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Hexaaquagallium(III) trinitrate trihydrate

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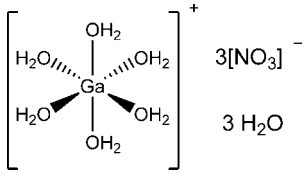
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(O–N) = 0.002 Å; R factor = 0.021; wR factor = 0.058; data-to-parameter ratio = 11.1.

The title compound, $[Ga(H_2O)_6](NO_3)_3\cdot 3H_2O$, is isostructural to other known M^{III} nitrate hydrates (M = Al, Cr, Fe). The structure contains two distinct octahedral $Ga(OH_2)_6$ units (each of $\overline{1}$ symmetry) which are involved in intermolecular hydrogen bonding with the three nitrate anions and three water molecules within the asymmetric unit.

Related literature

For the the aluminium analogue, see: Lazar, Ribár, Divjaković & Mészáros (1991). For the chromium analogue, see: Lazar, Ribár & Prelesnik (1991). For the iron analogue, see: Hair & Beattie (1977). For ionic radii, see: Shannon & Prewitt (1969). Gallium nitrate, used in the preparation, easily forms supersaturated solutions, see: Rudolph *et al.* (2002), and hence the sample was cooled to 248 K and a seed crystal was introduced to initiate crystallization.



Experimental

Crystal data

$[Ga(H_2O)_6](NO_3)_3\cdot 3H_2O$
M_r = 417.89
Monoclinic, $P2_1/c$
a = 13.9609 (6) Å
b = 9.6498 (5) Å
c = 10.9743 (5) Å
β = 95.448 (1)°
V = 1471.78 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.97 mm^{-1}
T = 296 K
0.40 × 0.34 × 0.29 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.479, T_{max} = 0.564
10587 measured reflections
3037 independent reflections
2509 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.058
S = 1.05
3037 reflections
274 parameters
18 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O18—H18···O8	0.801 (16)	2.26 (2)	2.9348 (18)	142 (2)
O16—H14···O18	0.825 (15)	2.072 (15)	2.8732 (19)	163.8 (19)
O5—H10···O7	0.823 (16)	1.908 (17)	2.7052 (17)	163 (2)
O1—H1···O16	0.814 (15)	1.846 (16)	2.6474 (16)	168 (2)
O4—H7···O14	0.809 (15)	1.833 (15)	2.6399 (15)	175 (2)
O5—H9···O17	0.810 (16)	1.869 (16)	2.676 (2)	174 (2)
O18—H17···O14	0.816 (16)	2.082 (17)	2.8729 (18)	163 (2)
O3—H6···O15^{i}	0.814 (15)	1.903 (16)	2.7150 (16)	175 (2)
O1—H2···O10^{i}	0.808 (15)	1.848 (16)	2.6545 (16)	175 (2)
O2—H4···O16^{i}	0.790 (16)	1.901 (16)	2.6895 (18)	175 (2)
O4—H8···O17^{ii}	0.821 (15)	1.816 (15)	2.6312 (16)	171 (2)
O17—H15···O9^{ii}	0.808 (15)	1.977 (16)	2.7791 (19)	171 (2)
O3—H5···O13^{iii}	0.792 (15)	1.961 (16)	2.7454 (16)	171 (2)
O6—H12···O12^{iv}	0.796 (15)	1.926 (16)	2.7179 (16)	174 (2)
O16—H13···O18^{v}	0.820 (16)	1.934 (16)	2.7525 (19)	177 (3)
O6—H11···O11^{vi}	0.800 (15)	1.895 (16)	2.6938 (17)	176 (2)
O2—H3···O8^{vii}	0.794 (15)	1.943 (16)	2.7269 (17)	169 (2)
O17—H16···O7^{viii}	0.802 (16)	2.026 (18)	2.7675 (18)	154 (2)

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MG2076).

