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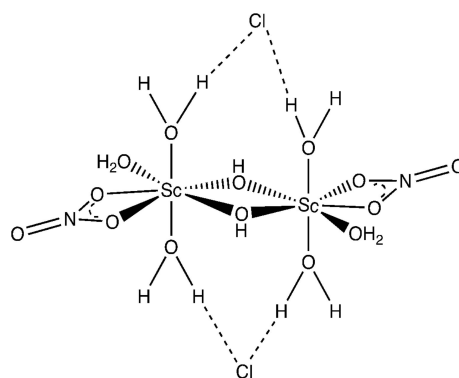
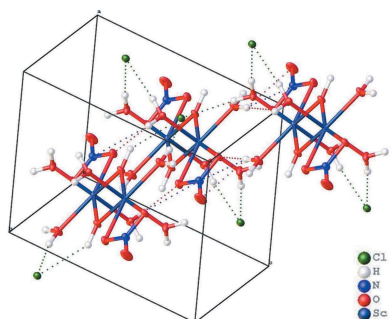
Crystal structure of hydroxy scandium nitrate chloride

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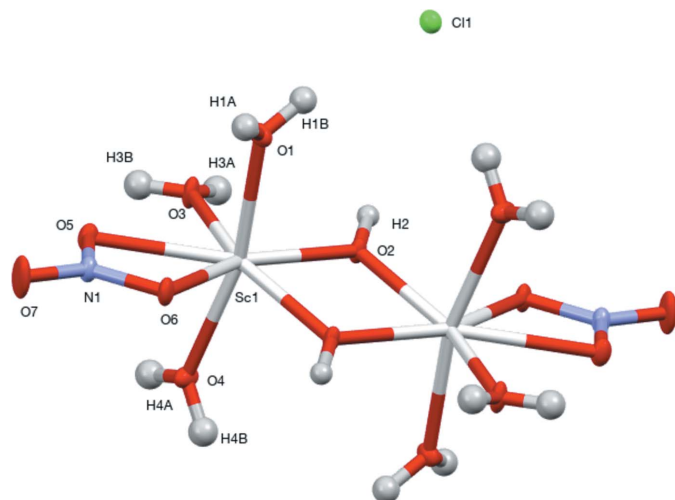
Each Sc³⁺ ion in the title salt, di- μ -hydroxido-bis[triaqua(nitrato- κ^2O,O')scandium(III)] dichloride, [Sc₂(NO₃)₂(OH)₂(H₂O)₆]Cl₂, is coordinated by a nitrate anion, two hydroxide ions and three water molecules to generate a distorted pentagonal-bipyramidal ScO₇ coordination polyhedron. The complete $\{[(\text{NO}_3)(\mu\text{-OH})\text{Sc}(\text{H}_2\text{O})_3]_2\}^{2+}$ ion is generated by crystallographic inversion symmetry. The nitrate anion binds in a bidentate fashion whereas the hydroxide ions are bridged between two Sc centers. Two charge-balancing Cl⁻ ions are located in the outer sphere. In the extended structure, O—H···O and O—H···Cl hydrogen bonds connect the components into a three-dimensional network.

1. Chemical context

Scandium nitrate compounds have found widespread utility in a diverse number of applications, including catalysts for aqueous-based organic reactions (Kobayashi, 1999), heterogeneous Lewis acid catalysts (Cao *et al.*, 2015), cyanosilylation catalysis (Zhang *et al.*, 2015) and films for use in optics and electronic manufacturing (Wang *et al.*, 2013). Previously, the structural properties of scandium salts were reviewed and the wide variety of structure types available for Sc metal were presented (Sears *et al.*, 2017). From this review, the diversity of structurally characterized scandium nitrate salts was illuminated. These were found to possess inner-sphere, outer-sphere and mixed-sphere nitrate ions. Additionally, a number of bridging ligands (OH⁻, OMe⁻) were present. As we continue to explore the fundamental coordination behavior of scandium with nitric acid as a means to recycle this multipurpose metal, another unusual scandium nitrate structure $[(\kappa^2\text{-NO}_3)(\mu\text{-OH})\text{Sc}(\text{H}_2\text{O})_3]_2(\text{Cl})$ (**1**) was isolated. This report details the structure and its relationship to known scandium nitrate derivatives.



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Figure 1

The molecular structure of the title compound, with non-H atoms shown as displacement ellipsoids at the 50% probability level. Only one Cl[−] ion is shown. Unlabeled atoms are generated by the symmetry operation $1 - x, -y, 1 - z$.

