# Crystal Structure of a Novel Neodymium Hydroxide Perchlorate Hydrate, $Nd_2(OH)_3(ClO_4)_3 \cdot 5H_2O$

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The crystal structure of the title complex has been determined using X-ray diffraction. The monoclinic unit cell with the space group symmetry *C2/c* has the dimensions $a = 13.660(5)$, $b = 6.837(2)$, $c = 18.427(10)$ Å and $\beta = 102.62(5)°$ and contains four formula units. The structure is one of the few known solid inner-sphere complexes between a trivalent rare earth and perchlorate ions. The $Nd^{3+}$ cations are bridged by the perchlorate groups so as to form infinite layers. The mean value for the distances between the $Nd^{3+}$ ion and the four coordinating perchlorate oxygens is 2.42 Å. The excess of positive charge in the Nd perchlorate framework seems to be neutralized by the negative charge of hydroxo groups directly coordinated to the lanthanoid cations. The eight-coordination around the $Nd^{3+}$ cation is completed by water molecules. The average Nd–O(W) contact distance, concerning the hydroxo and the aqua oxygens, is 2.48 Å. The structural model has been refined to a final linear *R* value of 0.044 for 3058 reflections.

Information on the structures of hydrated or solvated rare earth cations can often be obtained from the study of their perchlorates, both in solutions and in crystals, since perchlorate ions have a low tendency to form inner-sphere complexes with lanthanoids.[1] Thus, for instance, an X-ray diffraction study of the isomorphous compounds of $Me(ClO_4)_3 \cdot 6H_2O$, where Me = La, Tb or Er, showed the lanthanoid cations octahedrally coordinated by water molecules and the perchlorate anions sitting in the voids between the trivalent rare earth aqua-ions.[2]

In the course of attempts to make single crystals of the lighter lanthanoid perchlorates, crystals of a neodymium perchlorate compound were grown, with properties appreciably different from those of other known lanthanoid perchlorates. These novel crystals show higher density, lower symmetry, significantly less solubility in water and relatively higher stability in air than those with the regular composition $Ln(ClO_4)_3 \cdot 6H_2O$. The crystals have been examined by X-ray diffraction and various spectroscopic methods. The X-ray study, presented here, was undertaken in order to obtain information about the composition and structure of this novel compound and to compare it with other known, related structures. The results of the spectroscopic investigations, such as IR, Raman and electronic spectroscopy at room and liquid helium temperature, will be published elsewhere.[3]

## Experimental

*Sample preparation.* The title compound was prepared by treatment of $Nd_2O_3$ with $HClO_4$, in an almost stoichiometric ratio, in a water bath. From the solution two types of crystals were precipitated: shortly after preparation and evaporation the 'normal' $Nd(ClO_4)_3 \cdot 6H_2O$ crystallized, while single crystals of the new compound grew when the solution had been left standing for a long time. The pH of the final solution was in the range 2–3 and the $Nd^{3+}$ concentration was approximately 2 M. The lanthanoid concentration in the crystals was determined by complexometric titration. The crystal density, measured by the flotation method in a mixture of bromoform and 1,2-dibromoethane, was found to be 2.83 g $cm^{-3}$.

*X-ray data collection and reduction.* A total of 4082 reflections were collected (Mo$K\alpha$ radiation, $\lambda = 0.71069$ Å, $\theta_{max} = 30°$, $\omega$–$2\theta$ scan technique) from a selected light-violet single crystal of the title compound ($Nd_2Cl_3O_{20}H_{13}$, $M_w = 727.93$, $D_c = 2.88$ g $cm^{-3}$). The intensitites were corrected for background, Lorentz and polarization effects as well as for absorption ($\mu = 63.2$ $cm^{-1}$). The absorption corrections were based upon $\psi$ scans of reflections near $\chi = 90°$. The crystals showed limited stability in air. The selected single crystal, sealed in epoxy resin, proved to be stable during the time of data collection (~1 week), but decomposed slowly during the weeks after.