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supporting information

Acta Cryst. (2009). E**65**, i65 [doi:10.1107/S1600536809028086]

Hexaaquagallium(III) trinitrate trihydrate

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S1. Comment

The title compound is isostructural with $[Al(H_2O)_6](NO_3)_3.3H_2O$ (Lazar, Ribár, Divjaković & Mészáros, 1991), $[Cr(H_2O)_6](NO_3)_3.3H_2O$ (Lazar, Ribár & Prelesnik, 1991) and $[Fe(H_2O)_6](NO_3)_3.3H_2O$ (Hair & Beattie, 1977). Its unit cell volume is almost identical to that of the chromium derivative (1473.87 (17) $Å^3$) and intermediate between those of the aluminum (1448.9 (4) $Å^3$) and iron derivatives 1489.8 (2) $Å^3$), consistent with the values of ionic radii (Ga^{3+}, 0.760 Å; Cr^{3+}, 0.755 Å; Al^{3+}, 0.670 Å; Fe^{3+}, 0.785 Å) (Shannon & Prewitt, 1969). On each of the octahedral units there are two symmetry-related water molecules which hydrogen bond to two NO_3^- anions. The remaining metal-bound water molecules participate in intermolecular hydrogen bonding with one NO_3^- anion and one of the interstitial H_2O molecules.

S2. Experimental

The title compound was prepared by dissolving 5 grams of gallium(III) nitrate hydrate (Aldrich Chemical Company) in a minimum of H_2O (approximately 7 ml) and adding three drops of concentrated nitric acid to suppress hydrolysis. Because gallium nitrate easily forms supersaturated solutions (Rudolph *et al.*, 2002), the sample was cooled to 248 K and a seed crystal was introduced to initiate crystallization. A suitable crystal was sealed in a glass capillary to prevent water loss from this hygroscopic material.

S3. Refinement

The H atoms were found in the electron difference map and O-H distances fixed to 0.82 Å.

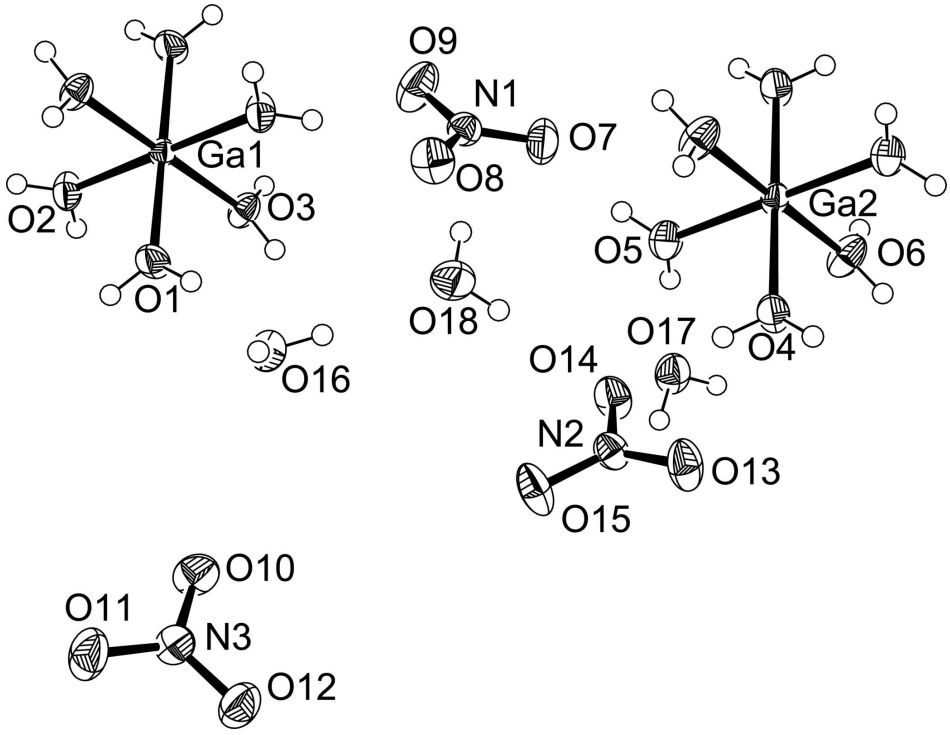

Figure 1

$[Ga(H_2O)_6](NO_3).3H_2O$ with thermal ellipsoids shown at 50% probability level.

