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Structure and vibration spectra of neodymium biglycinate

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Abstract

The X-ray diffraction analysis of neodymium biglycinate was carried out and it was ascertained that there are nine atoms in the internal coordination sphere of the neodymium ion: three aqua oxygen atoms, five oxygen atoms of carboxyl group, and a chlorine atom. Having such coordination the tested compound should be considered a dimer that has the structure: $\{[Nd(HGl)_2(H_2O)_3Cl]Cl_2\}_2$. Vibration (IR and Raman) spectra of the compound were measured in the range 4000–40 cm^{-1}. Calculations of the frequencies and the forms of normal vibrations for some molecular models, which describe the real complex, were made on the group calculation technique basis.

Keywords: Crystal structures; Lanthanides; Rare earths; Vibration spectra

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1. Introduction

Until the present time the formation of complexes from rare earths (RE) with natural amino acids were studied in solutions mainly [1,2]. As a rule the composition of a complex does not reflect the proportions of its components in solution, which can be explained by low stability constants of complexes formed from amino acids with RE and by the lability of the donor–acceptor bonds that form them [1]: RE ions with α-amino acids form weak complexes of the ion type, that results in a wide range of possible complex compounds with different compositions. The presence of complexes with different compositions in the solution at the same time leads to their co-crystallisation when the products of the reaction are extracted by mere evaporation. In our opinion this is the source of contradictions in the works of various authors [3]. Thus, the conclusion was made in work [4] that a solid RE biglycinate cannot exist, while solid lanthanide complexes with *N*-bis(2-hydroxyethyl)glycine were described in [5]. It is necessary to stress that in aqueous solution the RE ion is surrounded with a rather strong hydro shell comprising up to nine molecules of water. So neutral glycine molecules are monodentate ligands (the α-amino group is protonised and is not able to participate in the coordination) and the equivalent substitution of hydrating water molecules by glycine molecules can be expected. The assumption that a neutral glycine molecule is monodentate is corroborated by the data from IR spectra in [1]. As it is pointed out in work [6], the formation of bidentate bridge bonding and tridentate-bridge-cyclic bonding of a carboxyl with a RE ion can be expected (so one should not confuse monodenticity of a glycine molecule that has one carboxyl group participating in coordination, and denticity of a carboxyl group itself). It can be supposed that the bidentate bonding and the forming of bridge bonds exactly results in the lowering of the number of the coordinated water molecules in RE amino acid complexes (expected number of water molecules is four to five). So it is obvious that structural data for products of the interactions of RE ions with glycine is contradictory.

The results of X-ray diffraction and spectroscopic (IR, Raman) analyses of biglycinate neodymium obtained in the crystal form are presented in the paper.

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2. Experimental

For neodymium biglycinate synthesis we used the method, suggested in [11], of mixing initial substances with further concentration of the mother solution by evaporation and, after that, crystallisation by the method of isothermal evaporation. The used glycine was previously recrystallised in bidistilled water, and then dissolved with addition of sodium hydroxide. For preparation of the mother solution 0.3 M solutions of the initial substances were mixed in proportion M:L = 1:3 (total volume of mother solution was 100 ml). Then the mixture was evaporated to 25 ml, cooled to room temperature (r.t.) and placed in exsiccator for isothermal evaporation. The process of crystallisation was monitored visually, using a microscope, as described in [11]. Diffraction data were obtained for sphere samples with diameter 0.4 mm using a Syntex P2 diffractometer (λ Mo Kα graphite monochromator). The structures were calculated by the program complex SHELX-97 [12]. IR spectra in the range 4600–200 cm^{-1} were measured using a Perkin–Elmer-180 spectrophotometer. Long-wave IR spectra in the range 400–40 cm^{-1} were measured using a LAFS-1000 spectrophotometer in liquid petrolatum and KBr tablets. Raman spectra were measured using a SPEX Ramalog spectrophotometer. A crystal powder of the examined compound was placed into glass or quartz capillary tubes.

