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Redetermination of $[Gd(NO_3)_3(H_2O)_4]\cdot 2H_2O$

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Key indicators: single-crystal X-ray study; T = 173 K; mean σ(O–N) = 0.004 Å; R factor = 0.016; wR factor = 0.041; data-to-parameter ratio = 10.2.

The crystal structure of the title compound, tetraaquatris(nitrato-κ^2O,O')gadolinium(III) dihydrate, was redetermined from single-crystal X-ray data. In comparison with the first determination [Ma *et al.* (1991). *Wuji Huaxue Xuebao*, **7**, 351–353], all H atoms could be located, accompanied with higher accuracy and precision. The Gd^{III} atom shows a ten-coordination with three nitrate ligands behaving in a bidentate manner and the other positions being occupied by four water molecules, forming a distorted bicapped square antiprism. Two nitrate ions coordinate to the metal atom with similar bond lengths while the third shows a more asymmetric bonding behaviour. An intricate network of O—H···O hydrogen bonds, including the lattice water molecules, stabilizes the crystal packing.

Related literature

For a previous determination of the title compound, see: Ma *et al.* (1991). Isotypic $[RE(NO_3)_3(H_2O)_4]\cdot 2H_2O$ structures were described for RE = Nd by Rogers *et al.* (1983), for Tb by Moret *et al.* (1990), for Sm by Kawashima *et al.* (2000), for Eu by Stumpf & Bolte (2001), for Dy by Gao *et al.* (1990), and for La by Eriksson *et al.* (1980). $[RE(NO_3)_3(H_2O)_4]\cdot H_2O$ structures with one less water molecule were described for RE = Eu by Ribár *et al.* (1986), for Gd by Stockhause & Meyer (1997), and for Yb by Junk *et al.* (1999).

Experimental

Crystal data

$[Gd(NO_3)_3(H_2O)_4]\cdot 2H_2O$
M_r = 451.38
Triclinic, $P\bar{1}$
a = 6.6996 (2) Å
b = 9.1145 (3) Å
c = 11.6207 (3) Å
α = 69.8257 (10)°
β = 88.9290 (11)°
γ = 69.2170 (11)°
V = 618.36 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.45 mm^{-1}
T = 173 K
0.48 × 0.46 × 0.23 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.349, T_{max} = 0.745
19731 measured reflections
2256 independent reflections
2255 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.041
S = 1.20
2256 reflections
221 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 1.04 e Å^{-3}
$\Delta\rho_{min}$ = −1.10 e Å^{-3}

Table 1
Selected bond lengths (Å).

Gd1—O1	2.528 (2)	Gd1—O8	2.754 (2)
Gd1—O2	2.494 (3)	Gd1—O10	2.398 (2)
Gd1—O4	2.578 (2)	Gd1—O11	2.389 (2)
Gd1—O5	2.518 (3)	Gd1—O12	2.392 (3)
Gd1—O7	2.552 (2)	Gd1—O13	2.364 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O10—H1···O15^{i}	0.76 (5)	1.97 (5)	2.720 (4)	171 (5)
O10—H2···O4^{ii}	0.75 (4)	2.21 (4)	2.956 (3)	178 (6)
O11—H3···O14^{iii}	0.82 (4)	1.85 (4)	2.671 (3)	176 (5)
O11—H4···O7^{iv}	0.78 (5)	2.19 (5)	2.967 (4)	173 (5)
O12—H5···O4^{v}	0.77 (6)	2.50 (6)	3.185 (3)	149 (5)
O12—H5···O7^{v}	0.77 (6)	2.56 (6)	3.156 (4)	135 (5)
O12—H6···O8^{vi}	0.83 (5)	2.27 (5)	3.074 (3)	162 (4)
O12—H6···O9^{vi}	0.83 (5)	2.39 (5)	3.062 (4)	139 (4)
O13—H7···O15	0.90 (4)	1.84 (4)	2.721 (3)	169 (5)
O13—H8···O14	0.71 (4)	2.04 (4)	2.738 (4)	168 (5)
O14—H9···O9^{iv}	0.81 (5)	2.03 (5)	2.826 (4)	167 (4)
O14—H10···O3^{v}	0.76 (5)	2.45 (5)	3.008 (4)	132 (5)
O14—H10···O5^{vii}	0.76 (5)	2.31 (5)	2.888 (4)	134 (5)
O15—H11···O6^{ii}	0.80 (6)	2.02 (6)	2.819 (4)	176 (6)
O15—H12···O3^{viii}	0.76 (6)	2.30 (6)	2.903 (4)	139 (5)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, -y+1, -z$; (iv) $-x+2, -y+1, -z$; (v) $x-1, y, z$; (vi) $-x+1, -y+2, -z$; (vii) $x, y-1, z$; (viii) $-x+2, -y, -z+1$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2634).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Bruker (2008). *APEX*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Eriksson, B., Larsson, L. O., Niinisto, L. & Valkonen, J. (1980). *Inorg. Chem.* **19**, 1207–1210.

