalic acid demonstrate pronounced luminescent properties due to the antenna effect. The reason is that the terephthalate ion has a conjugated  $\pi$ -system and intensively absorbs light, promoting it into the singlet electronic excited state S<sub>n</sub>, which undergoes intersystem crossing into the lowest-energy triplet excited electronic state T<sub>1</sub>. Due to the relative proximity between the T<sub>1</sub> state energy level ( $\approx$ 20000 cm<sup>-1</sup>) and the atomic energy level <sup>5</sup>D<sub>1</sub> of the Eu<sup>3+</sup>

ion ( $\approx$ 19000 cm<sup>-1</sup>) or <sup>5</sup>D<sub>4</sub> of the Tb<sup>3+</sup> ion ( $\approx$ 20600 cm<sup>-1</sup>) [22], energy is transferred to the Eu<sup>3+</sup> or Tb<sup>3+</sup> ion, and this is followed by relaxation to the ground electronic state of the lanthanide ion as accompanied by a photon emission. At the same time, the excitation of the europium(III) and terbium(III) ions directly into f-f excited electronic states is much less probable as specified by the parity and orbital overlap selection rules. Consequently, the luminescence intensity of the aqua and hydroxo complexes of Eu<sup>3+</sup> and Tb<sup>3+</sup> should be significantly lower than that of the



**Fig. 4.** Absorption spectra of the aqueous solutions containing terephthalic acid, (a) europium(III) or (b) terbium nitrate, sodium hydroxide, and potassium nitrate; see Table 2 for the concentrations of the substances. 0.1 M KNO<sub>3</sub> was used as a reference solution.



**Fig. 5.** (a) Emission and (b) excitation spectra of the aqueous solutions containing terephthalic acid, europium(III) nitrate, sodium hydroxide, and potassium nitrate; see Table 2 for the concentrations of the substances. The excitation wavelength in the emission spectra was 280 nm. The luminescence wavelength in the excitation spectra was 615 nm.

λ, nm



**Fig. 6.** (a) Emission and (b) excitation spectra of the aqueous solutions containing terephthalic acid, terbium(III) nitrate, sodium hydroxide, and potassium nitrate; see Table 2 for the concentrations of the substances. The excitation wavelength in the emission spectra was 280 nm. The luminescence wavelength in the excitation spectra was 543 nm.

terephthalate complexes in the emission spectra measured under the same conditions. Thus, analysis of the emission spectra can also be useful for analyzing the distribution of the complex species and for testing the complexation model proposed on the basis of the potentiometric titration data.

I<sub>lum</sub>, rel. units

Two series of solutions corresponding to selected points in the curves of titration with sodium hydroxide of the mixture of terephthalic acid with europium or terbium nitrate were prepared (Table 2), and their optical properties were studied, to which end the absorption spectra (Fig. 4), as well as the emission and excitation spectra were measured (Figs. 5 and 6). The absorption spectra of all the prepared solutions are similar in shape; each of them contains an absorption band with a maximum at 250 nm and a shoulder at 290 nm, corresponding to  $\pi$ - $\pi$ \* electronic transitions of the terephthalate ion [22, 23]. The emission spectra of these solutions were measured upon 280-nm excitation into the absorption band of terephthalate ion. The choice of this wavelength was dictated, in particular, by identity of the absorbances of all the solutions at this wavelength

λ, nm

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Fig. 7. Normalized intensity of the peak luminescence upon excitation at a wavelength of 280 nm (1) in comparison with the molar fraction of the terephthalate complexes of (a) europium(III) and (b) terbium(III): (2) Lnbdc<sup>+</sup> and (3) Lnbdc<sup>-</sup><sub>2</sub> (Ln = Eu, Tb).

