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Synthesis and Structural Diversity of Lanthanum and Neodymium Triphenylacetate Complexes with Trimethyltriazacyclohexane and Crown Ether

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Abstract—The reactions of lanthanum and neodymium triphenylacetates with polydentate 6- and 18-membered N- and O-heterocyclic ligands give various types of mononuclear triphenylacetate complexes containing a polydentate auxiliary ligand (neutral or cationic and anionic): $[Nd(Ph_3CCOO)_3(Me_3TACH)] \cdot CHCl_3$ (**II**), $[La(Ph_3CCOO)_4(Me_3TACH)]^-[NH_4Me]^+$ (**III**), $[La(Ph_3CCOO)_3(18\text{-crown-}6)]$ (**IV**), and $[Nd(Ph_3CCOO)_2(18\text{-crown-}6)]^+[Nd(Ph_3CCOO)_4]^-$ (**V**) and binuclear complex $[La_2(Ph_3CCOO)_3\text{-}(Me_3TACH)] \cdot 2C_6H_5CH_3$ (**I**) (CIF files CCDC no. 1968815 (**I**), 1968813 (**II**), 1968814 (**III**), 1968812 (**IV**), and 1968816 (**V**)). A minor change in the ionic radius, e.g., on going from La to Nd, results in a changed coordination environment. These changes can be followed using macrocyclic polydentate ligands.

Keywords: rare earth elements, polydentate ligands, X-ray diffraction
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INTRODUCTION

The coordination compounds of lanthanides are quite attractive for the development of new luminescent and magnetic materials and are used as contrast agents for magnetic resonance imaging and cell optical imaging [1, 2].

All lanthanides have similar chemical properties because of similar ionic radii, which change by only 15% from the beginning to the end of the 4*f* series. Nevertheless, in the last 10–12 years, examples of different chemical behavior even for lanthanides located near each other in the 4*f* series were reported in the literature [3–5].

Therefore, it is of interest to detect examples of chemical behavior that would result in the formation of different types of coordination compounds for lanthanides with similar ionic radii. This phenomenon is rather due to different electronic configurations of lanthanide ions than to the minor changes in the radii of Ln^{3+} cations. However, the choice of ligands able to form such compounds and thus to demonstrate the desired differences is not obvious.

We assumed that saturated nitrogen- and oxygen-containing heterocyclic compounds containing three to six heteroatoms in the ring may prove to be good polydentate ligands for observing different coordination modes of lanthanide ions and elucidating the differences between the structures of lanthanide complexes, which usually demonstrate a similar type of structural organization.

This communication describes the preparation of lanthanum and neodymium triphenylacetate complexes with 1,3,5-trimethyl-1,3,5-triazacyclohexane (Me_3TACH) and a crown ether (18-crown-6) that implement basically different coordination environments for lanthanum and neodymium cations for the same set of ligands in the complex. The choice of 6- and 18-membered heterocyclic ligands is caused by their different conformational mobility, namely, structural rigidity of Me_3TACH and conformational flexibility of 18-crown-6, which would reveal the structural differences of interest.

EXPERIMENTAL

All experiments were carried out under argon using the Schlenk vessels and an argon vacuum line. Argon was purified by being passed through a column with molecular sieves (4 Å) impregnated with a Na/K eutectic (10 wt %) and a column with Mn(II) oxide supported on mica. Lanthanum and neodymium triphenylacetates were synthesized by a reported procedure [6]. 1,3,5-Trimethyl-1,3,5-triazacyclohexane (Aldrich) was distilled from calcium hydride, degassed, and stored over molecular sieves (4 Å). The organic solvents were distilled under argon, and 18-crown-6 (Aldrich) was dried in a dynamic vacuum prior to use. Hexane was distilled from a Na/K eutectic, toluene was distilled from sodium metal, and chloroform was distilled from calcium hydride. Elemental analysis was carried out on a PerkinElmer 2400 Series II analyzer. Lanthanides were quantified by direct complexometric titration with the Xylenol Orange indicator.

Synthesis of $[La_2(Ph_3CCOO)_6(Me_3TACH)] \cdot C_6H_5CH_3$ (I). A toluene solution of Me_3TACH (1.74 mL, 0.325 M, 0.57 mmol) was added with stirring to $[La(Ph_3CCOO)_3(THF)_3]$ (1.39 g, 1.140 mmol) in anhydrous toluene (20 mL). The reaction mixture was stirred for 2 h and then concentrated to 5 mL, and hexane (20 mL) was added. The precipitate was separated from the solution and dried *in vacuo*. The yield of **I** was 10.200 g (92%).

For $C_{133}H_{113}N_3O_{12}La_2$

Anal. calcd., %	C, 71.85	H, 5.12	N, 1.89	La, 12.50
Found, %	C, 71.51	H, 5.65	N, 1.81	La, 12.25

The crystals suitable for X-ray diffraction were formed upon slow diffusion of hexane into a solution of **I** in toluene. A single crystal of **I** contained three toluene solvate molecules per formula unit. Drying in vacuum was accompanied by removal of two toluene molecules.