Gao, S., Ma, H. & Yang, Z. (1990). *J. Northwest Univ. (China)*, **20**, 53–58.

Junk, P. C., Kepert, D. L., Skelton, B. W. & White, A. H. (1999). *Aust. J. Chem.* **52**, 497–505.

Kawashima, R., Sasaki, M., Satoh, S., Isoda, H., Kino, Y. & Shiozaki, Y. (2000). *J. Phys. Soc. Jpn*, **69**, 3297–3303.

Ma, H., Gao, S. & Zupci, Y. (1991). *Wuji Huaxue Xuebao*, **7**, 351–353.

Moret, E., Bünzli, J.-C. G. & Schenk, K. J. (1990). *Inorg. Chim. Acta*, **178**, 83–88.

Ribár, B., Kapor, A., Argay, Gy. & Kálmán, A. (1986). *Acta Cryst.* C**42**, 1450–1452.

Rogers, D. J., Taylor, N. J. & Toogood, G. E. (1983). *Acta Cryst.* C**39**, 939–941.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Stockhause, S. & Meyer, G. (1997). *Z. Kristallogr. New Cryst. Struct.* **212**, 315.

Stumpf, T. & Bolte, M. (2001). *Acta Cryst.* E**57**, i10–i11.

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supplementary materials

Acta Cryst. (2012). E**68**, i56–i57 [doi:10.1107/S1600536812028000]

Redetermination of $[Gd(NO_3)_3(H_2O)_4]\cdot 2H_2O$

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Comment

Single crystals of gadolinium nitrate hexahydrate, $[Gd(NO_3)_3(H_2O)_4]\cdot 2H_2O$, were obtained and its structure was re-determined using single-crystal X-ray diffraction analysis.

As shown in Figure 1, the Gd^{III} atom is ten-coordinated, being bound to six nitrate-oxygen atoms, O1, O2, O4, O5, O7, and O8, and four water-oxygen atoms O10, O11, O12, and O13. The nitrate ions are coordinated to the central gadolinium atom in a bidentate mode and show clearly the changes in bond lengths and angles noted previously for the isotypic Eu(III) structure (Stumpf & Bolte, 2001). The distances between the Gd^{III} ion and the nitrate-O atoms (Gd—O) are in the range 2.494 (3)–2.754 (2) Å, with a mean value of 2.571 Å. The differences between the Gd^{III} atom and the two oxygen atoms of the same nitrate group are 0.034 Å and 0.060 Å for nitrate N1 and nitrate N2, respectively. Nitrate N1 and nitrate N2 groups appear to be more symmetrically bonded to the Gd^{III} atom because the third nitrate group N3 exhibits one Gd—O distance that is 0.202 Å longer than the other. This asymmetric bonding seem to be associated with a steric effect of the coordinating water molecules. There is simply not space enough for all the oxygen atoms around the Gd^{III} atom at the same distance. The Gd—O$_{water}$ distances are in the range of 2.364 (3)–2.398 (2) Å with an average distance of 2.386 Å. Hence the water molecules are closer to the Gd^{III} atom than the nitrate groups by *ca.* 0.185 Å.

