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Acetylcarbamide Complexes of Some Lanthanide Bromides and Chlorides: Syntheses and Structures

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Abstract—Syntheses, IR spectroscopy, and X-ray diffraction analyses were carried out for acetylcarbamide (AcUr) and new complexes $La(AcUr)_2(H_2O)_5]Br_3$ (**I**), $[Pr(AcUr)_2(H_2O)_5]Br_3$ (**II**), $[Nd(AcUr)_2(H_2O)_5]Cl_3$ (**III**), and $[Sm(AcUr)_2(H_2O)_5]Cl_3$ (**IV**) (CIF files CCDC 992841 (AcUr), 992842 (**I**), 992844 (**II**), 992843 (**III**), and 992845 (**IV**)). The crystals of compounds **I–IV** contain complex cations $[Ln(AcUr)_2(H_2O)_5]^{3+}$ (coordination number of lanthanum is 9) and uncoordinated halide ions. The coordination polyhedron in the cases of compounds **I** and **IV** is a one-capped tetragonal antiprism, and it is a three-capped trigonal prism in the cases of compounds **II** and **III**. Molecules AcUr are coordinated to the Ln atoms via the bidentate mode through the oxygen atoms. They are transformed from the initial *cis–trans* conformation to the *trans–trans* conformation, which is due to the rotation of the $–C(O)–NH_2$ fragment around the nitrogen atom of the imino group. The angles between the O(1)C(1)N(3) and O(2)C(4)N(3) planes in the coordinated AcUr molecules are different, which can be due to the electronic structure of the central atom and spatial characteristics of the complexes. The coordinated AcUr molecules are joined to the halide ions and water molecules of the adjacent complex cations by hydrogen bonds.

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INTRODUCTION

Lanthanide complexes are used for the preparation of materials required by various areas of novel technology (catalysts, X-ray protectors, etc.). Some of them can serve as precursors in the synthesis of new biologically active compounds, in particular, ureide derivatives (acyl derivatives of carbamide). Therefore, the syntheses and structures of complexes of the simplest ureide (acetylcarbamide) used in plant cultivation as an insecticide are of interest, since the obtained data help to predict the structures and properties of complexes of more complicated ureides. Acetylcarbamide $CH_3CONHCONH_2$ (AcUr) can be coordinated to complexing atoms as a polydentate ligand, since its molecule contains two carbonyl groups, one amide group, and one imide group.

The studies of the interaction of lanthanide salts with acetylcarbamide have started more than 30 years ago. Complexes $LnBr_3 \cdot 2AcUr \cdot 4H_2O$ (Ln = La [1], Ce [2]) and $YbBr_3 \cdot 3AcUr$ [3] were obtained by crystallization from aqueous solutions. The acetylcarbamide derivatives of lanthanide perchlorates $Ln(ClO_4)_3 \cdot 4AcUr$ (Ln = La–Yb, Y) [4] were synthesized in a nonaqueous medium. The bidentate coordination mode of the ligand through the oxygen atoms was established by the data of IR spectroscopy. It is found by X-ray diffraction analyses that crystalline compounds $Ln(ClO_4)_3 \cdot 4AcUr$ form two isomorphic series (La–Pr and Nd–Yb, Y). However, the structures of the complexes were not studied in more detail.

The structure of crystalline acetylcarbamide was mainly studied by IR and Raman spectroscopies. Based on the character of the influence of the alkyl fragment on the bands corresponding to the imide group in the spectra of AcUr and its deuterated and alkyl analogs, the *cis–trans* conformation of the CO groups in an AcUr molecule with respect to the imide fragment was concluded [5, 6]. The X-ray diffraction study of the structure of AcUr made it possible to determine the unit cell parameters: $a = 7.07$, $b = 5.14$, $c = 13.74$ Å, $\beta = 96.3°$; $V = 496$ Å^3; $Z = 4$ [7].

The purpose of this work is to synthesize acetylcarbamide chlorides and bromide complexes of some light lanthanides using lanthanum and praseodymium bromides and neodymium and samarium chlorides as examples and to study their structures.