Synthesis of $[Nd(Ph_3CCOO)_3(Me_3TACH)] \cdot CHCl_3$ (II). A chloroform solution of Me_3TACH (4.35 mL, 0.164 M, 0.713 mmol) was added with stirring to a solution of $[Nd(Ph_3CCOO)_3(THF)_3]$ (0.83 g, 0.679 mmol) in chloroform (3 mL), the mixture was stirred for 1 h and then hexane (25 mL) was added without stirring. After a week, light blue crystals precipitated in the Schlenk vessel. The yield of **II** was 0.520 g (61%).

For $C_{67}H_{61}N_3O_6Cl_3Nd$

Anal. calcd., %	C, 64.13	H, 4.90	N, 3.35	Nd, 11.50
Found, %	C, 64.17	H, 5.07	N, 3.13	Nd, 11.49

The single crystal of **II** contained one hexane solvate molecule and one chloroform solvate molecule per formula unit. Drying in vacuum was accompanied by the loss of one hexane molecule.

Synthesis of $[La(Ph_3CCOO)_3(18\text{-crown-}6)]$ (IV). A solution of 18-crown-6 (6.62 mg, 0.234 mmol) in toluene (10 mL) was carefully added to a solution of $[La(Ph_3CCOO)_3(THF)_3]$ (0.255 g, 0.209 mmol) in anhydrous toluene (5 mL) in such a way that two layers formed. After 3 days, colorless crystals appeared in the Schlenk vessel. The vessel was kept for 24 h at −12°C. The crystals were separated from the solution by decantation and dried in vacuo. The yield of **IV** was 0.230 g (87%).

For $C_{72}H_{69}O_{12}La$

Anal. calcd., %	C, 68.42	H, 5.59	La, 11.08
Found, %	C, 68.35	H, 5.50	La, 10.98

The single crystal of **IV** contained toluene solvate molecules, which were removed on drying in vacuo.

Synthesis of $[Nd(Ph_3CCOO)_2(18\text{-crown-}6)]^+[Nd(Ph_3CCOO)_4]^-$ (V). A solution of 18-crown-6 (0.400 g, 1.515 mmol) in toluene (10 mL) was added to a solution of $[Nd(Ph_3CCOO)_3THF_3]$ (3.492 g, 2.858 mmol) in toluene (50 mL). A pale blue precipitate formed. The reaction mixture was stirred for 6 h. The precipitated blue crystals were collected on a filter and dried in vacuo. The yield of **V** was 3.227 g (99%).

The crystals of **V** suitable for X-ray diffraction were obtained by slow diffusion of a solution of 18-crown-6 into a chloroform solution of $[Nd(Ph_3CCOO)_3(THF)_3]$.

For $C_{132}H_{114}O_{18}Nd_2$

Anal. calcd., %	C, 69.63	H, 5.05	Nd, 12.67
Found, %	C, 69.89	H, 4.95	Nd, 12.40

The X-ray diffraction study of I–V was carried out on a SMART APEX 2 CCD diffractometer (MoK_α radiation, graphite monochromator, ω-scan mode). The structures were solved by direct methods and refined by the least-squares method in the full-matrix anisotropic approximation on F^2_{hkl}. The absorption corrections were applied semiempirically on the basis of equivalent reflections. The disordered groups were refined using constraints for atomic displacement parameters and positional parameters (DFIX and EADP). In the crystal of **V**, a highly disordered solvate molecule is located in one of the voids (the observed electron density distribution corresponds to superposition of $CHCl_3$ and toluene), whose contribution was eliminated from structure amplitudes by the SQUEEZE procedure [7]. The hydrogen atoms in all structures were calculated and refined by the riding model. All calculations were carried out using the SHELXL-2014/2017 software package [8]. Selected crystallographic data and structure refinement parameters for compounds **I**–**V** are summarized in Table 1.