These results are comparable to that reported for other lanthanide complexes with bidentately coordinating nitrate groups. The coordination polyhedron around the Gd^{III} ion can be best described as a distorted bi-capped square antiprism (Fig. 1), as previously reported for other hydrated lanthanide(III) nitrate complexes (Ribár *et al.*, 1986; Moret *et al.*, 1990; Ma *et al.*, 1991, Stockhause & Meyer, 1997; Kawashima *et al.*, 2000; Stumpf & Bolte, 2001; Junk *et al.*, 1999; Gao *et al.*, 1990; Eriksson *et al.*, 1980; Rogers *et al.*, 1983). The structure contains additional two lattice water molecules, which are associated with the complex by a network of hydrogen bonds. All hydrogen atoms and except for O1 and O2 all oxygen atoms are involved in a complicated network of O—H···O hydrogen bonds, stabilizing the crystal packing (Table 1, Fig. 2). H5, H6 and H10 act as bifurcated bridging atoms. In contrast to the original work of Ma *et al.* (1991), we report much more precise results. All hydrogen atoms could be located in the difference Fourier maps and were allowed to refine freely. The final *R*1 value changed from 0.076 to 0.0158 and the overall e.s.d.'s for the positional parameters dropped from *e.g.* Gd1 *x* = 0.8016 (1) to 0.80352 (2). The same is valid for distances *e.g.* Gd1—O1 dropped from 2.558 (56) Å to 2.528 (2) Å and for the angels *e.g.* O1—Gd1—O2 dropped from 51.1 (22) to 50.56 (7).

Experimental

Single crystals of $[Gd(NO_3)_3(H_2O)_4]\cdot 2H_2O$ have been obtained accidentally during the synthesis of a Gd—*N*,*N*-bis-(salicylicaldehyde)-*o*-phenylenediamine Schiff base complex. 1.0 mmol of the Schiff base were dissolved in 10 ml chloroform. To this solution was added in a drop-wise manner a 10 ml ethyl acetate solution of 2.0 mmol $Gd(NO_3)_3\cdot 6(H_2O)$. The reaction mixture was stirred for 2 h at room temperature. The yellow precipitate was filtered, washed several times with ethyl acetate and chloroform. Single crystals suitable for X-ray were obtained after a few days by the slow evaporation of the solvent in an open atmosphere.

Refinement

Hydrogen atoms could be located in difference Fourier maps and were allowed to refine freely. The residual electron density of $\Delta\rho$max = 1.04 e Å^{-3} is located 0.89 Å next to Gd1, whereas $\Delta\rho$min = 1.10 e Å^{-3} is located 0.96 Å next to Gd1.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON* (Spek, 2009).