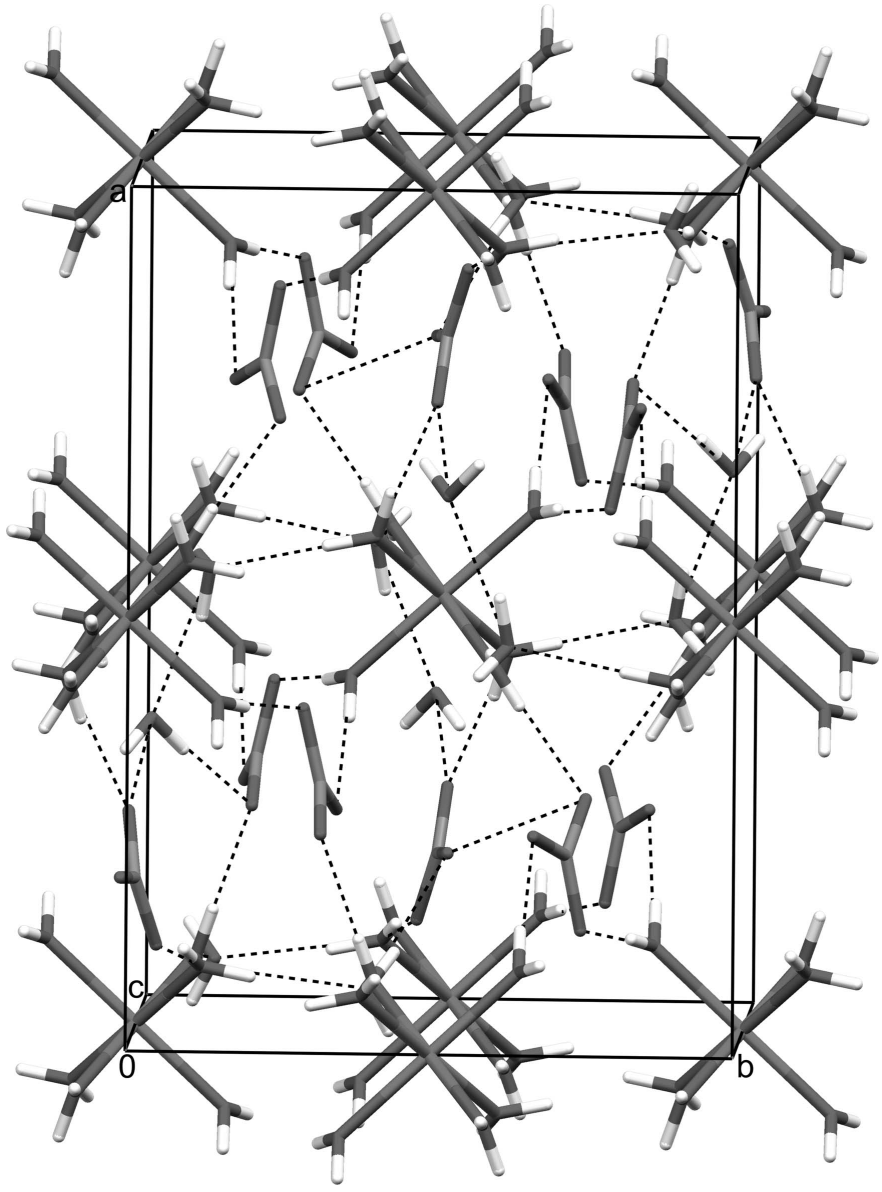

Figure 2

Packing diagram viewed down the c-axis with hydrogen bonds indicated by dashed lines.

Hexaaquagallium(III) trinitrate trihydrate

Crystal data

$[Ga(H_2O)_6](NO_3)_3{\cdot}3H_2O$
$M_r = 417.89$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.9609\ (6)$ Å
$b = 9.6498\ (5)$ Å
$c = 10.9743\ (5)$ Å
$\beta = 95.448\ (1)°$
$V = 1471.78\ (12)$ Å^3
$Z = 4$