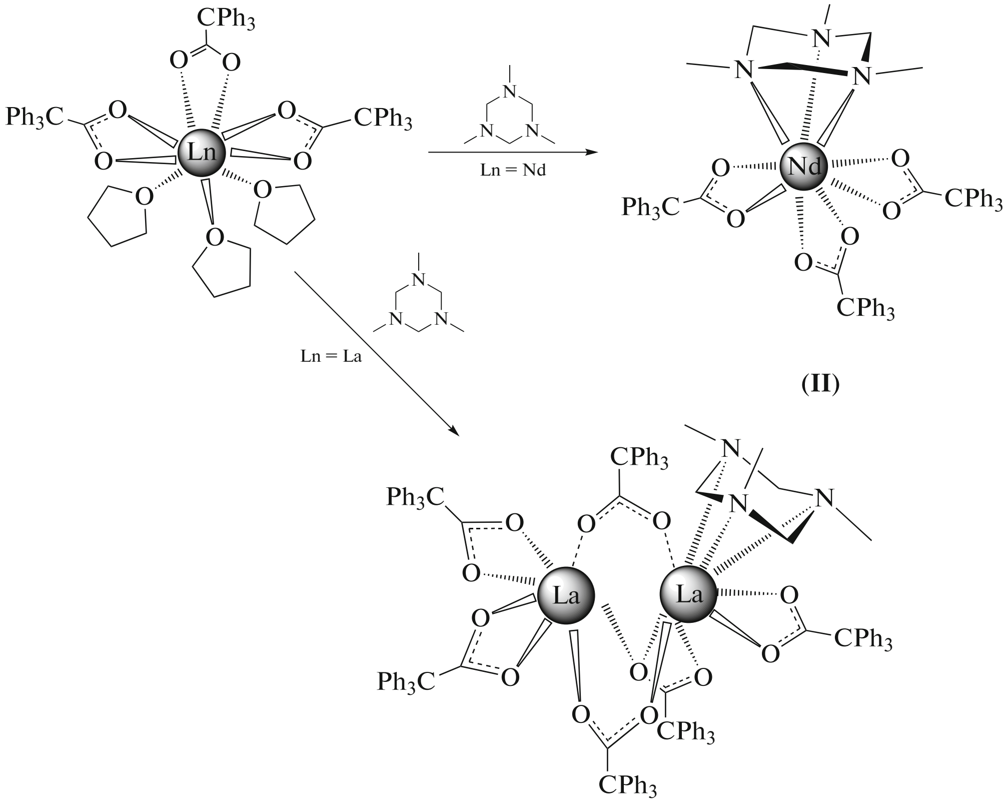
The atomic coordinates and other structure parameters were deposited with the Cambridge Crystallographic Data Centre (nos. 1968815 (**I**), 1968813 (**II**), 1968814 (**III**), 1968812 (**IV**), 1968816 (**V**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

Table 1. Selected crystallographic data and refinement parameters for compounds **I–V**

Parameter	Value				
	I	**II**	**III**	**IV**	**V**
Molecular formula	$C_{147}H_{129}N_3O_{12}La_2$	$C_{73}H_{75}N_3O_6Cl_3Nd$	$C_{87}H_{81}N_4O_8La$	$C_{88.93}H_{88.34}O_{12}La$	$C_{536}H_{464}O_{72}Cl_{24}Nd_8$
M	2407.34	1340.95	1449.46	1488.08	5030.88
T, K	100	100	100	100	100
System	Triclinic	Monoclinic	Monoclinic	Triclinic	Tetragonal
Space group	$P\bar{1}$	$P2_1/n$	$P2_1/n$	$P\bar{1}$	$I4_1/acd$
Z	2(1)	4(1)	4(1)	2(1)	
a, Å	14.5772(7)	8.244(2)	15.3326(11)	14.1523(9)	40.394(6)
b, Å	18.0163(9)	50.227(15)	26.8157(19)	15.3957(10)	40.394(6)
c, Å	24.1812(12)	15.742(4)	17.4929(12)	18.6036(12)	30.888(6)
α, deg	77.8907(9)	90	90	69.8082(11)	90
β, deg	75.2585(9)	95.856(10)	94.489(2)	87.5567(13)	90
γ, deg	77.4582(9)	90	90	73.4724(12)	90
V, Å^3	5914.2(5)	6484(3)	7170.2(9)	3640.2(4)	50399(17)
ρ(calcd.), g cm^{-3}	1.352	1.374	1.343	1.358	1.326
μ, cm^{-1}	7.78	9.79	8.31	6.52	10.05
F(000)	2484	2772	3008	1550	20576
$2\theta_{max}$, deg	58	58	58	58	52
Number of measured reflections	72493 (0.0413)	44111 (0.035)	88441 (0.0932)	43433 (0.0583)	151568 (0.1198)
Number of unique reflections	31369	14155	19051	19272	12382
Number of reflections with $I > 2\sigma(I)$	25064	12604	13522	14126	6191
GOOF	1.019	0.971	1.005	0.943	1.008
Number of refined parameters	1457	796	909	827	600
R, wR_2 ($I > 2\sigma(I)$)	0.0301, 0.0690	0.0549, 0.1049	0.0400, 0.0804	0.0543, 0.1365	0.0850, 0.1841
R, wR_2 (for all reflections)	0.0453, 0.0769	0.0652, 0.1091	0.0724, 0.0942	0.0877, 0.1625	0.1739, 0.2250
Residual electron density (min/max), e Å^{-3}	–0.745/1.290	–1.529/0.784	–0.885/0.776	–1.665/1.097	–0.984/0.875

RESULTS AND DISCUSSION

The reactions of lanthanum and neodymium triphenylacetates with Me_3TACH result in the formation of binuclear complex **I** in the case of lanthanum and mononuclear complex **II** in the case of neodymium (Scheme 1). The use of excess Me_3TACH (2 equiv.) in the reaction with lanthanum triphenylacetate did not change the reaction outcome.



Scheme 1.

