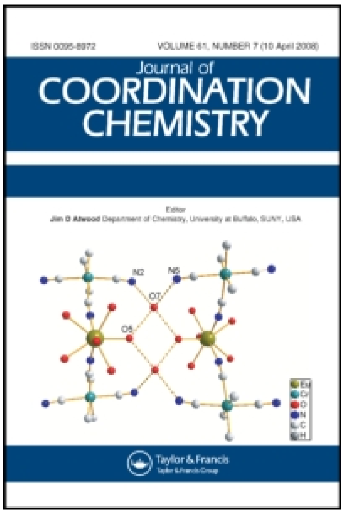

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THE CRYSTAL STRUCTURE AND PROPERTIES OF CATENA-POLY[TETRAAQUA-*BIS(μ-β-ALAmNAJO-K³*O,O',O”)-TETRAKIS (μ-β-ALANINATO-k^{2}O,O)DINEODYMIUM(III) HEXACHLORIDE

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THE CRYSTAL STRUCTURE AND PROPERTIES OF *CATENA*-POLY[TETRAAQUA-*BIS*(μ-β-ALANINATO-κ^3O,O',O'')-*TETRAKIS* (μ-β-ALANINATO-κ^2O,O')DINEODYMIUM(III)] HEXACHLORIDE

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A Nd(III) complex with β-alanine of empirical formula $[Nd(\beta\text{-ala})_3 \cdot 2H_2O]Cl_3$ has been synthesised and characterised. The crystal structure of the complex was studied using single-crystal X-ray diffraction methods. When heated the complex loses water molecules and subsequent decomposition to Nd_2O_3 takes place. The complex crystallizes in space group $P2_1/c$ with $a = 10.846(3)$, $b = 19.552(4)$, $c = 9.464(3)$ Å, $\beta = 102.02(3)°$, $Z = 4$. The structure consists of polymeric chains connected through hydrogen bonds. Neodymium(III) ions in the chains are bonded alternately through two and four carboxylate bridges from β-alanine molecules. Carboxylate groups are bidentate-chelating and tridentate chelating-bridging. Two molecules of water complete the coordination number of Nd(III), which is nine.

Keywords: Neodymium(III); amino acid complexes; crystal structure

INTRODUCTION

In spite of the fact that lanthanides almost do not occur in natural biomolecules,[1] many investigations in solution[2–4] as well as in the solid state on their interaction with amino acids and simple peptides have been carried out. Lanthanide(III) ions possess unique chemical properties which make

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them valuable for investigation of more complicated biological systems. These properties are especially useful in the determination of the nature of calcium containing proteins.[5,6]

In reported structures of lanthanides(III) with amino acids, different carboxylate coordination modes are observed while the amino group is uncoordinated. Main dissimilarities are observed in the behaviour of the carboxylate group. Taking this into consideration, the complexes may be divided into several groups. The first involves compounds where the carboxylate group is of bridging-bidentate character. Then metal ions are held together by one,[3,4,7,8] two[9–11] or four[2,12,13] carboxylate bridges or simultaneously by two and four bridges[14,15] and in the majority of the complexes polymeric species are formed. The type of structure in these complexes also depends on the molar ratio of Ln(III) to amino acid in which the complex is obtained. The next group concerns structures in which the carboxylate group forms a chelate ring with a metal ion. In this case, formation of monomeric structures is observed.[16,17] The terdentate character of the carboxylate group is relatively often observed in amino acid-lanthanide(III) compounds.[18,19] The last group is one in which a unidentate carboxylate group coordinates a lanthanide(III) ion.[11] When amino acids like serine or threonine coordinate lanthanides(III), the oxygen atoms from the hydroxyl groups also take part in binding to the metal ion.[7,20] The coordination number of lanthanide(III) complexes with amino acids is 8 or 9 and is completed by water molecules.