3. Results and discussion

According to the microscopic analysis data, crystallisation and phase transmutations in the RE–α-amino acid system under low values of pH < 5 lead to the conversion of all the mother solution into a glassy mass, or form surface films which prevent further evaporation. We found out that phases with highly symmetric packing of structural units are formed in the starting stage of the crystallisation process. Different combinations of cube and octahedral forms are generated from RE solutions in glycine under pH 6–7. The obtained crystals are unstable and are destructible when stored in the open air. The dissolution of the crystals in water is an endothermic process ($\Delta H_{\text{solv}} > 0$) that is connected with the way the structural units are packed in cell nodes. However, long isothermal incubation of the crystals in the mother solution causes the structural reconfiguration of the highly symmetric phase that seems to be connected with complicated interatomic

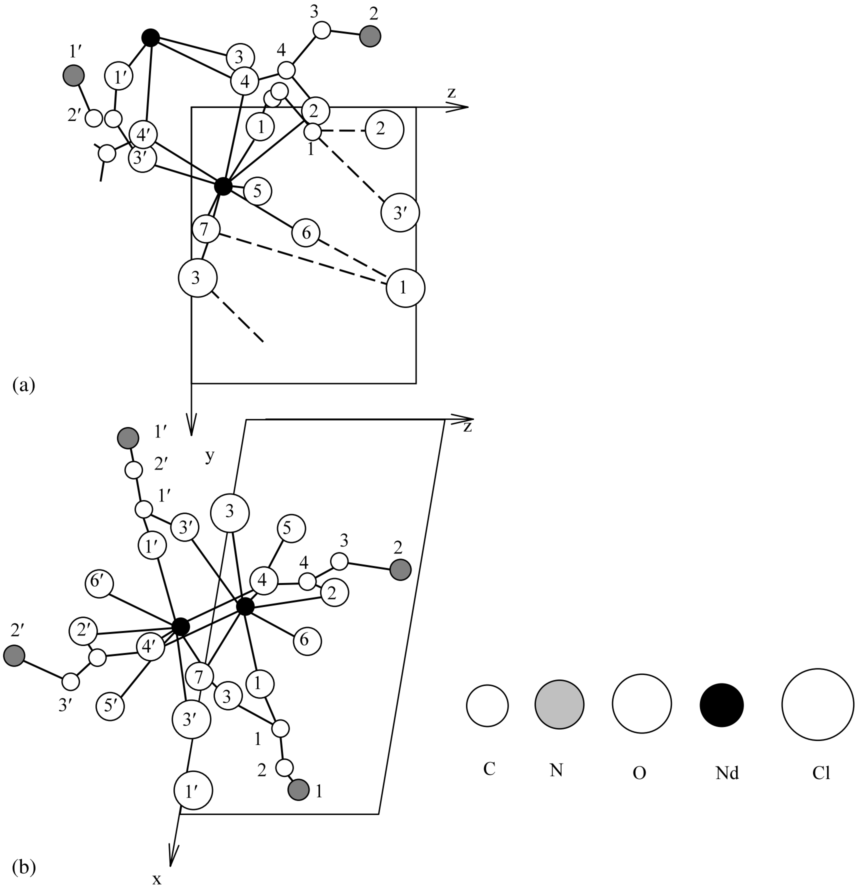

Fig. 1. The perspective drawing of yz (a) and zx (b) planes of $\{[\text{Nd(HGl)}_2(\text{H}_2\text{O})_3\text{Cl}]\text{Cl}_2\}_2$ and the numbering scheme of atoms.

and intermolecular interactions taking place in the solid phase. We observed crystal nucleation and growth of a daughter (dissymmetrical—D) phase in the mother (symmetrical—M) phase volume. The D-phase is simultaneously generated in several points of the M-phase with symmetry, respectively, to the fourth degree axis and grows exactly oriented in a way that seems to be connected with the influence of the crystal field. During the growth of the D-phase, the M-phase splits into three separate blocks due to generated tensions. Long isothermal incubation of the mother solution leads to enlargement and homogenisation of the crystals due to Oswald growth and aggregation. These crystals acquire form of rhombic dipyramids or prisms with edges of length up to 2 cm.