The structures of **I** and **II** were established by X-ray diffraction (Fig. 1, Table 2). In the binuclear complex **I**, only one lanthanum cation is coordinated to Me_3TACH, two metal cations are linked by three Ph_3CCOO^- bridging ligands, two of which are coordinated in the μ_2:^{1}-κ^1 mode, and the other one is coordinated in the μ_2:κ^2-κ^1 mode. The La(1) atom (C.N. 9) is additionally coordinated by one κ^2-Ph_3CCOO^- ligand and the Me_3TACH molecule according to the κ^3-type, while La(2) (C.N. 7) is κ^2-coordinated to two terminal triphenylacetate ligands. In the mononuclear complex **II**, the central atom is coordinated by three carboxylate ligands in the κ^2 mode and by Me_3TACH in the κ^3 mode (C.N.(Nd) = 9). The Nd–O (carboxylate ligand) distances in complex **II** are comparable with those observed in the mononuclear triphenylacetate $[Nd(Ph_3CCOO)_3$-$(THF)_3]$ [6].

In complexes **I** and **II**, coordinated Me_3TACH exists in the chair conformation, with the Ln–N(Me_3TACH) distances being comparable with those observed in the previously known $[Pr(CH(AlMe_3)_3)$-$(Me_3TACH)]$ [3] and $[(Sm(AlMe_4)_2$(*iso*-Pr_3TACH$)_2]$ [9] complexes.

Presumably, for both metals (Nd and La), a mononuclear complex is formed initially, but in the case of lanthanum, this complex is unstable because of the larger ionic radius. Upon the formation of the mononuclear adduct, the lanthanum coordination sphere is unsaturated, and further interaction with the initial lanthanum triphenyl acetate gives rise to the binuclear complex. Apparently, another way of destruction of the hypothetical mononuclear lanthanum complex $[La(Ph_3CCOO)_3(Me_3TACH)]$ is hydrolysis induced by traces of water in the solvent. Hydrolysis affects the coordinated triazacyclohexane ligand and results in the formation of compound **III**. Evidently, the methylammonium cation is generated via protonation of methylamine formed upon the hydrolysis of Me_3TACH. Several crystals of **III** suitable for X-ray diffraction were isolated from solutions of **I** in toluene (Fig. 2).

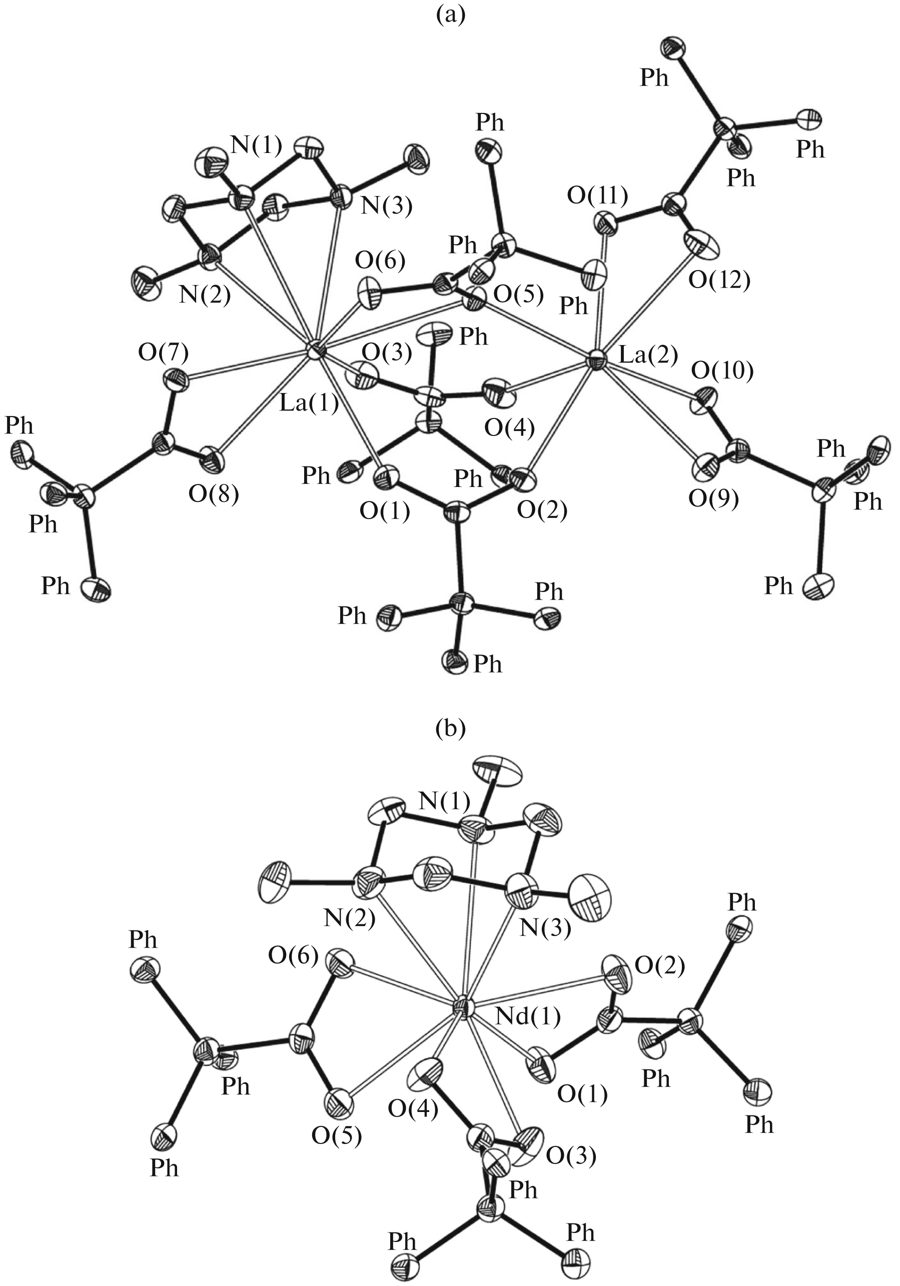


Fig. 1. Structures of complexes (a) **I** and (b) **II**. The hydrogen atoms and phenyl groups are omitted.