EXPERIMENTAL

The initial substances were lanthanum, praseodymium, neodymium, and samarium carbonates (reagent grade); hydrochloric and hydrobromic acids (analytical grade); and acetylcarbamide (high-purity grade). The additional purification of AcUr was carried out by recrystallization from ethanol. Crystalline hydrates of lanthanum, praseodymium, neodymium, and samarium chlorides and bromides were prepared by the reactions of the corresponding carbonates with concentrated hydrobromic or hydrochloric acids using a described procedure [8]. The content of a rare-earth element in crystalline hydrates of lanthanide halides was monitored trilonometrically [9].

The acetylcarbamide complexes were synthesized by mixing lanthanide halides with AcUr in a mole ratio of 1 : 2, 1 : 4, and 1 : 6. Ethanol (3–5 mL per g of lanthanide halide) was added to the reaction mixture, due to which the mixture was homogenized and the crystallization and the complexes was further accelerated. After a prolonged (for 15–60 days) storage in air, prismatic light green (Pr), light yellow (Sm), light violet (Nd), and colorless (La) crystals precipitated from the solutions corresponding to the mole ratio 1 : 2. We failed to isolate crystalline compounds corresponding to the mole ratios 1 : 4 and 1 : 6.

Trilonometric titration was used to determine the lanthanide content in the synthesized complexes [9]. Analyses to C, H, and N were carried out on a CHNSFlashEA1112 elemental analyzer (ThermoFinnigan, Italy). The determination error was 0.2–0.3% for C, H, and N. The results of analyses of the obtained complexes showed that they contained 2 moles of AcUr and 5 moles of water per mole of lanthanide halide.

For $[La(AcUr)_2(H_2O)_5]Br_3$ (**I**)
anal. calcd., %: C, 10.70; H, 3.27; N, 8.32; La, 20.65.
Found, %: C, 11.40; H, 3.28; N, 8.63; La, 18.35.

(mole ratio La : AcUr = 1 : 2.33).

For $[Pr(AcUr)_2(H_2O)_5]Br_3$ (**II**)
anal. calcd., %: C, 10.67; H, 3.26; N, 8.30; Pr, 20.88.
Found, %: C, 10.62; H, 2.96; N, 7.91; Pr, 21.77.

(mole ratio Pr : AcUr = 1 : 1.91).

For $[Nd(AcUr)_2(H_2O)_5]Cl_3$ (**III**)
anal. calcd., %: C, 13.21; H, 4.04; N, 10.28; Nd, 26.47.
Found, %: C, 13.95; H, 4.08; N, 10.57; Nd, 25.26.

(mole ratio Nd : AcUr = 1 : 2.15).

For $[Sm(AcUr)_2(H_2O)_5]Cl_3$ (**IV**)
anal. calcd., %: C, 13.07; H, 3.99; N, 10.16; Sm, 28.14.
Found, %: C, 13.67; H, 4.04; N, 10.54; Sm, 2 6.43.

(mole ratio Sm : AcUr = 1 : 2.14).

The deviations of the contents of the analyzed elements from the theoretical values are observed, because the compounds are hygroscopic and undergo gradual dehydration on keeping above a drying agent.

Table 1. Frequencies of maxima of the main absorption bands (cm^{-1}) in the IR spectra of AcUr and complexes **I**–**IV**

AcUr	**I**	**II**	**III**	**IV**	Assignment of bands
3377 3356 3213	3383 3221	3390 3199	3381 3212	3382 3214	ν(OH) + ν(NH)
1709 1691 1668	1719 1677 1624	1715 1674 1617	1716 1629	1718 1677 1630	ν(CO) + δ(NH_2) + δ(HOH)
1098	1041	1041	1040	1040	ρ(NH_2)
943	930	932	938	936	ν_s(CN)

The IR absorption spectra of AcUr and complexes **I**–**IV** were recorded on an EQUINOX 55 FT-IR spectrometer (BRUKER, Germany) in the range from 400 to 4000 cm^{-1}. The samples were prepared as suspensions in Nujol. The frequencies of the main absorption bands are given in Table 1.

X-ray diffraction analyses for AcUr and complexes I–IV. The experimental intensities of diffraction reflections were obtained at room temperature (293(2) K, StoeStadiVariPilatus, CuK_α or MoK_α radiation).