2. Structural commentary

The title compound (Fig. 1) is the third reported hydrated scandium nitrate salt. We previously isolated $[(\text{H}_2\text{O})_4\text{Sc}(k^2\text{-NO}_3)_2](\text{NO}_3)(\text{H}_2\text{O})$ and $[(\text{H}_2\text{O})_3\text{Sc}(k^2\text{-NO}_3)(\mu\text{-OH})]_2(\text{NO}_3)$ from the reaction of $[(\text{H}_2\text{O})_5\text{Sc}(\mu\text{-OH})]_2(\text{Cl})_2(\text{H}_2\text{O})$ with concentrated nitric acid at elevated and room temperatures, respectively. Similarities between $[(k^2\text{-NO}_3)(\mu\text{-OH})\text{Sc}(\text{H}_2\text{O})_3]_2(\text{Cl})$ and $[(\text{H}_2\text{O})_3\text{Sc}(k^2\text{-NO}_3)(\mu\text{-OH})]_2(\text{NO}_3)$ were expected and observed.

The axial water molecules are distorted from linearity more so for **1** [$\text{O1}-\text{Sc1}-\text{O4} = 166.48(2)^\circ$] than for $[(\text{H}_2\text{O})_3\text{Sc}(k^2\text{-NO}_3)(\mu\text{-OH})]_2(\text{NO}_3)$ [$\text{O4}-\text{Sc}-\text{O6} = 171.52(7)^\circ$]. The Sc—O (H₂O) bond distances of 2.124(1)–2.148(1) Å for **1** are comparable to the 2.114(2)–2.183(1) Å distances reported for the other hydroxide-bridged NO₃ salt. Bond angles between the axial water molecules and the remaining nearly coplanar equatorial ligands range from 79.56(2)–100.60(2)° for **1** and 82.50(6)–99.96(6)° for $[(\text{H}_2\text{O})_3\text{Sc}(k^2\text{-NO}_3)(\mu\text{-OH})]_2(\text{NO}_3)$. The angles between the equatorial oxygen atoms range from 55.61(2)–81.36(2)° and 55.76(5)–83.70(6)°, respectively. The shortest Sc—O bond distances, 2.0542(5)–2.0569(5) Å for **1** and 2.053(2)–2.076(1) Å for $[(\text{H}_2\text{O})_3\text{Sc}(k^2\text{-NO}_3)(\mu\text{-OH})]_2(\text{NO}_3)$, occur for the bridging hydroxide ions. In both salts, the bidentate NO₃ ions have the weakest interaction with Sc—O bond distances of 2.291(1)–2.314(1) Å for **1** and 2.114(2)–2.183(1) Å for $[(\text{H}_2\text{O})_3\text{Sc}(k^2\text{-NO}_3)(\mu\text{-OH})]_2(\text{NO}_3)$.

The precursor to **1**, $[(\text{H}_2\text{O})_5\text{Sc}(\mu\text{-OH})]_2(\text{Cl})_2(\text{H}_2\text{O})$, is also a seven-coordinate Sc salt. Rotation of the precursor reveals a capped trigonal-prismatic geometry about the Sc centers that is useful for comparison. Equatorial ligand angles for $[(\text{H}_2\text{O})_5\text{Sc}(\mu\text{-OH})]_2(\text{Cl})_2(\text{H}_2\text{O})$ had a much smaller range of 83.32–95.17°. Dihedral angles between axial and equatorial ligands for the precursor also have a significantly reduced

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1A}\cdots\text{O6}^{\text{i}}$	0.782 (16)	2.051 (16)	2.8175 (7)	166.6 (16)
$\text{O1}-\text{H1B}\cdots\text{Cl1}^{\text{ii}}$	0.787 (15)	2.300 (15)	3.0856 (6)	176.6 (14)
$\text{O2}-\text{H2}\cdots\text{Cl1}^{\text{iii}}$	0.692 (16)	2.617 (16)	3.2749 (5)	159.7 (17)
$\text{O3}-\text{H3A}\cdots\text{Cl1}^{\text{iii}}$	0.815 (16)	2.305 (16)	3.1017 (6)	165.9 (15)
$\text{O3}-\text{H3B}\cdots\text{O5}^{\text{iv}}$	0.811 (15)	1.989 (15)	2.7977 (7)	175.0 (14)
$\text{O4}-\text{H4A}\cdots\text{Cl1}^{\text{v}}$	0.859 (15)	2.316 (15)	3.1722 (6)	174.5 (13)
$\text{O4}-\text{H4B}\cdots\text{Cl1}$	0.824 (16)	2.242 (16)	3.0658 (6)	179.0 (15)

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

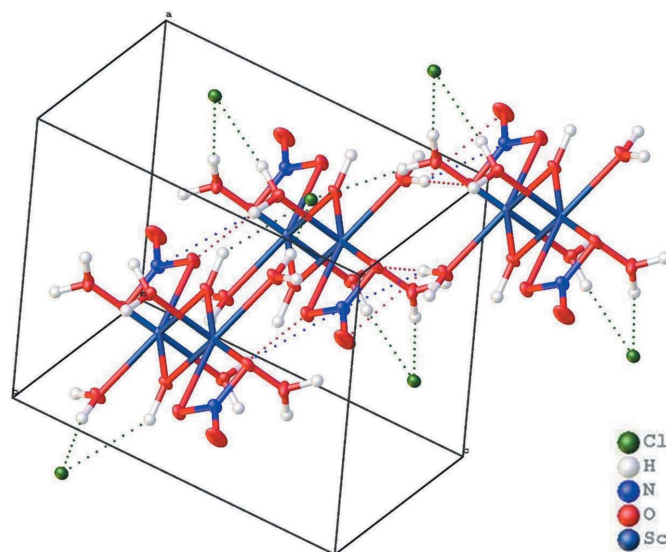
range of 77.18–79.09°. These differences further support the distorted pentagonal-bipyramidal geometry assigned to **1**.