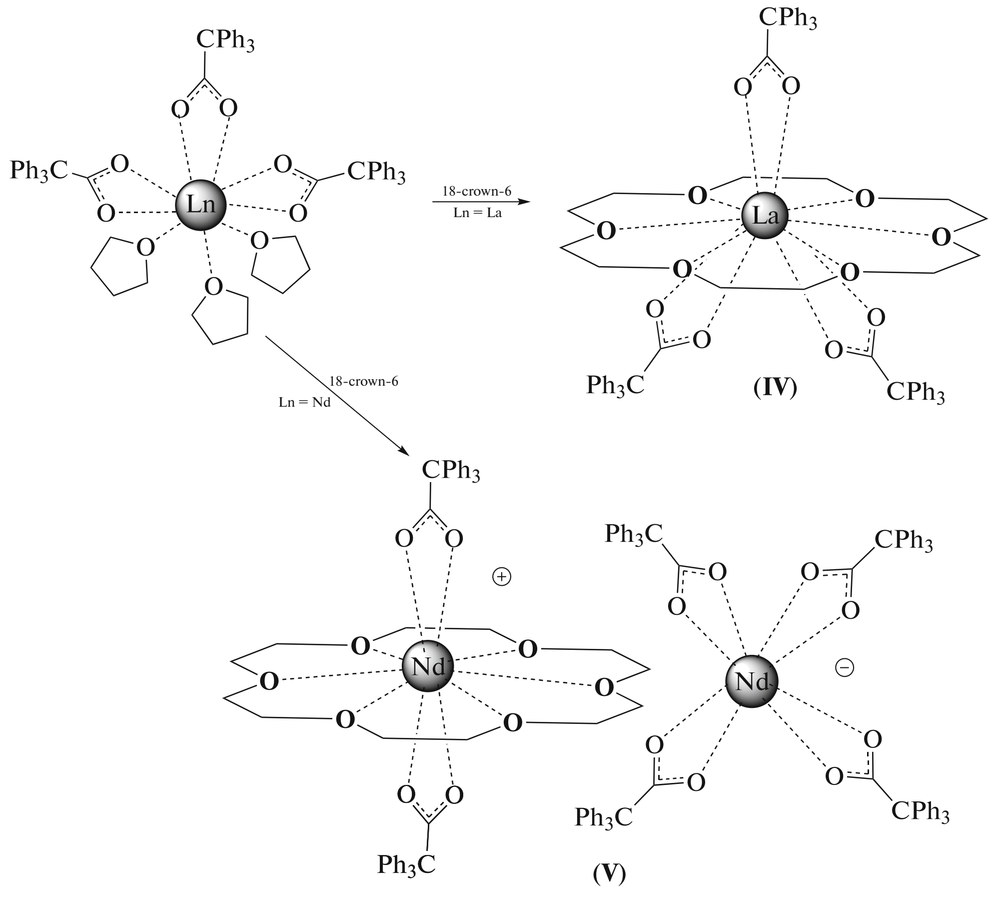
Complex **III** is a mononuclear ate-complex in which the lanthanum cation is coordinated by four terminal triphenylacetate ligands and one Me_3TACH molecule. The Ln–O (κ^2-Ph_3CCOO) and Ln–N (Me_3TACH) distances are longer than those in **I** by, on average, 0.12 and 0.07 Å, respectively (Table 2). This elongation of the distances is associated with the change in C.N.(La^{3+}) in **III** to 11. The $[NH_3Me]^+$ counter-ion forms hydrogen bonds with the carboxylate oxygen atoms. In view of the fact that the Ln-containing anion (proton acceptor) has an approximate C_4 symmetry and the $CH_3NH_3^+$ cationic group (proton donor) has a C_3 symmetry, the ammonium cation in the crystal is disordered over two positions, which form NH···O bonds (2.67–2.85 Å). The formation of a similar trifluomethanesulfonate ate-complex $[Pr_2(CF_3SO_3)_7(Et_3TACH)_2]^-[EtNH_3]^+$, which was isolated as a minor product in the synthesis of $[Pr(CF_3SO_3)_3(Et_3TACH)]$, was reported in [10].