Crystallographic calculations were performed using the WinGX [10] and SHELX-97 [11] program packages. The crystal structures of the synthesized compounds were determined by direct methods followed by the refinement of positional and thermal parameters in the anisotropic approximation for all non-hydrogen atoms. The hydrogen atoms were introduced in the calculated positions and refined in the isotropic approximation by the riding method.

The main experimental parameters and chrystallographic characteristics of AcUr and compounds **I**–**IV** are given in Table 2. The structures of the compounds obtained using the MERCURY CSD 1.5 program [12] are shown in Figs. 1–3.

The atomic coordinates and other parameters for the crystal structures of the studied compounds were deposited with the Cambridge Crystallographic Data Centre (CCDC 992841 (AcUr), 992842 (**I**), 992844 (**II**), 992843 (**III**), and 992845 (**IV**), http://www.ccdc.cam.ac.uk, data_request@ccdc.cam.ac.uk).

Table 2. Crystallographic characteristics and the experimental and refinement details for the structures of AcUr and complexes **I**–**IV**

Parameter	Value				
	AcUr	**I**	**II**	**III**	**IV**
Empirical formula	$C_3H_6N_2O_2$	$C_6H_{22}Br_3LaN_4O_9$	$C_6H_{22}Br_3N_4O_9Pr$	$C_6H_{22}Cl_3N_4NdO_9$	$C_6H_{22}Cl_3N_4O_9Sm$
FW	102.09	672.92	674.92	544.92	551.04
Crystal system	Monoclinic	Monoclinic	Monoclinic	Triclinic	Monoclinic
Space group	$P2_1$	$C2$	$C2/c$	$P\bar{1}$	$C2$
a, Å	6.8349(8)	18.490(2)	37.015(4)	7.8149(5)	17.9401(17)
b, Å	5.0988(5)	7.8164(5)	7.0708(7)	8.1230(6)	7.4898(6)
c, Å	6.9855(8)	7.7001(9)	19.0533(17)	16.7713(12)	7.6043(7)
α, deg	90	90	90	78.579(6)	90
β, deg	98.104(10)	113.159(8)	116.734(7)	83.883(6)	114.035(8)
γ, deg	90	90	90	70.157(5)	90
V, Å^3	241.01(5)	1023.20(18)	4453.7(7)	980.71(12)	933.18(14)
T, K	293(2)	293(2)	293(2)	293(2)	293(2)
Z	2	2	8	2	2
ρ_{calcd}, g/cm^3	1.407	2.184	2.013	1.845	1.961
Radiation	CuK_α	MoK_a	MoK_α	MoK_α	MoK_α
$\mu(K_\alpha)$, mm^{-1}	1.019	7.985	7.608	3.097	3.619
Range of θ angles, deg	6.40–69.68	3.72–30.59	3.07–28	2.99–28.00	2.97–3.58
Index range	$-8 \le h \le 8$, $-5 \le k \le 3$, $-7 \le l \le 8$	$-26 \le h \le 8$, $-11 \le k \le 11$, $-9 \le l \le 10$	$-48 \le h \le 43$, $-7 \le k \le 9$, $-21 \le l \le 25$	$-10 \le h \le 8$, $-10 \le k \le 8$, $-21 \le l \le 22$	$-17 \le h \le 25$, $-10 \le k \le 10$, $-10 \le l \le 4$
Crystal sizes, mm	0.17 × 0.04 × 0.04	0.4 × 0.4 × 0.4	0.3 × 0.3 × 0.3	0.4 × 0.4 × 0.4	0.3 × 0.3 × 0.3
Total number of reflections	487	2959	5260	2887	2483
Number of independent reflections	454	1943	923	2366	1653
Number of refined parameters	75	107	215	210	139
Goodness-of-fit	1.119	0.861	0.501	1.169	1.027
R factor R_1/wR_2 ($I \ge 2\sigma(I)$)	0.0689/0.1525	0.0608/0.1425	0.0635/0.1179	0.0592/0.1406	0.0332/0.0594
$\Delta\rho_{max}/\Delta\rho_{min}$, e/Å^3	0.355/–0.431	1.924/–2.541	1.237/–0.973	2.621/–2.880	0.771/–1.313