This paper discusses the properties and crystal structure of a neodymium(III) complex with β-alanine, which occurs in natural biosystems, *e.g.*, in coenzyme A.[21] The crystal structure of *catena*-poly[tetraaqua-*bis*(μ-β-alaninato-$\kappa^3 O,O',O''$)-*tetrakis*(μ-β-alaninato-$\kappa^2 O,O'$)-dineodymium(III)] hexachloride is very similar to that presented earlier in papers[19,22] but quite different compared to the Nd(III) complex with β-alanine[23] and complexes of lanthanides(III) with α-alanine.[2,4]

EXPERIMENTAL

Crystals of the investigated complex were grown by the slow evaporation of an aqueous solution of 0.2 M neodymium(III) chloride and 0.2 M β-alanine in a 1 : 3 ratio. Violet crystals formed after a few days. The percentage of carbon, hydrogen and nitrogen was determined with a Perkin-Elmer elemental analyser. Analytical data for $[Nd(C_3H_7O_2N)_3 \cdot 2H_2O]Cl_3$(%): found: C, 19.82; H, 3.70; N, 7.62. Calc.: C, 19.67; H, 3.82; N, 7.58. Experimental density of the crystals was measured by flotation in a $CCl_4/C_2H_4Br_2$ mixture.

Thermal Measurements

The thermal stability of the complex was examined by means of TG, DTG and DTA techniques. Measurements were made using an Q 1500D derivatograph. A sample (100 mg) was heated at a rate of 10 deg/min to 1000°C in a ceramic crucible in static air using α-Al_2O_3 as reference material. The obtained complex and the final solid product of decomposition were identified by the Debye-Scherrer method using a DRON 2 diffractometer equipped with monochromated CuKα radiation (Table I). The diffraction pattern of the thermal decomposition residue was compared with that obtained from the powder diffraction files of the Joint Committee on Powder Diffraction Standards.

Spectroscopic Measurements

IR spectra were recorded in the solid state with a Specord M80 spectrophotometer in the range 4000–400 cm^{-1} in KBr discs (Table II).

X-ray Structure Determination

Preliminary Weissenberg photographs gave the crystal system, space group and approximate unit cell dimensions. Crystallographic data and refinement procedures are given in Table III. The data were corrected for Lorentz and polarization effects. The structure was solved by the heavy-atom method using SHELXS-86[24] and refined by full-matrix least-squares methods using SHELXL-93.[25] H-Atoms of the water molecules were located from a

TABLE I X-ray powder diffraction data for $[Nd(\beta\text{-ala})_3\cdot 2H_2O]Cl_3$

$[Nd(\beta\text{-}ala)_3\cdot 2H_2O]Cl_3$	
d (Å)	$I/I_O \times 100$
6.7787	25
4.8636	1
4.8062	10
3.4270	100
2.4552	33
2.4194	18
2.4169	11
2.3631	11
2.2891	15
2.2464	15
2.0758	19
2.0713	14

TABLE II IR assignments of diagnostic bands of β-alanine and $[Nd(\beta\text{-ala})_3 \cdot 2H_2O]Cl_3$

β-alanine (cm^{-1})	*Complex* (cm^{-1})	*Vibrations modes*
3700–2300	3600–2400	νCH; νCH_2; νNH_3^+; νNH
1632	1660; 1625	$\delta_{as}NH_3^+$; $\beta_S NH_2$
1572	1580; 1540	$\nu_{as}COO$
1460	1452	$\delta_{as}CH_2$
1396	1440; 1420	$\nu_s COO$
1336	1337	$\delta_s CH_2$
1264	1260	ωCH_2
1160	1140	ωNH_2
1060; 992	1100; 960	νskel
888	870	ρCH_2
660	640	βCOO
576	565	ρCOO

TABLE III Experimental data and summary of intensity collection and structure data

Formula	$C_9H_{25}Cl_3N_3NdO_8$
Colour	Violet
M_r	553.91
Space group	$P2_1/c$
Temp. °C	20
Cell constants (40 reflections, $28.5 < \theta < 36.4°$)	25
a, Å	10.846(3)
b, Å	10.552(4)
c, Å	9.464(3)
β, °	102.02(3)
Cell volume, $Å^3$	1962.9(7)
Z	4
$F(000)$	1100
D_c, $g\,cm^{-3}$	1.874
D_m (flotation $CCl_4/C_2H_4Br_2$) $g\,cm^{-3}$	1.88
μ_{calc}, cm^{-1}	30.92
Crystal size, mm	0.20 × 0.20 × 0.20
Reflection measured	4186
2θ range, deg	4.16–55.16
Range of h, k, l	$0 \rightarrow 14$, $0 \rightarrow 25$, $-12 \rightarrow 12$
Criterion for observed reflections	$I > 2\sigma(I)$
No. of observed reflections	4186
Weights[a] (a, b, f)	0.0454, 2.4373, 1/3
GOF	1.096
$R = \Sigma(\lvert F_o \rvert - \lvert F_c \rvert)/\Sigma(\lvert F_o \rvert)$	0.025
$wR_2 = \{\Sigma(wF_o^2 - F_o^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$	0.072