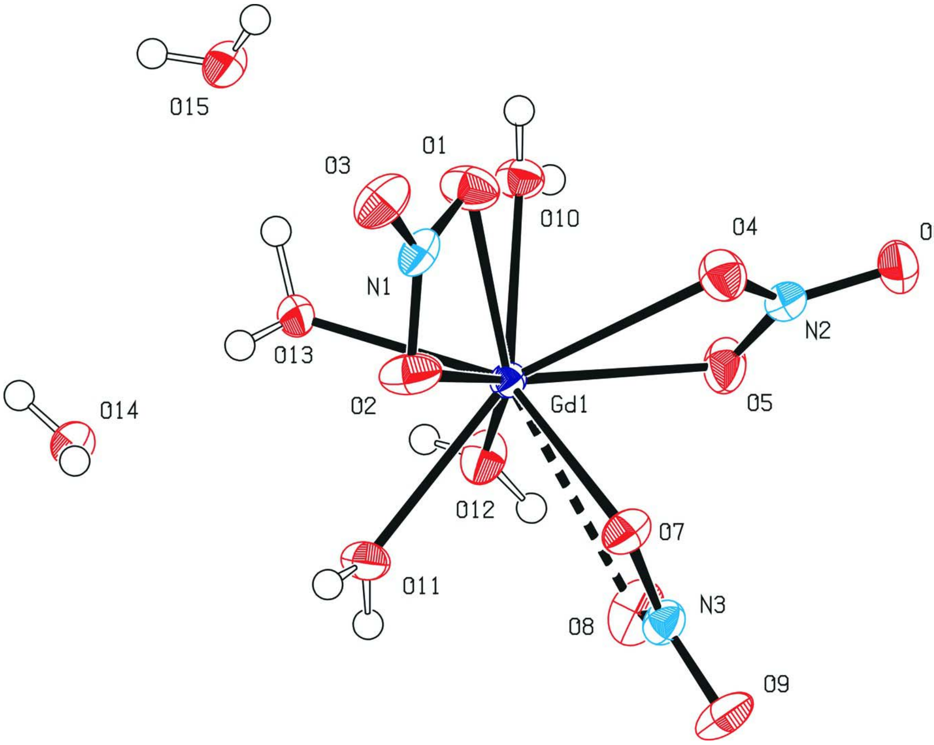

Figure 1

View of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

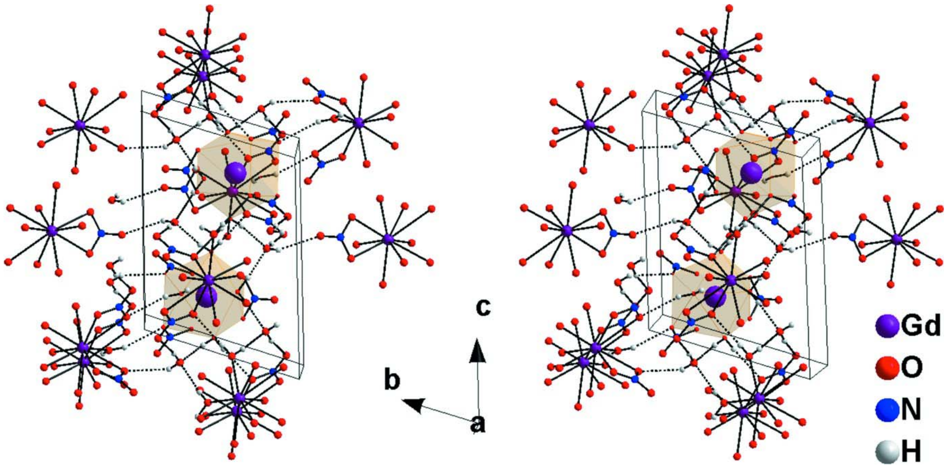

Figure 2

Stereo plot of the expanded unit cell. Hydrogen bonds (dotted lines) are limited to a distance O···H of 2.35 Å.

Tetraaquatris(nitrato-κ^2O,O')gadolinium(III) dihydrate

Crystal data

$[Gd(NO_3)_3(H_2O)_4]\cdot 2H_2O$
M_r = 451.38
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.6996 (2) Å
b = 9.1145 (3) Å
c = 11.6207 (3) Å
α = 69.8257 (10)°
β = 88.9290 (11)°
γ = 69.2170 (11)°
V = 618.36 (3) Å^3

Z = 2
$F(000)$ = 434
D_x = 2.424 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9802 reflections
θ = 1.9–25.4°
μ = 5.45 mm^{-1}
T = 173 K
Fragment, orange
0.48 × 0.46 × 0.23 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: rotating anode FR591
Graphite monochromator
Detector resolution: 16 pixels mm^{-1}
phi– and ω–rotation scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.349, T_{max} = 0.745

19731 measured reflections
2256 independent reflections
2255 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 25.4°, θ_{min} = 1.9°
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.041
S = 1.20
2256 reflections
221 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(F_o^2) + (0.0186P)^2 + 0.725P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.04 e Å^{-3}
$\Delta\rho_{min}$ = −1.10 e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$	Extinction coefficient: 0.0129 (7)

Special details

Experimental. Diffractometer operator E. Herdtweck scanspeed 10 s per frame dx 45 4932 frames measured in 9 data sets phi-scan with delta_phi = 0.50 omega-scans with delta_omega = 0.50