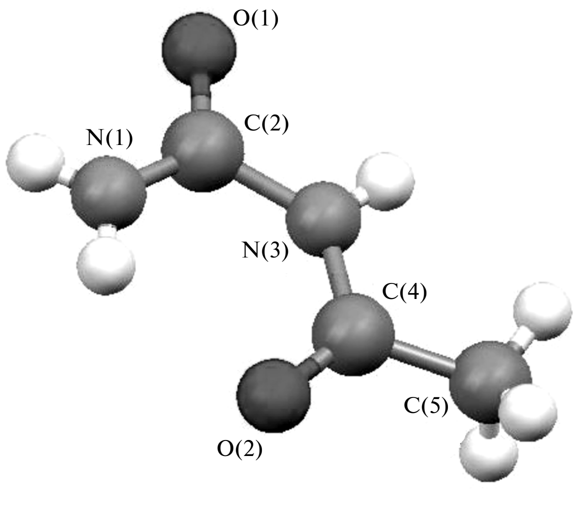

Fig. 1. Structure of acetylcarbamide $CH_3CONHCONH_2$.

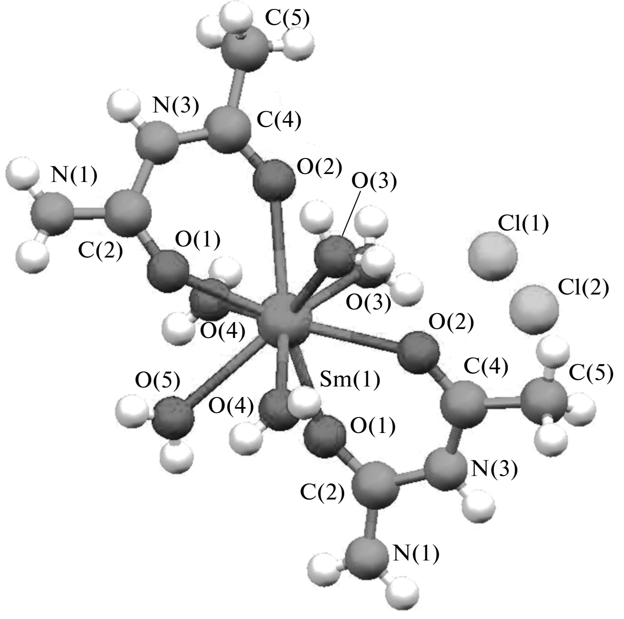

Fig. 2. Structures of complexes **I** and **IV** (exemplified by complex **IV**).

RESULTS AND DISCUSSION

The character of the IR spectra and the shifts of the absorption bands preliminarily suggest the coordination mode of the ligand in the cationic part of the complex. The absorption bands at 1617–1719 cm^{-1} including stretching vibrations of the carbonyl group are of special interest from the viewpoint of establishing the coordination mode.

The IR spectra of acetylcarbamide complexes **I–IV** (Table 1) are of the same type. The shifts of the absorption bands of stretching vibrations ν(CO) + $\delta(NH_2)$ at 1709, 1691, and 1668 cm^{-1} in the spectrum of free acetylcarbamide to lower frequencies (1715–1719, 1674–1677, and 1617–1630 cm^{-1}) in the IR spectra of complexes **I–IV** indicates that acetylcarbamide is coordinated through the oxygen atoms. In the spectra of the complexes, the shift of the absorption band corresponding to asymmetric stretching vibrations of the NH_2 group to higher frequencies (compared to AcUr) is probably due to a change in the structural configuration of the ligand upon complex formation. A similar conclusion was made [4] for the complexes of lanthanide perchlorates.

The X-ray diffraction analysis shows that AcUr has a layered structure with a distance between the layers of 3.383 Å and the layers are parallel to the crystallographic plane (2 0 –1). The unit cell of this compound includes two acetylcarbamide molecules, each of which is nearly planar (the dihedral angle between the O(1)C(1)N(3) and O(2)C(4)N(3) planes including the carbonyl fragments is 4.07° and that between the C(5)C(4)N(3) and N(1)C(2)N(3) planes is –3.65°).