These cause the borders where the original blocks coalesce to disappear. According to X-ray diffraction analysis data (Fig. 1, Table 1) the crystals are classified as crystals with monoclinic syngony with the lattice parameters: $\alpha = 10.037(3)$; $b = 14.630(4)$; $c = 10.630(2)$ Å; $\beta = 101.98(2)°$; $P2_1/m$, $Z = 4$. The crystals are stable in air and are transparent. The dissolving process of these monocrystals is exothermal ($\Delta H_{\mathrm{solv}} < 0$), that is connected with the arrangement of the complex molecules in the cell nodes. It can be supposed that the secondary solid-state phase change $O_h \rightarrow C^2/_n$ occurs in such systems; the volume of the elemental cell is enlarged four times under such conditions.

As one can see in Fig. 1 (around the neodymium ion in its internal coordination sphere), the neodymium ion is nine coordinated by three oxygen atoms of water, five oxygen atoms of carboxyl groups and one chlorine atom with a Nd–Cl bond length of 2.873 Å. There are no atoms of glycine nitrogen in the internal coordination sphere. The presence of one chlorine atom in the internal coordination sphere of the RE ion is an intermediate case between triglycinates, where chlorine ions are not in the internal coordination sphere [1], and monocomplexes, where two chlorine ions may be in the internal coordination sphere [6].

The difference between the biglycinate structure and structure of triglycinate crystals can also be observed in the way of glycine coordination. As one can see in Fig. 1, each neodymium ion is linked to four glycine molecules, two of them form only bidentate bridge bonds with the neighbouring neodymium ion and the other two are linked to one of the neodymium ions and bridge the Nd–O–Nd′ bond through one oxygen atom of the carboxyl group by means of tridentate-bridge-cyclic coordination with the formation of a four-unit ring. Having such coordination the tested compound should be considered a dimer that has the structure: $\{[Nd(HGl)_2(H_2O)_3Cl]Cl_2\}_2$. The data of X-ray diffraction analysis of neodymium biglycinate goes well with the results of the theoretical and experimental study of absorption spectra. Previously the calculations and empirical assignments were made for complexes of α-

Table 1
Selected bond lengths (Å) and bond angles (°) for $\{[Nd(HGl)_2(H_2O)_3Cl]Cl_2\}_2$