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except for 0 with very negative F^2 or flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating R_factor_obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Gd1	0.80352 (2)	0.59425 (1)	0.22568 (1)	0.0147 (1)
O1	1.0817 (4)	0.3431 (3)	0.3878 (2)	0.0294 (7)
O2	1.1029 (4)	0.3531 (3)	0.20104 (19)	0.0286 (6)
O3	1.3338 (4)	0.1425 (3)	0.3490 (2)	0.0304 (6)
O4	1.0578 (3)	0.6801 (3)	0.3296 (2)	0.0268 (6)
O5	0.7353 (4)	0.8617 (3)	0.2653 (2)	0.0305 (7)
O6	0.9570 (4)	0.9177 (3)	0.3616 (2)	0.0319 (7)
O7	1.0495 (3)	0.6937 (3)	0.07347 (19)	0.0245 (6)
O8	0.7274 (4)	0.8794 (3)	0.0190 (2)	0.0336 (7)
O9	0.9806 (4)	0.9195 (3)	−0.0904 (2)	0.0322 (7)
O10	0.6976 (4)	0.5590 (3)	0.4287 (2)	0.0224 (7)
O11	0.7203 (4)	0.5521 (3)	0.04330 (19)	0.0223 (6)
O12	0.4279 (4)	0.7621 (3)	0.1754 (2)	0.0259 (7)
O13	0.6339 (4)	0.3963 (3)	0.2792 (2)	0.0262 (7)
N1	1.1767 (4)	0.2751 (3)	0.3141 (2)	0.0213 (7)
N2	0.9171 (4)	0.8234 (3)	0.3202 (2)	0.0233 (8)
N3	0.9183 (4)	0.8342 (3)	−0.0015 (2)	0.0212 (7)
O14	0.5912 (4)	0.2215 (3)	0.1372 (2)	0.0239 (7)
O15	0.6255 (4)	0.2025 (3)	0.5159 (2)	0.0258 (7)
H1	0.608 (7)	0.632 (6)	0.437 (4)	0.038 (12)*
H2	0.760 (6)	0.500 (5)	0.490 (4)	0.028 (11)*
H3	0.625 (6)	0.625 (5)	−0.011 (4)	0.025 (10)*
H4	0.789 (7)	0.491 (6)	0.012 (4)	0.037 (12)*
H5	0.341 (8)	0.724 (6)	0.193 (5)	0.056 (15)*
H6	0.369 (7)	0.865 (6)	0.135 (4)	0.046 (12)*
H7	0.649 (7)	0.328 (6)	0.358 (4)	0.043 (11)*
H8	0.640 (6)	0.344 (5)	0.245 (3)	0.019 (10)*
H9	0.705 (7)	0.183 (5)	0.113 (4)	0.035 (11)*
H10	0.561 (7)	0.151 (6)	0.181 (4)	0.048 (14)*
H11	0.741 (9)	0.167 (6)	0.554 (5)	0.053 (15)*
H12	0.589 (9)	0.131 (7)	0.522 (5)	0.069 (18)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Gd1	0.0156 (1)	0.0131 (1)	0.0141 (1)	−0.0049 (1)	0.0013 (1)	−0.0038 (1)
O1	0.0320 (12)	0.0258 (11)	0.0220 (11)	0.0000 (9)	−0.0012 (9)	−0.0096 (9)
O2	0.0307 (12)	0.0234 (11)	0.0190 (10)	0.0007 (9)	0.0036 (9)	−0.0040 (9)
O3	0.0254 (11)	0.0149 (10)	0.0366 (12)	0.0005 (9)	0.0011 (9)	−0.0006 (9)
O4	0.0246 (11)	0.0262 (11)	0.0298 (11)	−0.0082 (9)	0.0003 (9)	−0.0116 (9)
O5	0.0296 (12)	0.0181 (10)	0.0389 (13)	−0.0076 (9)	−0.0101 (10)	−0.0053 (9)
O6	0.0422 (14)	0.0241 (12)	0.0326 (12)	−0.0157 (11)	−0.0049 (10)	−0.0102 (10)
O7	0.0239 (11)	0.0200 (11)	0.0241 (10)	−0.0073 (9)	0.0020 (8)	−0.0024 (8)
O8	0.0213 (11)	0.0266 (12)	0.0495 (14)	−0.0051 (9)	0.0113 (10)	−0.0140 (11)
O9	0.0416 (13)	0.0205 (11)	0.0261 (11)	−0.0107 (10)	0.0118 (10)	0.0002 (9)
O10	0.0258 (12)	0.0208 (12)	0.0175 (11)	−0.0050 (10)	0.0025 (9)	−0.0072 (10)
O11	0.0260 (11)	0.0204 (11)	0.0162 (10)	−0.0040 (9)	−0.0005 (9)	−0.0061 (9)
O12	0.0182 (11)	0.0195 (12)	0.0354 (12)	−0.0055 (9)	−0.0007 (9)	−0.0058 (10)
O13	0.0445 (14)	0.0267 (12)	0.0178 (11)	−0.0233 (11)	0.0068 (9)	−0.0101 (10)
N1	0.0203 (12)	0.0136 (12)	0.0264 (13)	−0.0063 (10)	0.0027 (10)	−0.0029 (10)
N2	0.0305 (14)	0.0202 (13)	0.0182 (12)	−0.0125 (11)	−0.0018 (10)	−0.0023 (10)
N3	0.0247 (13)	0.0162 (12)	0.0222 (12)	−0.0070 (10)	0.0054 (10)	−0.0070 (10)
O14	0.0277 (12)	0.0189 (11)	0.0232 (11)	−0.0083 (10)	0.0008 (9)	−0.0057 (9)
O15	0.0328 (14)	0.0201 (12)	0.0245 (11)	−0.0118 (10)	0.0039 (10)	−0.0061 (9)