The X-ray diffraction data confirm the *cis–trans* conformation of the CO groups with respect to the imide fragment in the AcUr molecule. This structure contains intramolecular hydrogen bonds H–N–H···O

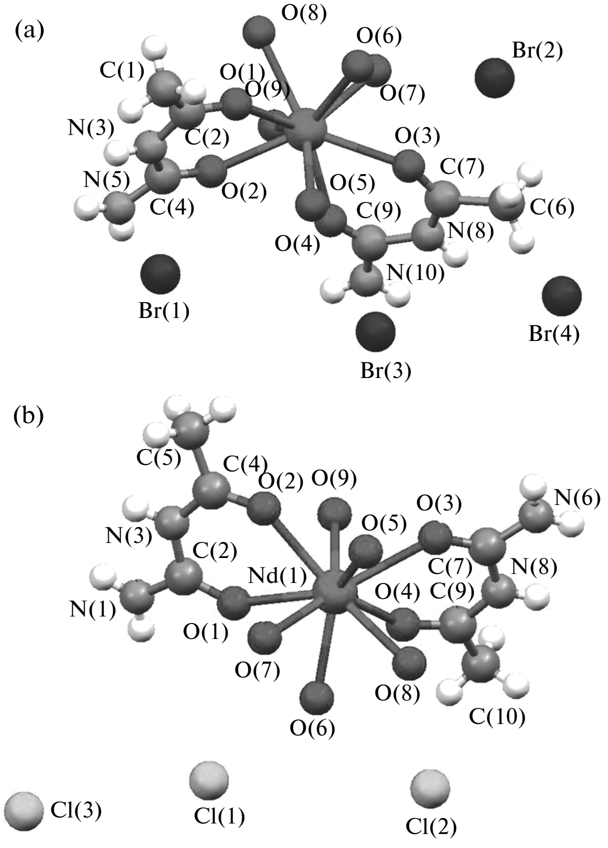

Fig. 3. Structures of complexes (a) **II** and (b) **III**. Hydrogen atoms of the coordinated water molecules are omitted.

Table 3. Selected bond lengths and bond angles in the structures of AcUr and complexes **I–IV**

Bond	AcUr	**I** (Ln = La)	**II** (Ln = Pr)	**III** (Ln = Nd)	**IV** (Ln = Sm)
	d, Å				
Ln(1)–O(1)		2.493(9)*	2.559(14)*	2.498(7)*	2.402(5)*
Ln(1)–O(2)		2.544(8)*	2.374(16)*	2.468(8)*	2.463(4)*
Ln(1)–O(3)		2.531(10)	2.521(14)*	2.499(7)*	2.442(5)
Ln(1)–O(4)		2.551(9)	2.472(14)*	2.520(8)*	2.447(5)
Ln(1)–O(5)		2.653(16)	2.495(14)	2.533(7)	2.598(9)
Ln(1)–O(6)			2.518(13)	2.486(7)	
Ln(1)–O(7)			2.487(17)	2.502(7)	
Ln(1)–O(8)			2.513(13)	2.508(8)	
Ln(1)–O(9)			2.571(16)	2.463(7)	
N(3)–C(2)	1.412(4)	1.395(15)	1.36(3)	1.384(15)	1.384(8)
N(3)–C(4)	1.359(7)	1.354(16)	1.42(2)	1.347(13)	1.346(8)
C(4)–O(2)	1.218(6)	1.217(14)	1.23(2)	1.248(12)	1.231(7)
C(2)–O(1)	1.253(6)	1.253(14)	1.19(3)	1.247(12)	1.240(8)
C(7)–O(3)			1.23(2)	1.235(12)	
C(9)–O(4)			1.16(2)	1.217(12)	
N(8)–C(7)			1.32(3)	1.405(14)	
N(8)–C(9)			1.44(3)	1.343(14)	
Angle	ω, deg				
C(2)O(1)Ln(1)		138.1(8)	141.1(15) 134.6(15)	137.8(7) 138.8(7)	138.0(4)
C(4)O(2)Ln(1)		139.6(8)	145.0(12) 139.9(17)	137.7(8) 137.7(8)	138.7(4)
O(1)C(2)N(1)	125.4(3)	122.8(11)	126(2)	122.1(11)	123.1(7)
O(1)C(2)N(3)	115.5(4)	121.1(10)	121(2)	123.6(9)	122.4(6)
N(1)C(2)N(3)	119.2(4)	116.0(10)	113(2) 110.5(19)	114.2(10) 113.7(10)	114.5(6)
C(4)N(3)C(2)	128.2(4)	126.7(10)	125.7(17) 123.4(19)	127.1(9) 127.3(9)	126.2(5)
O(2)C(4)N(3)	122.8(4)	123.6(11)	121.5(17)	124.2(10)	123.2(6)
O(2)C(4)C(5)	122.9(5)	122.5(12)	123(2) 118(2)	120.6(10) 119.4(10)	121.3(6)
N(3)C(4)C(5)	114.2(4)	113.8(11)	115(2) 114.2(19)	115.2(10) 117.2(10)	115.4(6)