The reaction of lanthanum and neodymium triphenylacetates with 18-crown-6, in which the ring size is much greater than that of trimethyltriazacyclohexane,

Table 2. Selected parameters of structures **I–III**

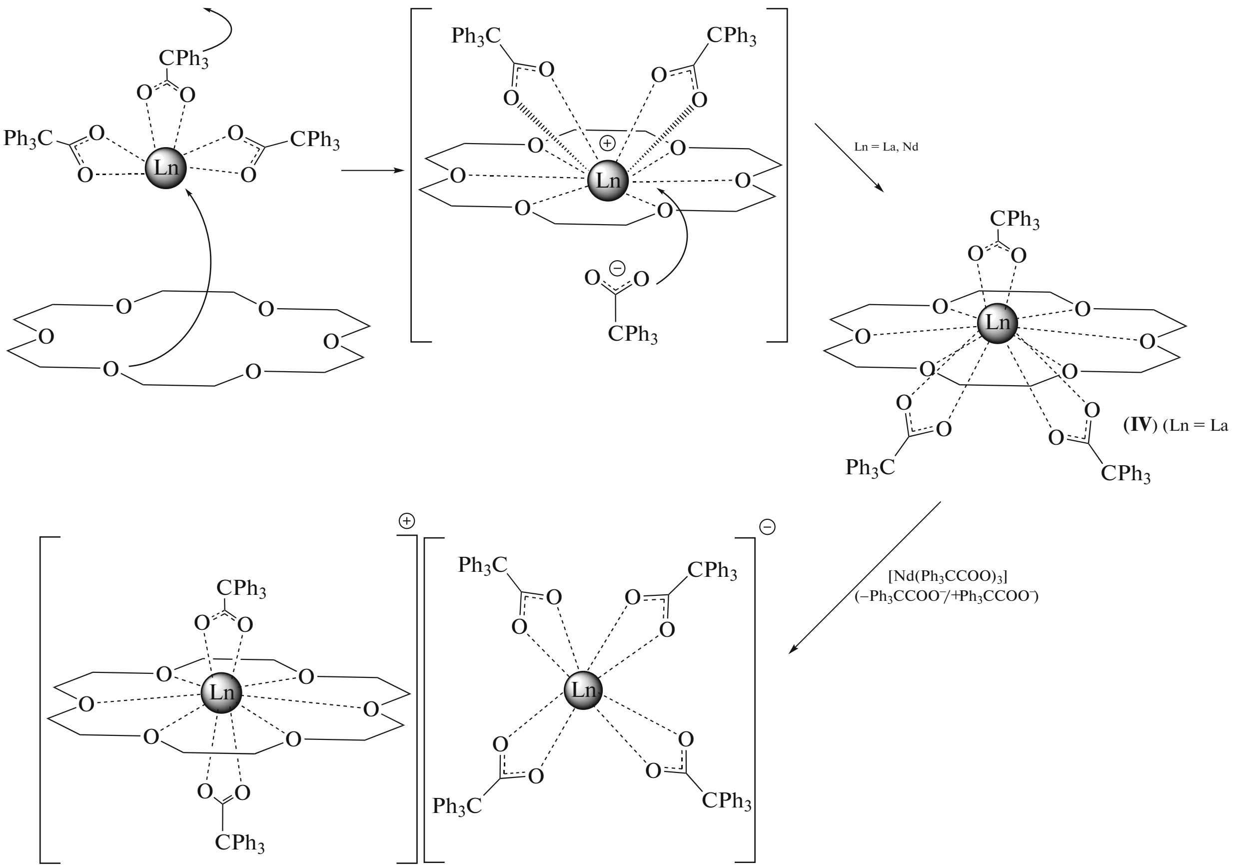
Parameter	**I** (La(1))	**I** (La(2))	**II** (Nd)	**III** (La)
Ln–O (κ^2-Ph_3CCOO)	2.5084(13)– 2.5289(13)	2.4618(14)–2.5490(14)	2.453(3)– 2.505(3)	2.570(1)–2.692(1)
Ln–O (μ_2-κ^1:κ^2-Ph_3CCOO)	2.5179(13), 2.7680(13)	2.5294(13)		
Ln–O (μ_2-κ^1:κ^1-Ph_3CCOO)	2.4357(14)–2.4793(14)	2.3781(14), 2.4562(13)		
Ln–N	2.7435(16)– 2.7580(17)		2.670(4)–2.692(2)	2.764(2)–2.880(2)
Ln···Ln	4.524			

also proved to be sensitive to the lanthanide ionic radius. The reaction of 18-crown-6 with lanthanum or neodymium triphenylacetate affords complexes **IV** and **V** insoluble in toluene (Scheme 2). Compound **IV** (metal to ligand molar ratio of 1 : 1) is formed in the case of lanthanum, while complex **V** (2 : 1 ratio) is formed for neodymium. The use of an excess of 18-crown-6 (up to 2.5 equiv.) in the reaction with neodymium triphenylacetate also results in the formation of compound **V**.



Scheme 2.

It is evident that 18-crown-6 coordination to the metal is possible only upon dissociation of the complex with detachment of one carboxylate ligand, because the triphenylacetate ligand is too large to fit in the crown ether cavity (Scheme 3). Presumably, complex **IV** is formed initially. In the case of La (C.N. 12), which has a smaller ionic radius than neodymium, this complex is rather stable. The Nd complex is coordinatively or sterically congested and undergoes metathesis to give the $[Nd(Ph_3CCOO)_2(18\text{-crown-}6)]^+$ complex cation and $[Nd(Ph_3CCOO)_4]^-$ complex anion and, hence, complex **V** is formed as a contact ion pair.