Geometric parameters (Å, º)

Gd1—O1	2.528 (2)	O7—N3	1.277 (3)
Gd1—O2	2.494 (3)	O8—N3	1.243 (4)
Gd1—O4	2.578 (2)	O9—N3	1.227 (3)
Gd1—O5	2.518 (3)	O10—H1	0.76 (5)
Gd1—O7	2.552 (2)	O10—H2	0.75 (4)
Gd1—O8	2.754 (2)	O11—H3	0.82 (4)
Gd1—O10	2.398 (2)	O11—H4	0.78 (5)
Gd1—O11	2.389 (2)	O12—H5	0.77 (6)
Gd1—O12	2.392 (3)	O12—H6	0.83 (5)
Gd1—O13	2.364 (3)	O13—H7	0.90 (4)
O1—N1	1.260 (4)	O13—H8	0.71 (4)
O2—N1	1.268 (3)	O14—H9	0.81 (5)
O3—N1	1.228 (4)	O14—H10	0.76 (5)
O4—N2	1.280 (4)	O15—H11	0.80 (6)
O5—N2	1.259 (4)	O15—H12	0.76 (6)
O6—N2	1.223 (4)		
O1—Gd1—O2	50.56 (7)	O8—Gd1—O12	68.70 (8)
O1—Gd1—O4	67.93 (8)	O8—Gd1—O13	130.50 (8)
O1—Gd1—O5	111.77 (8)	O10—Gd1—O11	140.10 (9)
O1—Gd1—O7	99.76 (8)	O10—Gd1—O12	79.72 (8)
O1—Gd1—O8	146.55 (9)	O10—Gd1—O13	71.04 (9)
O1—Gd1—O10	68.65 (9)	O11—Gd1—O12	78.30 (9)
O1—Gd1—O11	115.74 (9)	O11—Gd1—O13	71.47 (8)
O1—Gd1—O12	143.98 (8)	O12—Gd1—O13	75.61 (9)