Bond	*d*	Bond	*d*	Bond	*d*	Bond	*d*
Nd–C1(3)	2.872	C1(2)···N(1)	3.124	O(1)···(4)	3.124	O(3’)··· O(5)	2.978
Nd–O(l)	2.433	Cl(2)···O(7)	3.173	O(1)···N(1)	2.527	O(3)–C(1)	1.257
Nd–O(2)	2.612	Cl(3)···O(5)	3.185	O(1)···O(7)	2.888	O(3)···C(2)	2.348
Nd–O(3′)	2.428	C1(3)···N(1)	3.143	O(2)···O(4)	2.201	O(4)···O(4′)	2.939
Nd–O(4)	2.584	Cl(3)···O(7)	3.180	O(2)···O(5)	2.734	O(4)···C(1)	3.099
Nd–O(4′)	2.481	O(1)···O(2)	2.863	O(2)···O(6)	3.091	O(4)–C(4)	1.265
Nd–O(5)	2.527	O(1)··· O(2′)	2.525	O(2)–C(4)	1.246	O(4)···C(3)	2.386
Nd–O(6)	2.522	O(1)···O(4)	2.990	O(2)···C(3)	2.404	O(4)···O(7)	2.946
Nd–O(7)	2.486	O(1)···O(4′)	2.798	O(2)···N(2)	2.663	O(5)···O(6)	3.184
Nd–C(4)	2.956	O(1)···O(6)	3.183	O(2)···N(2′)	2.884	C(1)–C(2)	1.516
Cl(1)···O(6)	3.183	O(1)–C(1)	1.252	O(3′)···O(4)	2.847	C(1)···N(1)	2.442
Cl(1)···O(7)	3.135	O(1)···C(2)	2.364	O(3′)···O(4′)	2.976	C(2)–N(1)	1.480
Angle	*α*	Angle	*α*	Angle	*α*	Angle	*α*
O(1)–C(1)–O(3′)	127.65	Cl(3)–Nd–O(4)	144.54	O(2)–Nd–O(4)	50.12	O(3′)–Nd–O(7)	116.47
O(1)–C(1)–C(2)	116.96	Cl(3)–Nd–O(4′)	111.30	O(2)–Nd–O(4′)	69.40	O(4)–Nd–O(5)	85.12
O(3)–C(1)–C(2)	115.40	Cl(3)–Nd–O(5)	72.84	O(2)–Nd–O(1)	69.03	O(4)–Nd–O(6)	123.26
C(4)–C(3)–N(2)	109.01	Cl(3)–Nd–O(6)	78.73	O(2)–Nd–O(3′)	109.04	O(4)–Nd–O(7)	136.24
O(4)–C(4)–O(2)	122.43	Cl(3)–Nd–O(7)	72.41	O(2)–Nd–O(5)	132.00	O(4)–Nd–O(4′)	70.89
C(3)–C(4)–O(2)	120.15	O(1)–Nd–O(4)	73.09	O(2)–Nd–O(6)	79.81	O(5)–Nd–O(6)	77.49
C(3)–C(4)–O(4)	117.27	O(1)–Nd–O(4′)	69.40	O(2)–Nd–O(7)	71.94	O(5)–Nd–O(7)	138.40
C(4)–C(3)–N(2)	109.61	O(1)–Nd–O(3′)	134.01	O(3′)–Nd–O(4)	72.77		
Cl(3)–Nd–O(l)	142.05	O(1)–Nd–O(5)	132.06	O(3′)–Nd–O(4′)	70.89		
Cl(3)–Nd–O(2)	132.33	O(1)–Nd–O(6)	79.81	O(3′)–Nd–O(5)	73.84		
Cl(3)–Nd–O(3′)	74.86	O(1)–Nd–O(7)	71.94	O(3′)–Nd–O(6′)	145.67		