[a] $w = 1/[\sigma^2(F_o^2) + (a \cdot P)^2 + b \cdot P]$ where $P = [f \cdot \text{Max. of } (0 \text{ or } F_o^2) + (1 - f)F_c^2]$.

difference Fourier map and refined. The other hydrogen atoms were placed in geometrically calculated positions with isotropic temperature factors taken as being $1.2U_{eq}$ of neighbouring heavier atoms. Afterwards, positional parameters were calculated in a riding mode with U_{iso} fixed. Several

TABLE IV Fractional atomic coordinates and equivalent isotropic thermal parameters (Å^2) for non-hydrogen atoms with e.s.d. values in parentheses

Atom	*x/a*	*y/b*	*z/c*	*U_{eq}
Nd	0.00198(2)	0.004494(9)	0.21956(2)	0.01703(7)
Cl(1)	0.60346(10)	0.26749(5)	−0.00681(11)	0.0311(2)
Cl(2)	0.57746(12)	0.05425(6)	0.31152(14)	0.0407(3)
Cl(3)	0.27625(11)	0.33876(5)	0.06588(13)	0.0359(2)
O(1)	−0.0748(3)	−0.0626(2)	0.0039(3)	0.0289(6)
O(2)	−0.1252(3)	−0.1156(2)	−0.2033(3)	0.0319(7)
O(3)	0.1842(3)	−0.0545(2)	0.1541(3)	0.0354(7)
O(4)	0.1708(3)	−0.0741(2)	−0.0807(3)	0.0263(6)
O(5)	−0.0658(3)	0.0760(1)	0.3981(3)	0.0288(6)
O(6)	−0.0273(3)	0.0976(2)	0.6333(3)	0.0313(7)
O(7)	0.1957(3)	0.0175(2)	0.4198(3)	0.0321(7)
O(8)	−0.2112(3)	−0.0406(2)	0.2436(3)	0.0321(7)
N(1)	−0.3429(4)	−0.2010(2)	−0.2460(5)	0.0477(11)
N(2)	0.4120(4)	−0.0776(2)	0.3450(4)	0.0339(8)
N(3)	−0.2388(3)	0.1838(2)	0.2617(4)	0.0307(8)
C(1)	−0.1341(4)	−0.1091(2)	−0.0740(4)	0.0217(7)
C(2)	−0.2194(5)	−0.1571(2)	−0.0147(5)	0.0383(11)
C(3)	−0.3488(5)	−0.1638(2)	−0.1117(7)	0.0467(13)
C(4)	0.2306(4)	−0.0693(2)	0.0479(4)	0.0233(8)
C(5)	0.3714(4)	−0.0809(2)	0.0775(4)	0.0312(9)
C(6)	0.4221(5)	−0.1178(3)	0.2170(5)	0.0413(12)
C(7)	−0.0507(4)	0.1160(2)	0.5040(4)	0.0203(7)
C(8)	−0.0656(4)	0.1920(2)	0.4760(4)	0.0300(9)
C(9)	−0.1119(4)	0.2124(2)	0.3207(5)	0.0332(10)

*$U_{eq} = \frac{1}{3}\Sigma_{ij}U_{ij}a_i^* a_j^* \bar{a}_i \bar{a}_j$.

cycles of refinement of coordinates and anisotropic thermal parameters for non-hydrogen atoms reduced the *R* value to 0.025. Scattering factors were those incorporated in SHELXL-93. Final parameters and estimated standard deviations are listed in Table IV.

RESULTS AND DISCUSSION

Based on thermogravimetric measurements and analytical data, the empirical formula of the complex was determined as $[Nd(C_3H_7O_2N)_3{\cdot}2H_2O]Cl_3$. The powder diffraction data for the readily soluble complex in water is given in Table I. The complex is stable up to about 70°C. During further heating in air the complex loses two water molecules in the range 70–210°C. The theoretical weight loss for the dehydration stage is 6.5% while the experimental value is 6.2%. The unstable, anhydrous compound decomposes in the range 220–700°C to the oxide Nd_2O_3. The final theoretical weight loss upon decomposition is 66.6% and the experimental loss is

67.2%. The product of the decompostion was identified by X-ray powder diffraction.