The crystals are monoclinic with the space group symmetry *C2/c*. The unit cell, which contains four formula

*Table 1.* Fractional atomic coordinates and equivalent isotropic/isotropic temperature factors for the atoms. Estimated standard deviations, where given, are in parentheses.

| Atom | *x/a* | *y/b* | *z/c* | ($U_{eq}$[c]/*U*) |
|---|---|---|---|---|
| Nd | 0.33142(1) | 0.52447(3) | 0.10746(1) | 0.0111(1) |
| O(1W) | 0.1511(3) | 0.4849(4) | 0.0417(2) | 0.019(1) |
| O(2W) | 0.2395(3) | 0.4953(6) | 0.2043(2) | 0.030(1) |
| O(3W) | 0.4617(3) | 0.7356(5) | 0.0643(2) | 0.019(1) |
| O(4W) | 0.4866(3) | 0.3341(6) | 0.1420(3) | 0.033(1) |
| Cl(1) | 0.2848(1) | 0.4691(2) | −0.0890(1) | 0.0136(2) |
| O(11) | 0.3563(3) | 0.4178(5) | −0.0174(2) | 0.018(1) |
| O(12) | 0.2438(3) | 0.6621(5) | −0.0788(2) | 0.025(1) |
| O(13) | 0.2033(3) | 0.3245(5) | −0.1032(2) | 0.020(1) |
| O(14) | 0.3381(3) | 0.4680(6) | −0.1491(2) | 0.024(1) |
| Cl(2)[a] | 0.5000 | 0.8274(2) | 0.2500 | 0.0146(3) |
| O(21) | 0.5328(3) | 0.9492(6) | 0.1940(2) | 0.022(1) |
| O(22) | 0.4157(3) | 0.7033(6) | 0.2131(2) | 0.026(1) |
| H(1A) | 0.1359 | 0.5776 | 0.0191 | 0.01(1) |
| H(1B) | 0.1411 | 0.3898 | 0.0231 | 0.05(3) |
| H(2A) | 0.2804 | 0.5373 | 0.2499 | 0.09(4) |
| H(2B) | 0.1602 | 0.4722 | 0.1926 | 0.04(3) |
| H(3A) | 0.4824 | 0.8150 | 0.0978 | 0.03(2) |
| H(3B)[b] | 0.4426 | 0.8658 | 0.0381 | 0.34(20) |
| H(4A) | 0.4940 | 0.2253 | 0.1570 | 0.09(4) |

[a]Atom Cl(2) has the special position (0.5, *y*, 0.25), with s.o.f. = 0.5. [b]Atom H(3B) was assigned 50 % site occupancy because of the charge neutrality condition. [c]$U_{eq} = \frac{1}{3}\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* a_i a_j$.

units, has the dimensions $a = 13.660(5)$, $b = 6.837(2)$, $c = 18.427(10)$ Å and $\beta = 102.62(5)°$. The unit cell parameters were refined against the angular settings of 49 accurately centered reflections within the range $12 < 2\theta < 37°$.

*Solution and refinement of the structure.* The crystallographic asymmetric unit contains only one half of the formula unit, with one of the chlorine atoms in a special position [(0.5, *y*, 0.25) and s.o.f. = 0.5]. The position of the neodymium ion was derived from a Patterson map. Subsequent difference Fourier syntheses and full-matrix least-squares calculations with the SHELX program system[4] were used to complete and refine the structural model.

The positions of the non-hydrogen atoms were refined together with their anisotropic thermal parameters. The H atoms, also derived from difference electron density maps, were held fixed; only individual isotropic temperature factors were refined for them. One hydrogen, H(3B), with the lowest peak height and with a considerably higher temperature factor than those of the other H atoms (cf. Table 1), was assigned 50 % site occupancy because of the charge neutrality condition. Note that this reduction of the s.o.f. of H(3B) also proved to be a necessary condition for satisfactory convergence of the refinement calculation. The last refinement, including 123 variables and based on 3058 reflections with $F > 10\sigma(F)$, in which an empirical extinction correction factor[4] was also refined, converged to the final agreement factors $R = \Sigma|\Delta F|/\Sigma|F_o| = 0.044$, $R_w = \Sigma\sqrt{w}|\Delta F|/\Sigma\sqrt{w}|F_o| = 0.045$ and $R_G = [\Sigma w|\Delta F|^2/\Sigma w|F_o|^2]^{1/2} = 0.061$. The weights of the structure factors were calculated as $w = 1.0/[\sigma^2(F) + 0.0057F^2]$, where $\sigma(F)$ is derived from counting statistics. The atomic scattering factors for the O and Cl atoms were taken from Cromer and Mann,[5] those for the $Nd^{3+}$ ion from Cromer and Waber[8] and those for the H atoms from Stewart *et al.*[7]

The final atomic coordinates are listed in Table 1. The labelling of the atoms is shown in Fig. 1. Lists of the anisotropic thermal parameters of the non-hydrogen atoms as well as the observed and calculated structure factors may be obtained from the authors (I. C., A. E. and P. K.).