O1—Gd1—O13	78.10 (9)	Gd1—O1—N1	95.96 (16)
O2—Gd1—O4	93.36 (8)	Gd1—O2—N1	97.41 (18)
O2—Gd1—O5	141.08 (9)	Gd1—O4—N2	95.25 (17)
O2—Gd1—O7	68.28 (8)	Gd1—O5—N2	98.75 (19)
O2—Gd1—O8	109.78 (8)	Gd1—O7—N3	101.83 (17)
O2—Gd1—O10	117.19 (8)	Gd1—O8—N3	92.94 (17)
O2—Gd1—O11	69.75 (8)	Gd1—O10—H2	129 (3)
O2—Gd1—O12	145.36 (9)	H1—O10—H2	110 (5)
O2—Gd1—O13	81.74 (9)	Gd1—O10—H1	118 (3)
O4—Gd1—O5	49.97 (8)	H3—O11—H4	105 (5)
O4—Gd1—O7	69.84 (7)	Gd1—O11—H3	122 (3)
O4—Gd1—O8	89.68 (8)	Gd1—O11—H4	131 (4)
O4—Gd1—O10	75.39 (8)	Gd1—O12—H6	128 (3)
O4—Gd1—O11	144.41 (8)	Gd1—O12—H5	122 (4)
O4—Gd1—O12	120.88 (9)	H5—O12—H6	109 (5)
O4—Gd1—O13	138.89 (7)	Gd1—O13—H8	122 (3)
O5—Gd1—O7	84.58 (8)	Gd1—O13—H7	120 (3)
O5—Gd1—O8	64.27 (7)	H7—O13—H8	104 (5)
O5—Gd1—O10	70.94 (8)	H9—O14—H10	110 (5)
O5—Gd1—O11	130.51 (8)	H11—O15—H12	110 (6)
O5—Gd1—O12	71.40 (9)	O1—N1—O2	116.1 (3)
O5—Gd1—O13	133.03 (9)	O1—N1—O3	122.2 (2)
O7—Gd1—O8	47.73 (8)	O2—N1—O3	121.7 (3)
O7—Gd1—O10	145.14 (8)	O4—N2—O5	116.0 (3)
O7—Gd1—O11	74.76 (8)	O4—N2—O6	122.0 (3)
O7—Gd1—O12	116.18 (8)	O5—N2—O6	122.0 (3)
O7—Gd1—O13	140.84 (8)	O8—N3—O9	122.0 (3)
O8—Gd1—O10	131.16 (8)	O7—N3—O8	117.5 (2)
O8—Gd1—O11	68.65 (8)	O7—N3—O9	120.5 (3)

O2—Gd1—O1—N1	−0.36 (17)	O11—Gd1—O5—N2	−134.37 (16)
O4—Gd1—O1—N1	−114.9 (2)	O12—Gd1—O5—N2	170.31 (18)
O5—Gd1—O1—N1	−139.49 (18)	O13—Gd1—O5—N2	122.60 (17)
O7—Gd1—O1—N1	−51.5 (2)	O1—Gd1—O7—N3	−171.29 (17)
O8—Gd1—O1—N1	−63.6 (3)	O2—Gd1—O7—N3	148.39 (19)
O10—Gd1—O1—N1	162.8 (2)	O4—Gd1—O7—N3	−109.28 (19)
O11—Gd1—O1—N1	26.2 (2)	O5—Gd1—O7—N3	−60.09 (18)
O12—Gd1—O1—N1	132.50 (19)	O8—Gd1—O7—N3	−0.29 (16)
O13—Gd1—O1—N1	88.7 (2)	O10—Gd1—O7—N3	−104.8 (2)
O1—Gd1—O2—N1	0.36 (17)	O11—Gd1—O7—N3	74.51 (18)
O4—Gd1—O2—N1	57.96 (19)	O12—Gd1—O7—N3	6.1 (2)
O5—Gd1—O2—N1	75.6 (2)	O13—Gd1—O7—N3	105.6 (2)
O7—Gd1—O2—N1	124.7 (2)	O1—Gd1—O8—N3	16.5 (3)
O8—Gd1—O2—N1	148.82 (18)	O2—Gd1—O8—N3	−30.59 (19)
O10—Gd1—O2—N1	−17.3 (2)	O4—Gd1—O8—N3	62.87 (18)
O11—Gd1—O2—N1	−154.2 (2)	O5—Gd1—O8—N3	107.53 (19)
O12—Gd1—O2—N1	−130.31 (19)	O7—Gd1—O8—N3	0.29 (16)
O13—Gd1—O2—N1	−80.99 (19)	O10—Gd1—O8—N3	132.99 (18)
O1—Gd1—O4—N2	−148.15 (17)	O11—Gd1—O8—N3	−88.21 (18)