* The bonds of the complexing agent with the oxygen atoms of acetylcarbamide are marked.

(H···O 2.176 and 2.410 Å) and N–H···O (H···O 2.354 Å). The system of hydrogen bonds also stabilizes each layer and results in a 2D structure of the compound.

The crystals of complexes **I** and **IV** are isostructural, unlike nonisostructural crystals of complexes **II** and **III**. The crystals of all four studied compounds include complex cations $[Ln(H_2O)_5(AcUr)_2]^{3+}$ (coordination number 9) and uncoordinated halide ions. The coordinated AcUr molecules are joined with the halide ions and water molecules of the adjacent complex cations by hydrogen bonds. The water molecules are also coordinated in the complex cation. Two structural units fall onto the unit cell in the case of complexes **I**, **III**, and **IV**, whereas there are eight structural units per unit cell in the case of complex **II**.

The AcUr molecules are coordinated via the bidentate mode through the oxygen atoms. They transfer from the initial *cis–trans* conformation to the *trans–trans* conformation due to the rotation of the $-C(O)-NH_2$ fragment around the nitrogen atom of the imino group. As a result, the ligand molecule becomes nonplanar: the dihedral angles between the N(3)C(2)O(1) and N(3)C(4)O(2) planes in the coordinated AcUr molecules are equal to 14.30° in structure **I** and 13.39° in structure **IV**. In symmetric complexes **I** and **IV**, both ligands are distorted to the same extent.

The deviations from coplanarity of the first and second AcUr ligands differ for nonsymmetric complexes **II** and **III**: the dihedral angles between the N(3)C(2)O(1) and N(3)C(4)O(2) planes are 5.70° in structure **II** and 6.02° in structure **III**, and those between the N(8)C(7)O(3) and N(8)C(9)O(4) planes are 8.97° and 7.37° for structures **II** and **III**, respectively. This change in the shape of the amide ligand upon the coordination to the lanthanide atom is evidently caused by an additional stabilization after the formation of chelates involving the complexing atom and AcUr molecules (symmetric in the case of **I** and **IV** and nonsymmetric for **II** and **III**).

A similar rotation of the planar molecules of ligand AcUr upon complex formation has previously been mentioned for lanthanide chlorides and bromides with biurete $NH_2CONHCONH_2$ [13, 14]. However, the degree of coplanarity of the highly symmetric biurete molecule in complexes decreases monotonically in the lanthanide series, whereas the situation is more complicated for an asymmetric AcUr molecule coordinated to a lanthanide atom. It is most likely that spatial factors related to difficulties of arrangement about the central atom with respect to a fairly large and nonsymmetric ligand AcUr are imposed on the chelating effects. In addition to two AcUr molecules, five water molecules are coordinated in the internal sphere (Table 3).

In the case of complexes **I** and **IV**, the coordination polyhedron of the Ln atom is a one-capped distorted square antiprism. The bottom base of the antiprism is nearly planar, whereas the upper one is distorted and larger in size than the bottom base. The bidentate ligand AcUr is arranged at the lateral edges of the antiprism in such a way that the $O-C-CH_3$ fragment is localized in the bottom base and $O-C-NH_2$ lies in the upper one. The structure includes many hydrogen bonds, whose formation involves four of five coordinated water molecules. The O(5) water molecule localized in the cap forms no hydrogen bonds and is most strongly remote from the complexing atom.