*Crystallographic description of the structure, and discussion.* Fig. 2 is a stereo illustration of the coordination polyhedron around the $Nd^{3+}$ cation; Table 2 lists the Nd–O and Cl–O contact distances, both uncorrected and corrected for riding motion,[8] and the angles formed by these bonds. The neodymium ions are eight-coordinated. Four of the ligand oxygens belong to four perchlorate groups, while the four O(W) atoms are provided by the water medium. The O(W) atoms form a distorted, elongated tetrahedron around the neodymium cation, with a mean Nd–O(W) distance of 2.48[7] Å (the r.m.s.d. is given in square brackets). The coordinating perchlorate oxygens also have a somewhat irregular tetrahedral arrangement, with a slightly shorter mean Nd–O distance, 2.42[6] Å. A similar distribution of metal–oxygen distances has been observed also in some lanthanoid sulfate hydrates, such as $Nd_2(SO_4)_3 \cdot 5H_2O$,[9] $Nd_2(SO_4)_3 \cdot 8H_2O$[10] and $Ce_2(SO_4)_3 \cdot 4H_2O$.[11] The calculated arithmetic means of the Nd–O contact distances in the present study, 2.45[6] Å for the uncorrected ones and

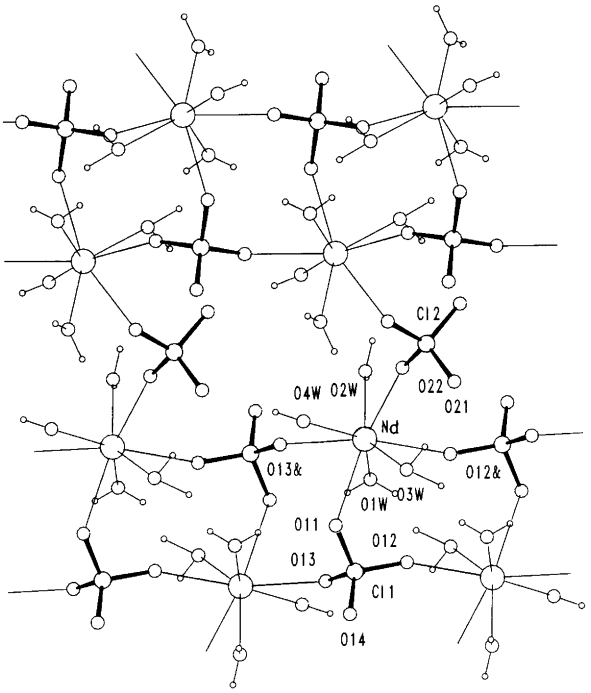


*Fig. 1.* Perspective view of a characteristic layer fragment of the $Nd_2(OH)_3(ClO_4)_3 \cdot 5H_2O$ crystal. The atoms of the crystallographic asymmetric unit are labelled as in the text ('&' denotes symmetry-related equivalent atomic positions).

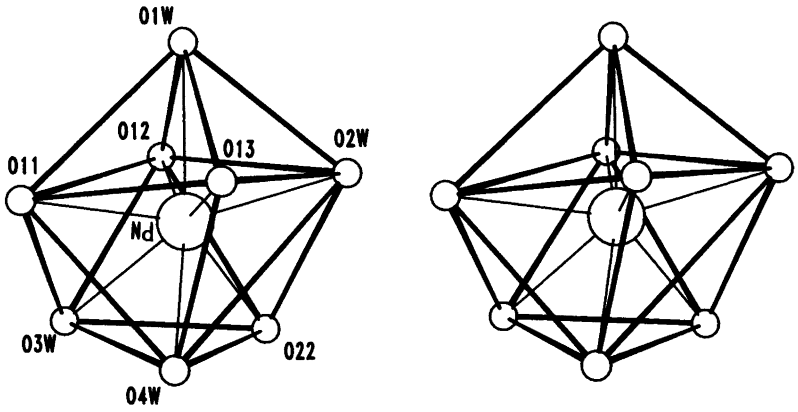


*Fig. 2.* Stereoscopic view of the coordination polyhedron around the $Nd^{3+}$ cation.