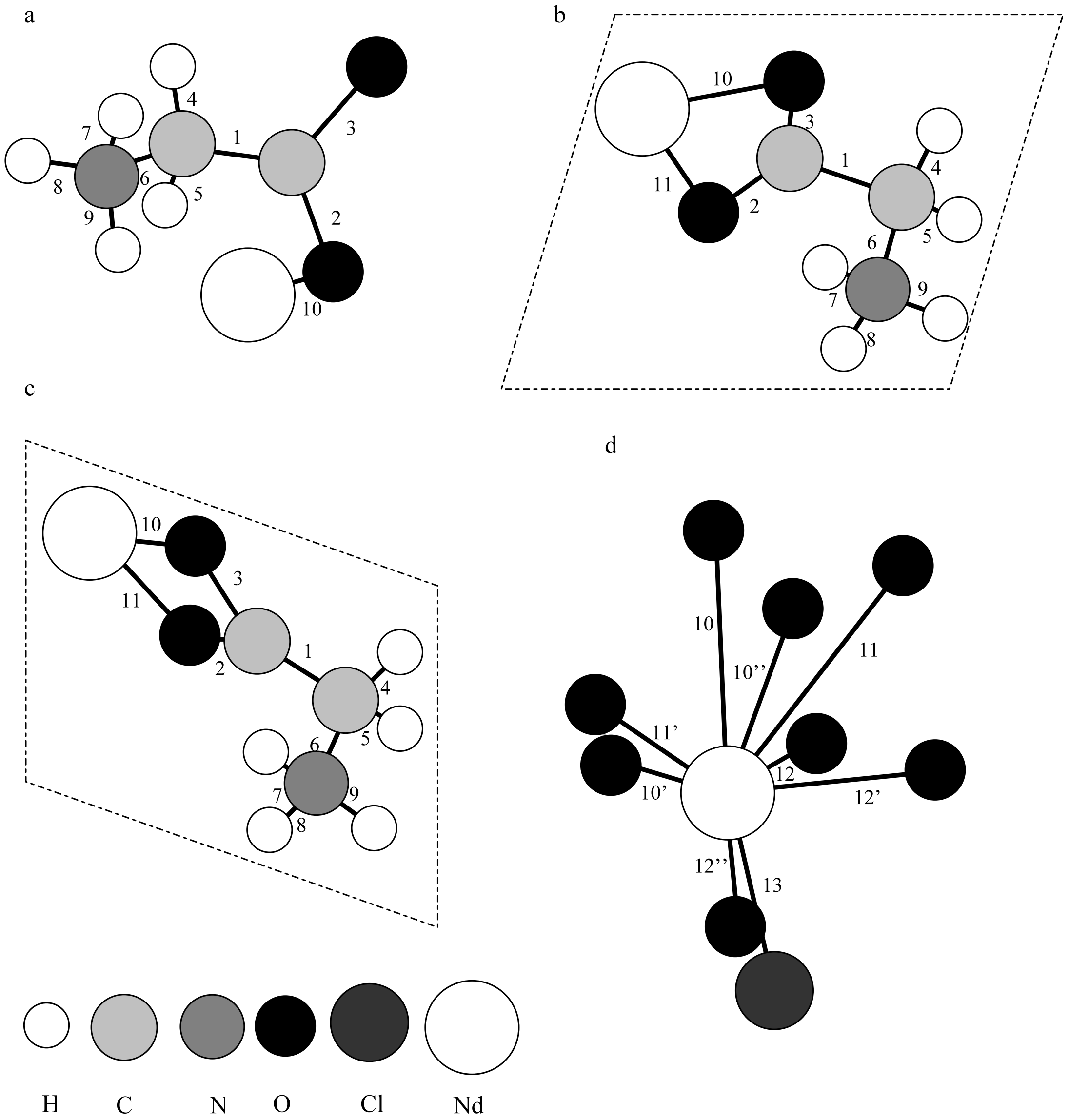

Fig. 2. Fragments of coordinated neodymium biglycinate polyhedron: (a) $G = 27A$; (b) $G = 17A' + 10A''$; (c) $G = 16A' + 11A''$; (d) $G = 24A$.

amino acid with d-elements which have coordination through oxygen of carboxyl groups and nitrogen of amino groups. The frequencies and forms of normal vibrations for neodymium biglycinate molecule were calculated by us using the previously suggested [2] group calculation technique. The idea of this method is that the heavy complexing ion damps the kinetic interactions of the separate ligands so that the spectrum of the real complex can be well described through its fragment groups. The peculiarity of such calculations is the invariability of force field constants in different models that describe the real complex (Fig. 2).

In our calculations we used force constants for a glycine molecule with the betaine structure (Tables 2 and 3) as initial data.