(0.15), which eliminates the inner filter effect. Analysis of the emission spectra of all the synthesized samples showed that an antenna effect was accomplished via 280-nm excitation into the absorption band of terephthalate ion, leading to the characteristic luminescence of Eu<sup>3+</sup> ions  $({}^{5}D_{0} \rightarrow {}^{7}F_{n}$  transitions) or Tb<sup>3+</sup> ions  $({}^{5}D_{4} \rightarrow {}^{7}F_{m}$  transitions). The luminescence bands of the solutions are associated with the following radiative transitions of terbium and europium ions:  ${}^{5}D_{0} \rightarrow {}^{7}F_{1}$  (591 nm),  ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$  (615 nm),  ${}^{5}D_{0} \rightarrow {}^{7}F_{3}$  (649 nm), and  ${}^{5}D_{0} \rightarrow {}^{7}F_{4}$  (696 nm) transitions in the case of the solutions containing europium(III) ions (Fig. 5a) and  ${}^5D_4 \rightarrow {}^7F_6$  (489 nm),  ${}^5D_4 \rightarrow {}^7F_5$ (543 nm),  ${}^{5}D_{4} \rightarrow {}^{7}F_{4}$  (584 nm), and  ${}^{5}D_{4} \rightarrow {}^{7}F_{3}$  (619 nm) in the case of the solutions containing terbium(III) ion (Fig. 6a). Broad bands in the excitation spectra (Figs. 5b and 6b) are associated with  $\pi - \pi^*$  transitions of the terephthalate ion, indicating that, in the terephthalate complexes, no direct electronic transition to the excited state of the europium and terbium ions occurs; rather, the antenna effect mechanism operates, with terephthalate ion acting as antenna.

Figure 7 presents the fractions of the europium(III) and terbium(III) terephthalate complexes for the studied solutions in comparison with the relative peak luminescence intensities [ $(\lambda_{exc} = 280 \text{ nm and } \lambda_{em} = 615 \text{ and } 543 \text{ nm for the solutions containing europium(III)}$  and terbium(III) ions, respectively] against those for the solutions, exhibiting the maximum luminescence intensity in the series (E<sub>4</sub> and T<sub>4</sub>). The obtained curves exhibit identical shapes, and solutions E<sub>4</sub> and

 $T_4$  characterized by maximal fractions of the Lnbdc<sup>+</sup> and Ln(bdc)<sub>2</sub><sup>-</sup> complex species display a maximum intensity of *f*-*f* luminescence of the europium(III) and terbium(III) ions upon excitation in the absorption band of the terephthalate ion. This observation further confirms the correctness of the complexation model proposed by us for the Eu(NO<sub>3</sub>)<sub>3</sub>-H<sub>2</sub>bdc-NaOH and Tb(NO<sub>3</sub>)<sub>3</sub>-H<sub>2</sub>bdc-NaOH systems under study.

# CONCLUSIONS

The stability constants of the complexes of europium(III) and terbium(III) ions with terephthalic acid (H<sub>2</sub>bdc) anions were determined by means of potentiometric titration over a wide pH range. The luminescent properties of these complexes were studied in the systems Eu(NO<sub>3</sub>)<sub>3</sub>-H<sub>2</sub>bdc-NaOH and Tb(NO<sub>3</sub>)<sub>3</sub>-H<sub>2</sub>bdc-NaOH. Equilibrium modeling under the assumption of the coexistence of various complex species [Lnbdc<sup>+</sup>, Ln(bdc)<sub>2</sub><sup>-</sup>, Ln(OH)<sup>2+</sup>, Ln(OH)<sub>2</sub><sup>+</sup>, and Ln(OH)<sub>3</sub>] in solution and calculation of their stability constants were carried out in the ReactLab pH PRO program. Analysis of the distribution of the complex species showed that, with increasing pH, the dominant complex species changes in the series  $Ln^{3+}-Ln(1,4-bdc)^{+}$ ,  $Ln(1,4-bdc)_{2}^{-}-Ln(OH)^{2+}-Ln(OH)_{2}^{+}-Ln(OH)_{3}$ . Thus, the slightly acidic medium (pH 4-6.5) is preferred for the formation of terephthalate complexes of europium(III) and terbium(III); with increasing pH the terephthalate complexes undergo destruction to form stable hydroxo complexes of the lanthanides. Based on analysis of the

titration curves, the overall formation constants of the complexes of europium(III) and terbium(III) ions with terephthalate ion were calculated to be  $1.6 \times 10^8$ ,  $10^{14}$ ,  $3.2 \times 10^7$ , and  $1.6 \times 10^{13}$  for Eu(1,4-bdc)<sup>+</sup>, Eu(1,4-bdc)\_2^-, Tb(1,4-bdc)^+, and Tb(1,4-bdc)\_2^-, respectively.