(**IV**) (Ln = La

(**V**) (Ln = Nd)

Scheme 3.

This process (dissociation of the complex) in toluene, which has a low relative dielectric permittivity (ε = 2.4), was unexpected [11]. Usually, crown ether complexes are prepared in media with high dielectric permittivity. For example, previously known $[Eu(NO_3)_2(\text{dicyclohexyl-}18\text{-crown-}6)_2]_2[Eu(NO_3)_5]$ was obtained in ethanol [12], while $[LaCl_3(18\text{-crown-}6)]$ and $[Ln(NO_3)_3(18\text{-crown-}6)]$ (Ln = Ce, Pr, Nd) were formed in an acetonitrile–methanol mixture [13–15].

The structure of **IV** and **V** was established by X-ray diffraction. The lanthanum complex **IV** is composed as a mononuclear complex, with the triphenylacetate ligands being located above and below the crown ether ring and the central lanthanum cation being coordinated by three Ph_3CCOO^- ligands in the κ^2-mode and by 18-crown-6 in the κ^6-mode (Fig. 3, Table 3). The distance from La to one triphenylacetate ligand is markedly longer than the distances to the other two ligands, while the La–O (18-crown-6) distances are in the 2.596(2)–2.781(2) Å range (Table 3). The structure of complex **IV** is similar to that of $[La(NO_3)_3(18\text{-crown-}6)]$ [16]; however, in the latter complex, the $Ln\text{–}O(NO_3^-)$ distances are equal and the Ln–O(18-crown-6) distances are in the 2.662–2.780 Å range. It is clear that these differences are attributable to repulsion of the phenyl groups of the triphenylacetate and macrocyclic ligands.

Complex **V** is a contact ion pair (Fig. 4). The $[Nd(Ph_3CCOO)_4]^-$ anionic moiety has C_2 symmetry and is shaped like a highly flattened tetrahedron (the dihedral angle is 36°) with the vertices being occupied by κ^2-coordinated Ph_3CCOO^- ligands. In the centrosymmetric moiety of **V**, the Nd cation is coordinated by two κ^2-Ph_3CCOO^- ligands and by 18-crown-6. The Nd–O(carboxylate ligand) distances (Table 3) are comparable with those found for $[Nd(Ph_3CCOO)_3(THF)_3]$ [6].

It was interest to compare the 18-crown-6 conformations in **IV** and **V**. For example, in complex **V**, the conformation of 18-crown-6 is close to that observed in the $[Nd(NO_3)_2(18\text{-crown-}6)]_3^+[Nd(NO_3)_6]^{3-}$ nit-

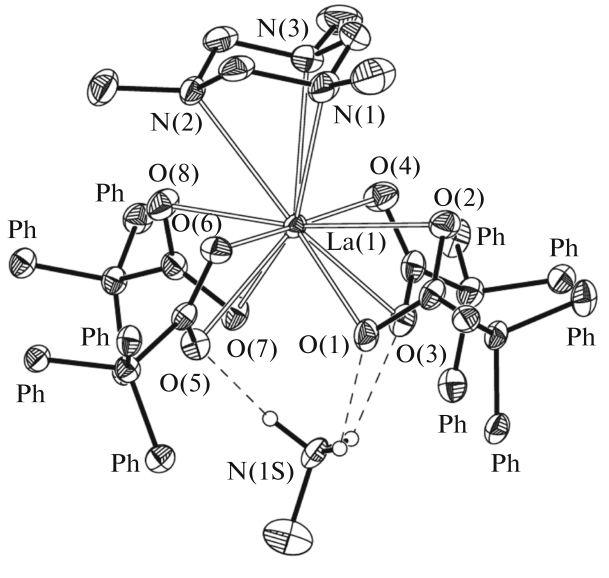


Fig. 2. General view of complex **III**. Only one position of the $CH_3NH_3^+$. cation is shown. The hydrogen atoms and the carbon atoms of phenyl groups are omitted.

rate complex [14] and considerably differs from that observed in **IV**. The crown ether oxygen atoms in **V** form a nearly planar six-member ring, being deviated from the plane by not more than 0.05 Å. In **IV**, the arrangement of crown ether oxygen atoms resembles a bowl conformation. Apparently, distortion of the 18-crown-6 structure is caused by higher steric strain present in **IV** in comparison with **V** as a result of location of two triphenylacetate ligands on the same side of the macrocycle.

Previously, lanthanide nitrate adducts with 18-crown-6 were prepared, namely, $[Ln(NO_3)_3(18\text{-crown-6})]$ [15, 17] (M : L = 1 : 1) and $[Ln(NO_3)_2(18\text{-crown-6})]_3^+[Ln(NO_3)_6]^{3-}$ (Ln = La, Nd) [14, 17] (M : L = 4 : 3). In the case of lanthanum, only the former complex is produced, while in the case of neodymium, a mixture of two complexes is formed, in which the former complex can be converted to the latter one on heating.