Data received from empirical equations [7] and refined during calculations are represented in Table 4. These data allowed us to define more precisely empirical assignments [7] and specify main bands in IR spectra. Measurement of Raman spectra complements the experimental data. Changes of intensity in IR and Raman bands allow us to evaluate the local symmetry of separate coordinated groups of ligands. In our calculations we used X-ray diffraction analysis data as the initial geometric data (Table 1). As one can see from the experimental data, the carboxyl group of the ligand has two absorption bands: the first at about 1600 cm^{-1} $\nu_{as}(COO^-)$ corresponds to symmetric stretching vibrations and the second at about 1400 cm^{-1} $\nu_s(COO^-)$ corresponds to asymmetric stretching vibrations. This fact as well as the absence of absorption bands for nonionised carboxyl groups means that the two oxygen atoms are indistinguishable and, consequently, that the electronic density in carboxyl group ligand is deloca-

Table 2
Force field constants of glycine $N^+H_3CH_2COO^-$

Vibration coordinate	10^6 (cm^{-2})	Vibration coordinate	10^6 (cm^{-2})	Vibration coordinate	10^6 (cm^{-2})
K_1	7.02	$H_3{}^2$	0.92	$A_3{}^{1,3}$	0.43
K_2	9.15	$H_4{}^1$	0.25	$A_4{}^{1,4}$	0.20
K_3	9.15	$H_6{}^1$	0.35	$A_6{}^{1,6}$	0.20
K_4	8.34	$H_6{}^4$	0.20	$A_6{}^{7,8}$	0.20
K_6	8.60	$H_5{}^4$	0.30	$A_7{}^{6,7}$	0.18
K_7	10.77	$\mathrm{H}_7{}^6$	0.45	$A_7{}^{7,8}$	0.18
$K_{1,2}$	0.39	$H_8{}^7$	0.25	$A_{1,3}^{1,2}$	0.10
$K_{1,3}$	0.39	$A_1{}^{1,2}$	0.04	$A_{2,3}^{1,2}$	0.10
$K_{2,3}$	0.48	$A_1{}^{1,3}$	0.04	$A_{1,3}^{2,3}$	0.10
$K_{1,4}$	0.91	$A_1{}^{1,4}$	0.20	$A_{1,4}^{1,2}$	0.05
$K_{4,5}$	0.71	$A_1{}^{1,6}$	0.22	$A_{1,5}^{1,4}$	0.05
$K_{4,6}$	0.89	$A_2{}^{1,2}$	0.43	$A_{4,5}^{1,6}$	−0.05
$K_{6,7}$	0.88	$A_2{}^{1,3}$	0.43	$A_{6,8}^{6,7}$	0.05
$K_{7,8}$	0.73	$A_2{}^{2,3}$	0.43	$A_{7,9}^{7,8}$	−0.05
$H_2{}^1$	0.59	$A_3{}^{2,3}$	0.43	$A_{6,7}^{1,6}$	0.07
$H_3{}^1$	0.59	$A_3{}^{1,2}$	0.43	$A_{6,7}^{4,6}$	0.02

lised, and also that both oxygen atoms participate in coordination. That is why IR spectra confirm the formation of bidentate bridge and tridentate-bridge-cyclic bonding of carboxyl with the RE ion. The results of the calculation for model 2a are not represented in Table 4 because of the assumption of monodentate carboxyl group coordination. The results of the calculation for model 2c are almost equivalent to the results for model 2b (they differ only in vibration forms of the coordinated carboxyl group). Besides that, model 2b is fully equivalent to the fragment of the molecular complex of neodymium biglycinate, which has local C_S symmetry ($G = 17A' + 10A''$), and with bidentate coordination of the carboxyl group lying in the symmetry plane of the fragment.

Our aim was not to achieve complete coincidence of experimental and theoretical data, because the experiment was carried out for isolated molecules or fragments, and in the experiment we deal with spectra of the crystal structure containing not only absorption bands but also partial frequencies and overtones and also vibrations of the crystal lattice. Moreover one of the limits for our experiment was the usage of a single force field for all models.