*Table 2.* Nd–O coordination distances and O–Nd–O angles, and covalent bond lengths and bond angles within the perchlorate groups. Estimated standard deviations, where given, are in parentheses.

| Atoms | Distances / Å | |
|---|---|---|
| | Uncorrected | Corrected for riding motion[c] |
| Nd–O(1W) | 2.511(4) | 2.514 |
| Nd–O(2W) | 2.404(5) | 2.415 |
| Nd–O(3W) | 2.549(4) | 2.553 |
| Nd–O(4W) | 2.451(4) | 2.463 |
| Nd–O(11) | 2.506(4) | 2.509 |
| Nd–O(12)[a] | 2.385(4) | 2.394 |
| Nd–O(13)[b] | 2.430(4) | 2.434 |
| Nd–O(22) | 2.374(4) | 2.382 |
| Cl(1)–O(11) | 1.501(3) | 1.506 |
| Cl(1)–O(12) | 1.461(4) | 1.474 |
| Cl(1)–O(13) | 1.469(4) | 1.476 |
| Cl(1)–O(14) | 1.454(4) | 1.464 |
| Cl(2)–O(21) | 1.471(4) | 1.479 |
| Cl(2)–O(22) | 1.472(4) | 1.484 |

| Atoms involved | Angle / ° | Atoms involved | Angle / ° |
|---|---|---|---|
| O(13)[b]–Nd–O(22) | 126.0(1) | O(12[a]–Nd–O(22) | 80.0(2) |
| O(12)[a]–Nd–O(13)[b] | 143.4(2) | O(11)–Nd–O(22) | 139.8(2) |
| O(11)–Nd–O(13)[b] | 75.5(1) | O(11)–Nd–O(12)[a] | 101.4(2) |
| O(4W)–Nd–O(22) | 79.8(2) | O(4W)–Nd–O(13)[b] | 68.8(2) |
| O(4W)–Nd–O(12)[a] | 147.3(2) | O(4W)–Nd–O(11) | 78.5(2) |
| O(3W)–Nd–O(22) | 73.6(2) | O(3W)–Nd–O(13)[b] | 133.6(2) |
| O(3W)–Nd–O(12)[a] | 73.7(1) | O(3W)–Nd–O(11) | 68.6(1) |
| O(3W)–Nd–O(4W) | 76.0(2) | O(2W)–Nd–O(22) | 70.9(2) |
| O(2W)–Nd–O(13)[b] | 79.2(2) | O(2W)–Nd–O(12)[a] | 88.2(2) |
| O(2W)–Nd–O(11) | 148.7(2) | O(2W)–Nd–O(4W) | 109.0(2) |
| O(2W)–Nd–O(3W) | 142.4(2) | O(1W)–Nd–O(22) | 134.4(2) |
| O(1W)–Nd–O(13)[b] | 73.4(1) | O(1W)–Nd–O(12)[a] | 70.1(1) |
| O(1W)–Nd–O(11) | 80.9(1) | O(1W)–Nd–O(4W) | 140.4(1) |
| O(1W)–Nd–O(3W) | 126.0(1) | O(1W)–Nd–O(2W) | 74.5(2) |
| O(11)–Cl(1)–O(14) | 109.3(3) | O(11)–Cl(1)–O(12) | 107.1(2) |
| O(11)–Cl(1)–O(13) | 108.8(2) | O(12)–Cl(1)–O(13) | 109.5(2) |
| O(12)–Cl(1)–O(14) | 111.8(2) | O(13)–Cl(1)–O(14) | 110.2(2) |
| O(21)–Cl(2)–O(22) | 109.3(2) | | |

The oxygen positions are generated using the coordinates of Table 1, with the following symmetry operations:

| Superscript | Symmetry operation |
|---|---|
| none | *x*, *y*, *z* |
| [a] | 0.5−*x*, 1.5−*y*, −*z* |
| [b] | 0.5−*x*, 0.5−*y*, −*z*. |

[c]Corrections according to Ref. 8.