Presumably, 1 : 1 adduct with the 12-coordinate neodymium cation, analogous to lanthanum complex **IV**, is unstable because of steric congestion; therefore, a less strained structure of complex **V** with 10- and 8-coordinate neodymium cations is implemented.

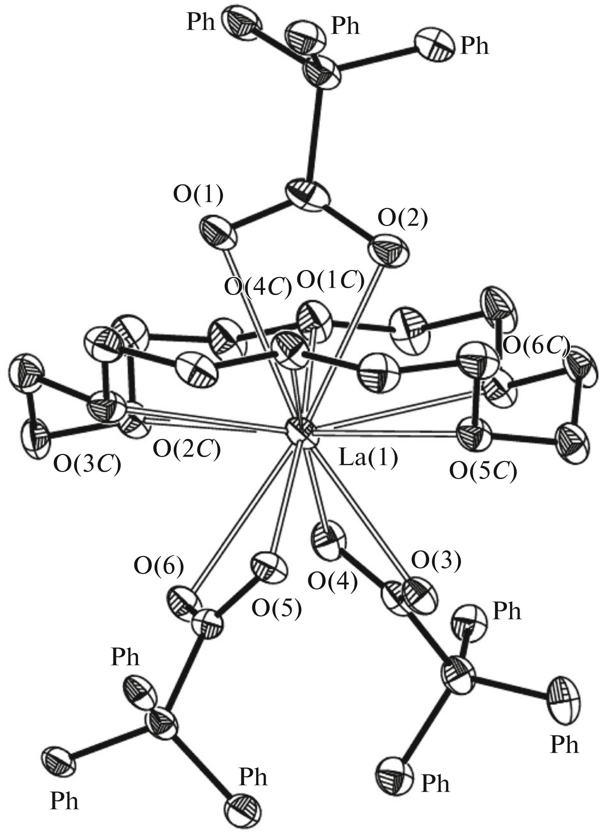


Fig. 3. Structure of complex **IV**. The hydrogen atoms and the carbon atoms of phenyl groups are omitted.

Thus, in this study, we prepared the triphenylacetate lanthanum and neodymium complexes with 6- and 18-membered saturated heterocyclic ligands. The obtained compounds of La(III) and Nd(III) (whose ionic radii differ by not more than 4%) containing the same set of ligands have fundamentally different structures.

FUNDING

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Table 3. Selected parameters of structures **IV** and **V**

Parameter	**IV** (La)	**Va** (Nd(1))*	**Vb** (Nd(2))*
Ln–O (κ^2-Ph_3CCOO)	2.596(2)–2.781(2)	2.43(1)–2.46(1)	2.34(1)–2.47(1)
Ln–O (18-crown-6)	2.6783(2)–2.873(2)	2.598(6)–2.693(6)	2.51(1)–2.80(1)
Ln···Ln		7.789	
C.N.(Ln^{3+})	12	10	8

* **Va** and **Vb** correspond to the cationic and anionic parts of complex **V**, respectively.

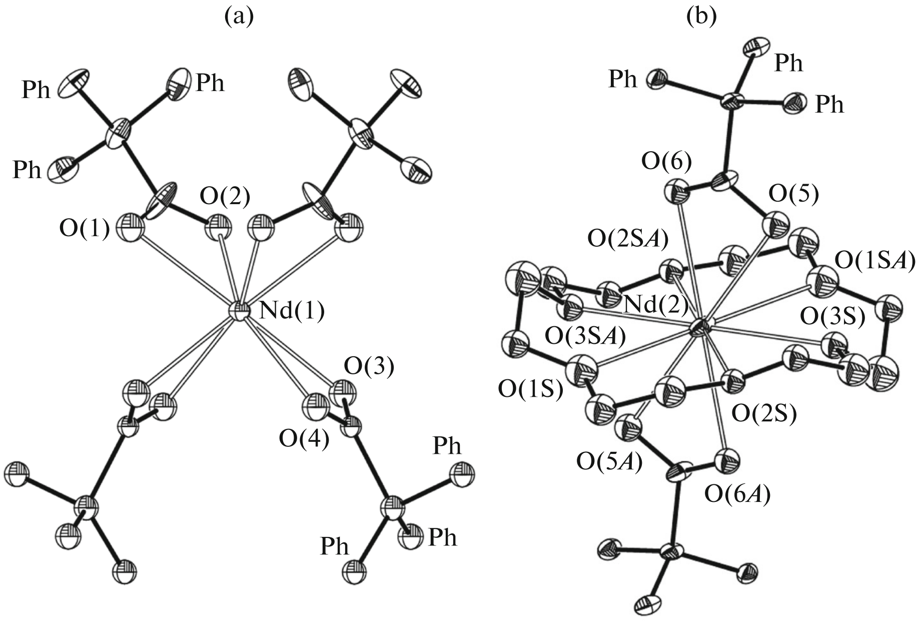

Fig. 4. (a) Cationic and (b) anionic moieties of the crystal of **V**. The disorder is not shown.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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