Table 3
Force field of neodymium biglycinate $\{[Nd(HGl)_2(H_2O)_3Cl]Cl_2\}_2$

Vibration coordinate	10^6 (cm^{-2})	Vibration coordinate	10^6 (cm^{-2})
K_{10}	3.63	$K_{2,3}$	0.60
K_3	10.80	$K_{10,11}$	0.55
K_2	12.60	$H_2{}^{10}$	0.35
K_1	8.60	$A_{10,2}^{10}$	0.20
$K_{10,3}$	0.66	$A_{10,2}^{10,11}$	0.18

Model 2d (Fig. 2) corresponds to the polyhedron of the coordination of the neodymium ion ($G = 24A$). In the calculations we used a diagonal matrix of bonds and angles force constants, without nondiagonal elements (so their physical meanings are not clear for the chosen model). That is why frequencies and forms of vibrations for metal–ligand bonds and Nd–O–Nd′, O–Nd–O′ angles are only given in Table 4. At the same time the calculations made for the model 2d allowed us to distinguish Nd–O frequencies (for oxygen atoms of aqua and glycine in the internal coordination sphere) and to specify Nd–Cl frequencies [9,10]. The results of calculations for $Nd \leftarrow OH_2$ model are given in Table 4 in columns 7 and 8 in brackets.

4. Conclusion

As one can see in Table 4, the calculations are in good conformity with the experimental data. The following advantages of the calculations can be stressed: the approach of frequencies of $\nu_{as}(OH)$ and $\nu_s(OH)$ in molecules of coordinated aqua; the confirmation of ‘admixing’ of deformation vibrations of the ionised α-amino group in the range of absorption bands of $\nu_{as}(COO^-)$; localization of M–L absorption bands in long-wave (< 600 cm^{-1}) range. So we can come to the conclusion that the usage of the single set of force field constants allows to study different possible configurations of the glycine molecule in neodymium biglycinate. We suggest applying this method when there is no X-ray diffraction analysis data for complexes of RE with α-amino acids.

Table 4
Experimental and calculated frequencies and forms of normal vibrations for neodymium biglycinate