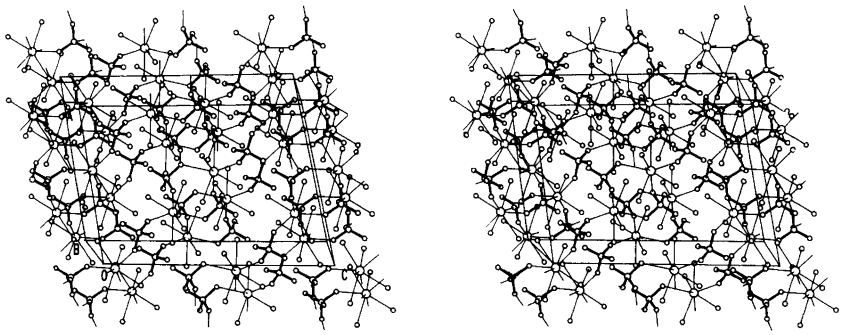

*Fig. 3.* Stereoscopic packing illustration of the crystal structure of $Nd_2(OH)_3(ClO_4)_3 \cdot 5H_2O$. The hydrogens are omitted for clarity.

2.46[6] Å for the values corrected for riding motion,[8] are comparable with the mean Nd–O distances observed in $RbNd(PO_3)_4$ (2.438 Å)[12] and in $CaKNd(PO_4)_2$ (2.472 Å)[13] but are slightly shorter than the mean values published for $Nd_2(SO_4)_3$ (2.488 Å),[14] for $Nd_2(SO_4)_3 \cdot 5H_2O$ (2.50 Å)[9] and for $[Nd(H_2O)_9](CF_3SO_3)_3$ (2.495 Å)[15] and significantly shorter than the corresponding values published for the three crystallographically independent $Nd^{3+}$ ions in the $Na_3Nd(VO_4)_2$ crystal (2.54, 2.53 and 2.52 Å).[16]

The coordination polyhedron (cf. Fig. 2) is not a regular one. It seems to be an intermediate which can approximately be described either as a strongly distorted dodecahedron (Dod) with A and B sites occupied by the O(W) and the perchlorate oxygens, respectively; or by a deformed bicapped trigonal prism (BTCP) with atoms O(1W) and O(3W) on the cap.[17] Characteristic dihedral angles, δ and φ for Dod, and δ for BTCP, calculated according to Drew,[17] are listed in Table 3. The lengths of the polyhedron edges vary between 2.758[5] and 3.953[6] Å, with a mean value of 3.10[32] Å.

The crystal structure is a beautiful example of an $Nd^{3+}$–$ClO_4^-$ inner-sphere complex. This is surprising, because the crystals were grown from water solution. In weakly to moderately coordinating solvents spectroscopic studies recently confirmed the formation of perchlorate–lanthanoid inner-sphere complexes.[18] In water solutions, however, the perchlorate ions usually have no tendency to enter the inner sphere of the rare earth cations.[1,19,20] An exception is the recently published crystal structure of praseodymium glutamate perchlorate hydrate,[20] where one of the perchlorate groups was found to be directly coordinated to the rare earth cation. Nevertheless, in the related holmium and dysprosium complexes[21,22] the perchlorate anions are completely shielded from the lanthanoid

*Table 3.* Dihedral angles between selected planes of the coordination polyhedron around the neodymium cation.[a] Estimated standard deviations are given in parentheses.