For two series of solutions, corresponding to selected points in the curves of titration with sodium hydroxide of a mixture of terephthalic acid with europium or terbium nitrate, the absorption, emission and excitation spectra were measured. The absorption spectra of all the prepared solutions have similar shapes. Analysis of the emission spectra of all the synthesized samples revealed accomplishment of an antenna effect upon 280-nm excitation into the absorption band of terephthalate ion, leading to the characteristic luminescence of Eu<sup>3+</sup> ions  $({}^{5}D_{0} \rightarrow {}^{7}F_{n}$  transitions) or Tb<sup>3+</sup> ions  $({}^{5}D_{4} \rightarrow {}^{7}F_{m}$ transitions). For the solutions studied, the fractions of the europium(III) and terbium(III) terephthalate complex were presented in comparison with the relative peak luminescence intensities against those for the solutions exhibiting the maximum luminescence intensities in the studied series. Identical shapes of the obtained curves confirm the correctness of the complexation model proposed by us for the Eu(NO<sub>3</sub>)<sub>3</sub>-H<sub>2</sub>bdc-NaOH and Tb(NO<sub>3</sub>)<sub>3</sub>-H<sub>2</sub>bdc-NaOH systems studied.

## EXPERIMENTAL

Complexation in 1 : 8  $Ln^{3+}-H_2bdc$  systems (where  $Ln^{3+} = Eu^{3+}$ ,  $Tb^{3+}$  is lanthanide ion, and  $H_2bdc$ , terephthalic acid) was studied by potentiometric titration using a combination glass electrode with an amplifier (LPP014, LPP Co., Ltd, China), connected to an iCP12 USB oscilloscope (PICcircuit, Malaysia), at the temperature that was kept constant at 25°C with a BHS-1 liquid thermostat (JoanLab, China). The electrode was calibrated using sets of buffer solutions. The sample was a 50-mL solution prepared by mixing appropriate aliquots of solutions of terephthalic acid, lanthanide nitrate, and background electrolyte (KNO<sub>3</sub>); the analytical concentrations of the components were 0.194, 0.024, and 100 mM, respectively. Since preliminary point-bypoint titration did not reveal pH instability areas, it was assumed that equilibrium in the system was established quickly. Therefore, titration was carried out with a freshly prepared 0.01 M NaOH solution at a feed rate of the titrant of 3 mL/min using a QHZS-001B syringe pump (LERORA, China).

Solutions were prepared using europium nitrate pentahydrate (99.9%), terbium nitrate pentahydrate (99.9%) (Khimkraft, Kaliningrad), sodium hydroxide, aqueous ammonia, nickel chloride hexahydrate, potassium nitrate, murexide, Trilon B (Nevareaktiv, St. Petersburg), and terephthalic acid (>98%) (Sigma-Aldrich). All chemicals were used without further purification. Standardization of the solutions of the lanthanide salts was carried out complexometrically using back titration. Specifically, to 0.5 mL of a 0.2-0.3 M solution of the metal analyte, 10 mL of 0.1 N Trilon B, 10-15 mL of ammonia buffer (pH = 10), 30 mL of distilled water, and 0.2 g of murexide were added, whereupon excess Trilon B was titrated with a 0.05 M NiCl<sub>2</sub> solution until the color of the indicator changed from purple to yellow. Standardization of the solutions of terephthalic acid and sodium hydroxide was carried out using pH-metry. To prepare a saturated solution of terephthalic acid, 1 g of H<sub>2</sub>bdc was dissolved in 1 L of 0.1 M KNO<sub>3</sub>, which was followed by cooling to room temperature and filtration.

Equilibrium modeling under the assumption of the coexistence of certain probable complex species in solution and calculation of their stability constants were carried out in the Reactlab pH PRO program [14]. The initial data were the experimentally obtained dependences of pH of the solution on the volume of the added titrant or the degree of titration f, i.e., the number of alkali equivalents per proton of terephthalic acid.

Absorption spectra were measured using an SF-2000 spectrophotometer (OKB Spektr). Luminescence spectra were recorded on a Fluoromax-4 spectrofluorometer (Horiba Jobin Yvon) under identical conditions. Absorption and luminescence spectra were measured in a 1x1 cm quartz cuvette at a temperature of 25°C.

## AUTHOR INFORMATION

Ya.E. Saitov, ORCID: https://orcid.org/0009-0000-9424-0627

Yu.N. Toikka, ORCID: https://orcid.org/0000-0003-2926-0079

N.A. Bogachev, ORCID: http://orcid.org/0000-0002-9495-0669

M.Yu. Skripkin, ORCID: http://orcid.org/0000-0001-9841-150X

A.S. Mereshchenko, ORCID: http://orcid.org/0000-0001-9390-1446

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### CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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