Experiment		Group attribution [1,5–8]	Calculation of tested models normal vibrations				
IR	Raman		$G=17A'+10A''$			$G=24A$	
			Calculated frequency	Form	Coordinate	Frequency (Nd–OH$_2$)	Coordinate
3410m	3422m	$\nu_{as}OH(H_2O)$				(3489)	$\nu_{as}OH(H_2O)$
3365s		$\nu_sOH(H_2O)$, $\nu_{as}NH_3$	3532	A'	q_9	(3416)	$\nu_sOH(H_2O)$
			3172	A''	$q_9+q_8-q_7$		
3070s	3010m	ν_sCH, $\nu_\alpha NH_3$	3070	A'	$q_9+q_8+q_7$		
2970s	2973m	$\nu_\alpha CH$, ν_sNH_3	2986	A''	q_4-q_5	-	-
	2968s						
	2940m	ν_sCH, ν_SNH_3	2914	A'	q_4+q_5		
	2845m						
1637w	1670w	$\delta(H_2O)$				(1621)	$\delta(H_2O)$
1618m		$\delta_{as}NH_3$					
1604s	1600s	$\nu_{as}COO^-$, $\delta_{as}NH_3$	1560	A''	q_2-q_3		
	1570m	$\nu_{as}COO^-$, $\delta_{as}NH_3$	1566	A'	$\alpha_{8,9}+\alpha_{9,7}-\alpha_{8,7}$		
1512m	1506m	δ_SNH_3	1478	A'	$\alpha_{8,9}+\alpha_{9,7}+\alpha_{8,7}$		
	1481m	$\delta_\alpha CH_2$					
	1458w	$\delta_\alpha CH_2$					
1445m	1444m	$\delta_\alpha CH_2$	1453	A'	$\alpha_{5,4}$		
1413s	1414m	ν_SCOO^-	1397	A'	q_2+q_3		
1338s	1357s	δ_wCH_2					
1318s	1303s	δ_tCH_2	1352	A'	q_5,q_4		
1284m		δ_SCH_2, δNCH					
1230w	1210m	δ_rNH_3					
1165w		δCCN, $\delta ONdCI$					
1140w	1140m	δ_rNH_3, ρ_wNH_3	1152	A''	$\alpha_{8,9}-\alpha_{9,7}-\alpha_{8,7}$		
1100m	1112m	$\delta_\rho H_2O$				(1112)	$\delta_\rho H_2O$
1042m	1037m	νCN	1033	A'	q_6		
1007m	1019m	δ_tNH_3	1120	A''	$\alpha_{8,9}-\alpha_{9,7}-\alpha_{8,7}$		
1016m	921m	νCC, δCCN	920	A'	$\alpha_{1,6}$		
876w	894m	νCC, ρ_SCOO^-, $\delta_r(H_2O)$	868	A'	q_1	(984)	$\delta_\rho H_2O$
830w	850m	δ_rNH_3, δCCN					
	775w	δ_rCH_2					
772m	700m	δCCN, δCCH, δNCH	746	A'	$\alpha_{1,4}$		
662m	650m	δ_bCOO^-	673	A''	$\alpha_{3,2}-\alpha_{3,1}+\alpha_{2,1}$		
615w	600m	δ_wCOO^-	574	A'	$\alpha_{3,1}+\alpha_{2,1}$		
580w	-	$\nu NdO(H_2O)$, δ_rCOO^-				(522)	$\nu NdO(H_2O)$
538w	530m	ρ_wH_2O, ρ_tH_2O				443–443	q_{12}, $q_{12'}$,$q_{10'}$
507w	506m	δCCN, δCNH, νNdO	528	A''	$\alpha_{7,6}$	467, 474	q_{11}, $q_{11'}$
460m		ρNH_3	492	A'	$\alpha_{8,9}+\alpha_{9,7}+\alpha_{8,7}$	461, 457	q_{10}, $q_{10''}$
426w	420w	$\nu NdCI$,ρ_SH_2O,$\nu NdO(H_2O)$				429	$q_{12''}$
376m	390m		368	A'	q_3-q_{11}		
332w	362m		353	A'	$\alpha_{2,3}$		
318m							
305m							
280w	284w	νNdO, $\delta OndO$					
258w	265m	νNdO, $\delta ONdO$					
241m		νNdO, $\delta ONdO$					
232m		$\nu NdCI$				283	q_{13}
219m	200w	$\delta CONd$, νNdO	178	A'	$\alpha_{10,11}$	186	$\alpha_{10'',10'}$
	195w	ρ_tCOO^-, ρ_tCH_2	172	A''	$\alpha_{10,11}$	133	$\alpha_{13,11}$
162w	166w	$\nu NdCI$				126	$\alpha_{10'',11'}$
	152w	ρ_wCONd				114,114	$\alpha_{11',13}$, $\alpha_{11,10'}$
130m	130m	$\nu NdCI$	130, 3	A''	$\alpha_{3,10}+\alpha_{2,11}+\alpha_{10,11}$	111, 109	$\alpha_{11,11'}$, $\alpha_{13,10''}$
	119m	$\rho(H_2O)$, $\nu NdCI$				108, 91	$\alpha_{13,10}$, $\alpha_{10,10''}$
	110w	νNdO	112	A''	$\alpha_{11,10}$	88	$\alpha_{11,10''}$
95m	89m	$\delta ONdO$, δCOO^-, δN^+H_3				74, 72	$\alpha_{13,12''}$, $\alpha_{11,10}$
	76w	$\delta N^+H_3\delta NdO$, $\delta ONdO$				68, 63	$\alpha_{13,10'}$, $\alpha_{13,12'}$
	53w					57	$\alpha_{13,12}$

w, weak; s, strong, m, medium.

5. Supplementary material

Supplementary data are available from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-123-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk) on request, quoting the deposition number 182136.

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