| Atoms defining the planes | | Angle / ° |
|---|---|---|
| Plane 1 | Plane 2 | |
| Assuming Dod symmetry | | |
| O(1W)–O(13)–O(11) | O(13)–O(11)–O(4W) | $\delta_{1(57)3} = 52.7(2)$ |
| O(1W)–O(12)–O(11) | O(12)–O(11)–O(3W) | $\delta_{1(67)4} = 20.2(2)$ |
| O(2W)–O(13)–O(22) | O(13)–O(22)–O(4W) | $\delta_{2(58)3} = 18.5(2)$ |
| O(2W)–O(12)–O(22) | O(12)–O(22)–O(3W) | $\delta_{2(68)4} = 49.4(2)$ |
| $O(11)–O(22)–\frac{O(1W)+O(2W)}{2}$ | $O(1W)–O(2W)–\frac{O(11)+O(22)}{2}$ | $\varphi_{12/78} = 25.7(2)$ |
| $O(13)–O(12)–\frac{O(4W)+O(3W)}{2}$ | $O(4W)–O(3W)–\frac{O(13)+O(12)}{2}$ | $\varphi_{34/56} = 21.8(2)$ |
| Assuming BTCP symmetry | | |
| O(11)–O(3W)–O(1W) | O(3W)–O(1W)–O(12) | $\delta_{1(57)3} = 24.0(2)$ |
| O(11)–O(13)–O(1W) | O(13)–O(1W)–O(2W) | $\delta_{1(67)4} = 58.4(2)$ |
| O(4W)–O(3W)–O(22) | O(3W)–O(22)–O(12) | $\delta_{2(58)3} = 59.2(2)$ |
| O(4W)–O(13)–O(22) | O(13)–O(22)–O(2W) | $\delta_{2(68)4} = 18.5(2)$ |

[a]Calculated according to Ref. 17.

*Table 4.* Bond distances (Å) and angles (°) in possible hydrogen bonds. Estimated standard deviations, where given, are in parentheses.

| Atoms involved | O···O | O–H | H···O | ∢ O–H···O |
|---|---|---|---|---|
| O(1W)–H(1A)···O(3W)[a] | 2.920(4) | 0.762 | 2.208 | 156 |
| O(1W)–H(1B)···O(11)[b] | 2.788(4) | 0.734 | 2.106 | 155 |
| O(2W)–H(2A)···O(14)[c] | 2.754(5) | 0.946 | 1.856 | 157 |
| O(2W)–H(2B)···O(21)[d] | 2.805(6) | 1.069 | 1.752 | 167 |
| O(3W)–H(3A)···O(21)[e] | 2.787(5) | 0.824 | 1.981 | 166 |
| O(3W)–H(3B)···O(1W)[a] | 2.920(4) | 1.019 | 2.005 | 148 |
| O(4W)–H(4A)···O(21)[f] | 2.824(5) | 0.792 | 2.038 | 172 |

Symmetry operations: [a] 0.5−*x*, 1.5−*y*, −*z*. [b] 0.5−*x*, 0.5−*y*, −*z*. [c] *x*, 1−*y*, 0.5+*z*. [d] −0.5+*x*, −0.5+*y*, *z*. [e] *x*, *y*, *z*. [f] *x*, −1+*y*, *z*.

cation, as usual. The perchlorate groups often occur in a more or less disordered form in crystal structures.[2,22] However, the directly coordinated $ClO_4^-$ ions in the present structure, as well as in that of the praseodymium complex, show no sign of rotational disorder. The mean Cl–O distances in the present study, 1.48[2] Å for the bond lengths corrected for riding motion[8] and 1.47[2] Å for the uncorrected ones, are slightly longer than 1.44 Å, the commonly accepted average bond length for such bonds.[23]

The neodymium ions are bridged by the perchlorate groups so that eight- and sixteen-membered rings are formed, which are fused into a layer structure (cf. Fig. 1) in the following way: the perchlorate groups containing Cl(1) coordinate with three of their oxygens each to three different $Nd^{3+}$ ions, forming the smaller rings. These rings are fused into infinite zig-zag chains in the direction of the *b* axis. The chains are then crosslinked via the perchlorate groups containing Cl(2), which coordinate by only two of their four O atoms. Thus, the larger sixteen-membered rings are formed by two tridentate and two bidentate $ClO_4^-$ anions and four neodymium cations. In the layer of the neodymium–perchlorate framework the excess of positive charge seems to be neutralized by the negative charge of hydroxo groups directly coordinated to the rare earth ions. The coordination around the $Nd^{3+}$ cation is completed by water molecules, which together with the hydroxo groups also stabilize the crystal structure by forming hydrogen bonds (cf. Table 4).

In summary, the crystal structure presented gives evidence that perchlorate groups can compete successfully with water molecules in coordination to rare earth cations.

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