$F(000) = 856$
$D_x = 1.886$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5403 reflections
$\theta = 2.3–28.3°$
$\mu = 1.97$ mm^{-1}
$T = 296$ K
Irregular, colourless
0.40 × 0.34 × 0.29 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.479, T_{max} = 0.564
10587 measured reflections
3037 independent reflections
2509 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 26.5°, θ_{min} = 2.6°
h = −17→17
k = −12→10
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.058
S = 1.05
3037 reflections
274 parameters
18 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2 + 0.4119P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *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}
Ga1	0.5000	0.0000	0.0000	0.01867 (8)
Ga2	0.0000	0.0000	0.5000	0.02131 (8)
N1	0.19260 (10)	0.00919 (13)	0.12573 (13)	0.0285 (3)
N2	0.29851 (9)	0.18907 (14)	0.70878 (11)	0.0268 (3)
N3	0.78610 (9)	0.28563 (14)	0.71869 (11)	0.0293 (3)
O1	0.57018 (8)	0.08802 (13)	0.13967 (10)	0.0306 (3)
H2	0.6187 (12)	0.132 (2)	0.1335 (19)	0.051 (6)*
H1	0.5546 (14)	0.088 (2)	0.2093 (15)	0.051 (6)*
O2	0.57327 (8)	0.10539 (13)	−0.11126 (10)	0.0294 (2)
H4	0.5629 (16)	0.1854 (17)	−0.121 (2)	0.053 (7)*
H3	0.6243 (12)	0.082 (2)	−0.1307 (18)	0.054 (6)*
O3	0.40601 (8)	0.14903 (12)	−0.00574 (10)	0.0269 (2)
H6	0.3964 (15)	0.190 (2)	0.0569 (16)	0.051 (6)*
H5	0.3607 (13)	0.150 (2)	−0.0550 (17)	0.051 (6)*
O4	0.07132 (8)	0.09427 (14)	0.63446 (10)	0.0327 (3)