IR spectra of the complex and free ligand were examined (Table II). Both spectra are characterized by a broad band in the region 2300–3400 cm^{-1}. These peaks are due to overlapping of many vibration bands of CH, CH_2 and NH_3^+ groups as well as the OH stretching vibrations. The β-alanine ligand occurs in the solid state in the zwitterion form and bands at 1572 cm^{-1} and 1396 cm^{-1} may be assigned to asymmetric and symmetric stretching vibrations of the carboxylate group. Bands assigned to the asymmetric and symmetric vibrations of the carboxylate group in the complex are split. Splitting of the COO bands occurs when angles in the carboxylate group vary. Based on this fact, we suggest that metal ions are bonded by the carboxylate group in different modes.

The molecular structure of complex of neodymium(III) with β-alanine was determined by single crystal X-ray diffraction. The complex $[Nd(C_3H_7O_2N)_3{\cdot}2H_2O]Cl_3$ crystallizes in monoclinic crystal system, space group $P2_1/c$. A part of the molecular structure is shown in Figure 1. Selected bond lengths and angles are presented in Table V.

Each neodymium(III) ion is surrounded by nine oxygen atoms. Seven of them involve six carboxylate groups of β-alanine molecules; two remaining oxygen atoms belong to two water molecules. The neighbouring neodymium(III) ions are connected by bridging carboxylate groups of ligands creating a one-dimensional, linear polymer. Chloride anions are in the second

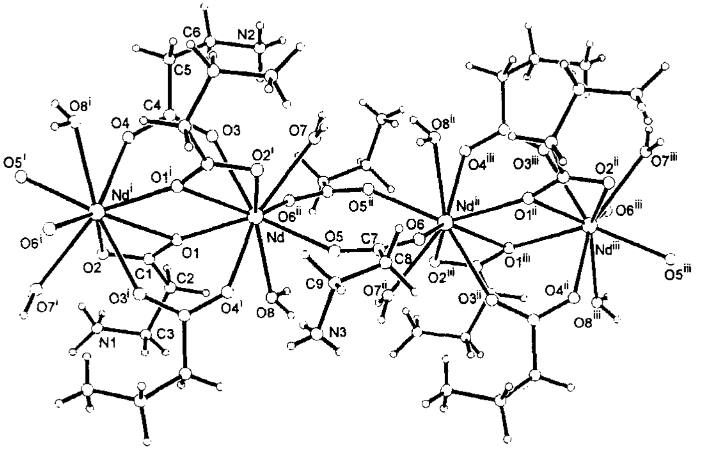

FIGURE 1 The molecular structure of $[Nd(C_3H_7O_2N)_3{\cdot}2H_2O]Cl_3$. Symmetry codes are: (i) $-x$, $-y$, $-z$; (ii) $-x$, $-y$, $1-z$; (iii) x, y, $1+z$.

TABLE V Selected bond lengths (Å) and angles (°) with standard deviations in parentheses

Nd-Nd^{i}	5.320(1)	Nd-Nd^{ii}	4.151(1)
Nd-O(6)^{i}	2.417(3)	Nd-O(2)^{ii}	2.571(3)
Nd-O(5)	2.422(3)	Nd-O(1)^{ii}	2.662(3)
Nd-O(1)	2.424(3)	Nd-O(8)	2.528(3)
Nd-O(4)^{ii}	2.462(3)	Nd-O(7)	2.532(3)
Nd-O(3)	2.476(3)		

Carboxylate groups

O(1)-C(1)	1.259(4)	O(3)-C(4)	1.247(5)	O(5)-C(7)	1.255(5)
O(2)-C(1)	1.253(5)	O(4)-C(4)	1.258(5)	O(6)-C(7)	1.249(4)
O(1)-C(1)-O(2)	120.7(3)				
O(3)-C(3)-O(4)	125.7(4)				
O(5)-C(7)-O(6)	124.7(4)				

Bond lengths in β-alanine molecules

C(1)-C(2)	1.506(5)	C(4)-C(5)	1.511(6)	C(7)-C(8)	1.512(5)
C(2)-C(3)	1.514(7)	C(5)-C(6)	1.505(6)	C(8)-C(9)	1.505(6)
C(3)-N(1)	1.478(7)	C(6)-N(2)	1.467(6)	C(9)-N(3)	1.482(6)

Symmetry code: (i) $-x, -y, -z+1$; (ii) $-x, -y, -z$.