O2—Gd1—O4—N2	167.14 (15)	O12—Gd1—O8—N3	−173.6 (2)
O5—Gd1—O4—N2	1.56 (14)	O13—Gd1—O8—N3	−126.70 (18)
O7—Gd1—O4—N2	101.75 (16)	Gd1—O1—N1—O2	0.6 (3)
O8—Gd1—O4—N2	57.34 (16)	Gd1—O1—N1—O3	179.5 (3)
O10—Gd1—O4—N2	−75.63 (16)	Gd1—O2—N1—O1	−0.6 (3)
O11—Gd1—O4—N2	108.04 (19)	Gd1—O2—N1—O3	−179.5 (3)
O12—Gd1—O4—N2	−7.40 (18)	Gd1—O4—N2—O5	−2.7 (2)
O13—Gd1—O4—N2	−111.58 (18)	Gd1—O4—N2—O6	177.6 (2)
O1—Gd1—O5—N2	28.63 (19)	Gd1—O5—N2—O4	2.7 (2)
O2—Gd1—O5—N2	−24.9 (2)	Gd1—O5—N2—O6	−177.5 (2)
O4—Gd1—O5—N2	−1.59 (14)	Gd1—O7—N3—O8	0.5 (3)
O7—Gd1—O5—N2	−69.73 (17)	Gd1—O7—N3—O9	−179.1 (2)
O8—Gd1—O5—N2	−114.96 (19)	Gd1—O8—N3—O7	−0.5 (3)
O10—Gd1—O5—N2	85.07 (17)	Gd1—O8—N3—O9	179.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O10—H1···O15^{i}	0.76 (5)	1.97 (5)	2.720 (4)	171 (5)
O10—H2···O4^{ii}	0.75 (4)	2.21 (4)	2.956 (3)	178 (6)
O11—H3···O14^{iii}	0.82 (4)	1.85 (4)	2.671 (3)	176 (5)
O11—H4···O7^{iv}	0.78 (5)	2.19 (5)	2.967 (4)	173 (5)
O12—H5···O4^{v}	0.77 (6)	2.50 (6)	3.185 (3)	149 (5)
O12—H5···O7^{v}	0.77 (6)	2.56 (6)	3.156 (4)	135 (5)
O12—H6···O8^{vi}	0.83 (5)	2.27 (5)	3.074 (3)	162 (4)
O12—H6···O9^{vi}	0.83 (5)	2.39 (5)	3.062 (4)	139 (4)
O13—H7···O15	0.90 (4)	1.84 (4)	2.721 (3)	169 (5)
O13—H8···O14	0.71 (4)	2.04 (4)	2.738 (4)	168 (5)
O14—H9···O9^{iv}	0.81 (5)	2.03 (5)	2.826 (4)	167 (4)
O14—H10···O3^{v}	0.76 (5)	2.45 (5)	3.008 (4)	132 (5)
O14—H10···O5^{vii}	0.76 (5)	2.31 (5)	2.888 (4)	134 (5)
O15—H11···O6^{ii}	0.80 (6)	2.02 (6)	2.819 (4)	176 (6)
O15—H12···O3^{viii}	0.76 (6)	2.30 (6)	2.903 (4)	139 (5)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*; (iv) −*x*+2, −*y*+1, −*z*; (v) *x*−1, *y*, *z*; (vi) −*x*+1, −*y*+2, −*z*; (vii) *x*, *y*−1, *z*; (viii) −*x*+2, −*y*, −*z*+1.