H8	0.0636 (14)	0.093 (2)	0.7077 (14)	0.043 (5)*
H7	0.1244 (12)	0.121 (2)	0.6219 (19)	0.051 (6)*
O5	0.07408 (9)	0.10908 (14)	0.39031 (10)	0.0339 (3)
H10	0.0971 (15)	0.084 (2)	0.3275 (17)	0.065 (7)*
H9	0.0633 (16)	0.1911 (17)	0.381 (2)	0.054 (7)*
O6	−0.09607 (8)	0.14720 (14)	0.49563 (11)	0.0358 (3)
H12	−0.1406 (13)	0.147 (2)	0.4446 (18)	0.054 (7)*
H11	−0.1078 (16)	0.183 (2)	0.5582 (17)	0.055 (7)*
O7	0.11323 (8)	0.04401 (15)	0.16045 (11)	0.0439 (3)
O8	0.26475 (9)	−0.00369 (13)	0.19969 (12)	0.0430 (3)
O9	0.19968 (11)	−0.01084 (15)	0.01616 (12)	0.0529 (4)
O10	0.72635 (8)	0.25702 (13)	0.62972 (10)	0.0369 (3)
O11	0.87278 (8)	0.26164 (15)	0.71226 (11)	0.0473 (3)
O12	0.75836 (8)	0.33667 (15)	0.81328 (10)	0.0424 (3)
O13	0.26276 (8)	0.16544 (14)	0.80530 (10)	0.0395 (3)
O14	0.24653 (7)	0.18488 (13)	0.60783 (9)	0.0339 (3)
O15	0.38523 (7)	0.21721 (14)	0.70780 (10)	0.0400 (3)
O16	0.53516 (9)	0.12179 (13)	0.37069 (11)	0.0333 (3)
H14	0.4793 (11)	0.108 (2)	0.3864 (17)	0.043 (6)*
H13	0.5662 (15)	0.076 (2)	0.4232 (18)	0.063 (7)*
O17	0.05170 (9)	0.38374 (14)	0.36978 (11)	0.0362 (3)
H15	0.0906 (14)	0.427 (2)	0.4140 (19)	0.057 (7)*
H16	0.0021 (13)	0.420 (2)	0.383 (2)	0.056 (7)*
O18	0.35675 (10)	0.02473 (15)	0.45021 (13)	0.0410 (3)
H17	0.3188 (14)	0.075 (2)	0.482 (2)	0.064 (8)*
H18	0.3232 (17)	−0.016 (2)	0.399 (2)	0.062 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ga1	0.01679 (12)	0.02124 (13)	0.01796 (12)	0.00073 (8)	0.00156 (8)	−0.00138 (8)
Ga2	0.01777 (12)	0.02999 (15)	0.01606 (12)	−0.00007 (8)	0.00097 (8)	−0.00210 (8)
N1	0.0297 (7)	0.0278 (8)	0.0280 (7)	−0.0019 (5)	0.0029 (6)	0.0003 (5)
N2	0.0238 (6)	0.0302 (7)	0.0261 (6)	−0.0027 (5)	0.0002 (5)	0.0037 (5)
N3	0.0290 (7)	0.0308 (7)	0.0275 (7)	0.0044 (6)	−0.0005 (5)	−0.0004 (5)
O1	0.0271 (6)	0.0417 (7)	0.0227 (6)	−0.0094 (5)	0.0012 (4)	−0.0071 (5)
O2	0.0258 (6)	0.0284 (7)	0.0356 (6)	0.0032 (5)	0.0116 (5)	0.0065 (5)
O3	0.0245 (5)	0.0316 (6)	0.0240 (6)	0.0097 (5)	−0.0002 (4)	−0.0029 (5)
O4	0.0246 (6)	0.0536 (8)	0.0196 (6)	−0.0108 (5)	0.0018 (4)	−0.0078 (5)
O5	0.0395 (6)	0.0376 (8)	0.0265 (6)	−0.0047 (6)	0.0119 (5)	0.0013 (5)
O6	0.0314 (6)	0.0480 (8)	0.0269 (6)	0.0158 (5)	−0.0024 (5)	−0.0064 (5)
O7	0.0317 (6)	0.0576 (8)	0.0446 (7)	0.0009 (6)	0.0141 (5)	−0.0047 (6)
O8	0.0379 (7)	0.0536 (9)	0.0361 (7)	0.0013 (5)	−0.0045 (5)	0.0039 (5)
O9	0.0524 (9)	0.0741 (11)	0.0329 (7)	0.0144 (7)	0.0072 (6)	−0.0110 (6)
O10	0.0353 (6)	0.0431 (7)	0.0301 (6)	0.0072 (5)	−0.0080 (5)	−0.0057 (5)
O11	0.0271 (6)	0.0721 (10)	0.0422 (7)	0.0116 (6)	0.0006 (5)	−0.0133 (6)
O12	0.0339 (6)	0.0605 (9)	0.0324 (6)	0.0064 (6)	0.0018 (5)	−0.0149 (6)
O13	0.0301 (6)	0.0633 (9)	0.0255 (6)	−0.0049 (6)	0.0053 (5)	0.0088 (6)

O14	0.0262 (5)	0.0503 (7)	0.0242 (5)	−0.0054 (5)	−0.0023 (4)	0.0028 (5)
O15	0.0214 (5)	0.0629 (9)	0.0350 (6)	−0.0103 (5)	−0.0009 (4)	0.0128 (6)
O16	0.0336 (6)	0.0346 (7)	0.0323 (6)	0.0031 (5)	0.0066 (5)	0.0016 (5)
O17	0.0311 (6)	0.0496 (8)	0.0279 (6)	−0.0003 (6)	0.0024 (5)	−0.0019 (5)
O18	0.0383 (7)	0.0469 (8)	0.0365 (7)	−0.0027 (6)	−0.0027 (6)	−0.0091 (6)

Geometric parameters (Å, º)