The $La-O(5)(H_2O)$ bond for complex **I** is 2.653 Å ($Sm-O(H_2O)$ in complex **IV** is 2.598 Å), whereas the lengths of other $La-O(H_2O)$ bonds are 2.531–2.551 Å in complex **I** and the $Sm-O(H_2O)$ bond lengths are 2.442–2.447 Å in compound **IV** (Table 3).

In the case of complexes **II** and **III**, the coordination polyhedron of the L atom is a three-capped trigonal prism. The acetylcarbamide molecules in complex **II** are localized at the edges of two "caps" and two $O-C-CH_3$ fragments are generatrices of the triangular base. The O(9) water molecule occupies the vertex of the third "cap." The length of this $Pr-O(H_2O)$ bond is 2.571 Å, whereas the range of other $Pr-O(H_2O)$ bond lengths is 2.487–2.518 Å (Table 3).

The acetylcarbamide molecules in complex **III** lie along the edges of the prism bases in such a way that all four oxygen atoms form a lateral face and the $O-C-CH_3$ bonds form diagonals of the lateral face. The oxygen atoms of three water molecules serve as vertices of the "caps" in structure **III**, and the cap of the lateral face formed by the oxygen atoms of AcUr is characterized by a shortest Nd–O(9) distance of 2.463 Å (Table 3).

According to the lengths of the complexing agent–oxygen bonds, ligands AcUr and H_2O are coordinated with approximately equal strengths. Thus, in the case of AcUr, no tendency to the predominant coordination of the oxygen-containing amide ligand compared to the water molecules is observed, which was characteristic of the earlier studied complexes of lanthanide halides with related amide ligands (carbamide, acetamide, and biurete) [13–18]. Probably, this fact is a reason for an increase in the coordination number of the L atoms and coordination of the additional (fifth) water molecule.

It is known that the complexes with the coordination number 9 are characterized by two geometric configurations: a three-capped trigonal prism and somewhat less stable one-capped tetragonal antiprism. The both polyhedra should be distorted due to the migration of the vertices to the spherical surface and a decrease in the sizes of the large faces [20]. If all nine ligands are monodentate, the most energetically favorable shape of the three-capped trigonal prism is formed by its compression along the 3-fold axis. This increases the distance from the central atom to the "capped" ligand. A one-capped square antiprism is similar to a three-capped trigonal prism (any of three "capped" atoms of a three-capped trigonal prism can be considered as a special "capped" atom of a square antiprism).

If the complex with the coordination number 9 contains four equivalent bidentate ligands and one monodentate ligand (of the [M(bidentate ligand)$_4$(monodentate ligand)] type), the most energetically stable polyhedron is a one-capped square antiprism in which the bidentate ligands are arranged on the edges that bind two square faces of the antiprism. It was shown on the basis of the calculation of the repulsion energy [19] that bidentate ligands should be bound asymmetrically in the complexes with the configuration of a one-capped square antriprism. The atoms forming the "capped" face experience a weaker repulsion than those of other faces. Therefore, the ligand localized in the "cap" forms the weakest bond with the central atom.

Similar assumptions can be made for the studied complexes **I–IV** assigned to the [M(bidentate ligand)$_2$(monodentate ligand)$_5$] type: (a) the bidentate ligands are nonsymmetrically arranged and (b) the central atom–"capped" ligand (in our case, the water molecule) bond is elongated.

The revealed differences in geometric characteristics of complexes **I–IV** can be due to fine distinctions in the electronic structure of the central atom.

The packings of the complexes are different. Complexes **I** and **IV** include wavy surfaces formed by two coordinated water molecules and uncoordinated halide ions joined to each other by hydrogen bonds. In the case of compound **II**, the structure contains cavities including columns of bromide ions.

A fairly low strength of the lanthanide–AcUr bond shows that the studied complexes are promising for use as intermediates of further syntheses of compounds with complicated organic (including heterocyclic) ligands.

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