coordination sphere, making the complex neutral. A complex of β-alanine with samarium(III)[19,22] displays the same mode of coordination. However, this compound crystallizes in the triclinic space group $P\bar{1}$ and perchlorate anions are in the second coordination sphere. Distances between neodymium(III) ions in the present complex are not the same, being alternately 4.151(1) and 5.320(1) Å. This difference is caused by the fact that in the polymeric chain pairs of adjacent neodymium(III) ions are linked alternately through four and two bridges. When the neodymium(III) ions are bonded by two carboxylate bridges the Nd–Nd distance is 5.320(1) Å. Subsequent ions are bonded by the two bidentate-bridging carboxylate groups of β-alanine molecules and simultaneously are conneced by two terdentate bridging-chelating carboxylate groups of two further amino acid molecules. This decreases the Nd–Nd distance to 4.151(1) Å. Analogous polymeric structures were found for other complexes of lanthanide(III) ions with amino acids of the molar ratio 1:3, *e.g.*, complexes of glycine[14,26] and proline.[15] However, in the neodymium(III) complex with *L*-proline and in the complex of erbium(III) with glycine[26] there are only bidentate-bridging carboxy- late groups. On the other hand the complex of Nd(III) with β-alanine prepared in the stoichiometric ratio 1 : 1 presents a quite different crystal structure; Nd(III) ions are linked only through one carboxylate bridge in the polymeric chain.[23] The nature of lanthanide coordination changes also as the result of the position of the NH_2 group in the amino acid. For example, in complexes of holmium(III) and dysprosium(III) with α-alanine,

TABLE VI Hydrogen bond lengths (Å) and bond angles (°)

D–H···A	D–H	H···A	D···A	∠D–H···A
O(7)–H(1)···Cl(3)^{i}	0.87	2.38	3.174(3)	153
O(8)–H(4)···Cl(2)^{ii}	0.78	2.38	3.118(3)	158
N(1)–H(1A)...Cl(1)^{iii}	0.89	2.46	3.329(5)	164
N(1)–H(1B)...Cl(3)^{iv}	0.89	2.38	3.189(5)	151
N(1)–H(1C)...O(2)	0.89	2.24	2.853(6)	128
N(2)–H(2B)...O(3)	0.89	2.13	2.777(5)	129
N(2)–H(2C)...Cl(2)	0.89	2.32	3.195(4)	169
N(3)–H(3A)...O(4)^{iv}	0.89	2.20	2.934(5)	139
N(3)–H(3B)...Cl(1)^{ii}	0.89	2.33	3.201(4)	168
N(3)–H(3C)...Cl(1)^{v}	0.89	2.33	3.193(5)	165

Symmetry code: (i) $x, 0.5-y, 0.5+z$; (ii) $-1+x, y, z$; (iii) $-x, -0.5+y, -0.5-z$; (iv) $-x, -y, -z$; (v) $-1+x, 0.5-y, 0.5+z$.

metal ions are linked by four in the dimeric and one carboxylate bridge in the polymeric structures and the Ln–Ln distances are 4.468(1) Å and 5.771(1) Å, 5.811(1) Å, respectively.[2,4]

In the coordination polyhedron around Nd(III) ions, the Nd–$O_{carboxyl}$ bond lengths are in the range 2.417(3)–2.662(3) Å. The longest Nd–O bond distance is observed when carboxylate group takes part in chelating the Nd(III) ion. The water molecules are bonded to the same extent and the Nd–O_{water} distances are 2.528(3) and 2.571(3) Å. In the dimeric complex of Ho(III) the distances Ln–O are significantly shorter.[2] It is noteworthy that the angles in the carboxylate groups differ markedly. That is in good agreement with the different behaviour of the carboxylate groups. In the bidentate-bridging carboxylate groups O–C–O angles are in the range 124.7(4) to 125.7(4)°. Significantly smaller angles of 120.7(3)° were observed in the bridging-chelating groups. These differences are clearly reflected in IR spectra of the complex. In the carboxylate groups, C–O bond lengths are in the range 1.247(5)–1.259(4) Å.

The compound exhibits a system of *inter*molecular and *intra*molecular hydrogen bonds. Corresponding distances and angles are presented in Table VI. Each water molecule gives rise to one O–H···Cl bond. The protonated NH_3^+ groups of β-alanine molecules take part in the hydrogen bonds (N–H···Cl and N–H···O) as proton acceptors. Chloride anions and carboxylate oxygen atoms are proton donors in hydrogen bonds, creating a stable network in the crystal.

Supplementary Material

Lists of H atom positions, thermal parameters and observed and calculated structure factors are available from the authors.

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