Ga1—O1	1.9354 (10)	Ga2—O5	1.9654 (11)
Ga1—O1^{i}	1.9354 (10)	N1—O9	1.2311 (19)
Ga1—O3	1.9438 (10)	N1—O8	1.2385 (18)
Ga1—O3^{i}	1.9438 (10)	N1—O7	1.2513 (18)
Ga1—O2	1.9515 (11)	N2—O13	1.2343 (16)
Ga1—O2^{i}	1.9515 (11)	N2—O15	1.2418 (16)
Ga2—O4^{ii}	1.9280 (10)	N2—O14	1.2660 (16)
Ga2—O4	1.9280 (10)	N3—O11	1.2407 (17)
Ga2—O6^{ii}	1.9510 (12)	N3—O12	1.2435 (17)
Ga2—O6	1.9510 (12)	N3—O10	1.2533 (16)
Ga2—O5^{ii}	1.9654 (11)		
O1—Ga1—O1^{i}	180.0	O6^{ii}—Ga2—O6	180.00 (8)
O1—Ga1—O3	89.45 (5)	O4^{ii}—Ga2—O5^{ii}	87.28 (5)
O1^{i}—Ga1—O3	90.55 (5)	O4—Ga2—O5^{ii}	92.72 (5)
O1—Ga1—O3^{i}	90.55 (5)	O6^{ii}—Ga2—O5^{ii}	89.74 (6)
O1^{i}—Ga1—O3^{i}	89.45 (5)	O6—Ga2—O5^{ii}	90.26 (6)
O3—Ga1—O3^{i}	180.0	O4^{ii}—Ga2—O5	92.72 (5)
O1—Ga1—O2	90.62 (5)	O4—Ga2—O5	87.28 (5)
O1^{i}—Ga1—O2	89.38 (5)	O6^{ii}—Ga2—O5	90.26 (6)
O3—Ga1—O2	89.23 (5)	O6—Ga2—O5	89.74 (6)
O3^{i}—Ga1—O2	90.77 (5)	O5^{ii}—Ga2—O5	180.0
O1—Ga1—O2^{i}	89.38 (5)	O9—N1—O8	119.25 (15)
O1^{i}—Ga1—O2^{i}	90.62 (5)	O9—N1—O7	119.68 (14)
O3—Ga1—O2^{i}	90.77 (5)	O8—N1—O7	121.06 (14)
O3^{i}—Ga1—O2^{i}	89.23 (5)	O13—N2—O15	121.45 (12)
O2—Ga1—O2^{i}	179.999 (2)	O13—N2—O14	120.01 (12)
O4^{ii}—Ga2—O4	180.0	O15—N2—O14	118.54 (12)
O4^{ii}—Ga2—O6^{ii}	88.81 (5)	O11—N3—O12	120.28 (13)
O4—Ga2—O6^{ii}	91.19 (5)	O11—N3—O10	119.68 (13)
O4^{ii}—Ga2—O6	91.19 (5)	O12—N3—O10	120.04 (12)
O4—Ga2—O6	88.81 (5)		

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) −*x*, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O18—H18···O8	0.80 (2)	2.26 (2)	2.9348 (18)	142 (2)
O16—H14···O18	0.83 (2)	2.07 (2)	2.8732 (19)	164 (2)
O5—H10···O7	0.82 (2)	1.91 (2)	2.7052 (17)	163 (2)

O1—H1···O16	0.81 (2)	1.85 (2)	2.6474 (16)	168 (2)
O4—H7···O14	0.81 (2)	1.83 (2)	2.6399 (15)	175 (2)
O5—H9···O17	0.81 (2)	1.87 (2)	2.676 (2)	174 (2)
O18—H17···O14	0.82 (2)	2.08 (2)	2.8729 (18)	163 (2)
O3—H6···O15^{iii}	0.81 (2)	1.90 (2)	2.7150 (16)	175 (2)
O1—H2···O10^{iii}	0.81 (2)	1.85 (2)	2.6545 (16)	175 (2)
O2—H4···O16^{iii}	0.79 (2)	1.90 (2)	2.6895 (18)	175 (2)
O4—H8···O17^{iv}	0.82 (2)	1.82 (2)	2.6312 (16)	171 (2)
O17—H15···O9^{iv}	0.81 (2)	1.98 (2)	2.7791 (19)	171 (2)
O3—H5···O13^{v}	0.79 (2)	1.96 (2)	2.7454 (16)	171 (2)
O6—H12···O12^{vi}	0.80 (2)	1.93 (2)	2.7179 (16)	174 (2)
O16—H13···O18^{vii}	0.82 (2)	1.93 (2)	2.7525 (19)	177 (3)
O6—H11···O11^{viii}	0.80 (2)	1.90 (2)	2.6938 (17)	176 (2)
O2—H3···O8^{i}	0.79 (2)	1.94 (2)	2.7269 (17)	169 (2)
O17—H16···O7^{ix}	0.80 (2)	2.03 (2)	2.7675 (18)	154 (2)

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