3. Supramolecular features

A network of scandium hydroxy nitrate dimer chains that interact *via* separate equatorial coordinated water molecules and nitrate ions with one another is observed for $[(k^2\text{-NO}_3)(\mu\text{-OH})\text{Sc}(\text{H}_2\text{O})_3]_2(\text{Cl})$. These chains are further linked into a three-dimensional network (Fig. 2) by O—H \cdots Cl hydrogen bonds between axially as well as equatorially coordinated water molecules and outer sphere Cl[−] anions indicated by the symmetry operations in Table 1.

4. Database survey

There are two reports of hydrated scandium nitrates, $[(\text{H}_2\text{O})_4\text{Sc}(k^2\text{-NO}_3)_2](\text{NO}_3)(\text{H}_2\text{O})$ (Boyle *et al.*, 2015) and $[(\text{H}_2\text{O})_3\text{Sc}(k^2\text{-NO}_3)(\mu\text{-OH})]_2(\text{NO}_3)$ (Wang *et al.*, 2013; Boyle *et al.* 2015), and both contain outer-sphere nitrate anions. As expected a similar network is observed for $[(\text{H}_2\text{O})_3\text{Sc}(k^2\text{-NO}_3)(\mu\text{-OH})]_2(\text{NO}_3)$. Salt **1** is the first reported hydrated scandium nitrate to contain outer-sphere chloride anions.


Figure 2

Partial packing diagram of the title compound, showing hydrogen bonds as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	[Sc ₂ (NO ₃) ₂ (OH) ₂ (H ₂ O) ₆]2(Cl)
<i>M</i> _r	426.95
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.7221 (3), 7.6279 (4), 8.5181 (4)
α , β , γ (°)	100.904 (2), 110.125 (2), 102.329 (2)
<i>V</i> (Å ³)	383.87 (3)
<i>Z</i>	1
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	1.30
Crystal size (mm)	0.52 × 0.24 × 0.21
Data collection	
Diffraction	Broker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2016)
<i>T</i> _{min} , <i>T</i> _{max}	0.634, 0.749
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	21806, 5393, 4597
<i>R</i> _{int}	0.025
($\sin \theta/\lambda$) _{max} (Å ⁻¹)	0.944
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.024, 0.056, 1.04
No. of reflections	5393
No. of parameters	119
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.50, -0.35

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

5. Synthesis and crystallization

Salt **1** was isolated from a cooled (273 K) mixture of [(H₂O)₅Sc(μ -OH)]₂4(Cl)2(H₂O) dissolved in water and an equal volume of concentrated HNO₃(aq). The reaction was slowly warmed to room temperature and set aside for slow evaporation until crystals formed. From this mixture, a single

crystal of **1** was selected and used for single crystal X-ray analysis. Note: Both [(H₂O)₄Sc(κ^2 -NO₃)₂]NO₃(H₂O) and [(H₂O)₃Sc(κ^2 -NO₃)(μ -OH)]₂2(NO₃) have also been isolated from this preparatory route (Boyle *et al.*, 2015).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Di- μ -hydroxido-bis[triaqua(nitrato- κ^2 O, O')scandium(III)] dichloride

Crystal data

[Sc₂(NO₃)₂(OH)₂(H₂O)₆]₂(Cl)

$M_r = 426.95$

Triclinic, $P\bar{1}$

$a = 6.7221$ (3) Å

$b = 7.6279$ (4) Å

$c = 8.5181$ (4) Å

$\alpha = 100.904$ (2)°

$\beta = 110.125$ (2)°

$\gamma = 102.329$ (2)°

$V = 383.87$ (3) Å³

$Z = 1$

$F(000) = 216$

$D_x = 1.847$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9960 reflections

$\theta = 2.7$ – 43.4 °

$\mu = 1.30$ mm⁻¹

$T = 100$ K

Plate, colourless

$0.52 \times 0.24 \times 0.21$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus tube

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2016)

$T_{\min} = 0.634$, $T_{\max} = 0.749$

21806 measured reflections

5393 independent reflections

4597 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 42.1$ °, $\theta_{\min} = 2.7$ °

$h = -12 \rightarrow 12$

$k = -14 \rightarrow 14$

$l = -16 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.024$

$wR(F^2) = 0.056$

$S = 1.04$

5393 reflections

119 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.026P)^2 + 0.0527P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.50$ e Å⁻³

$\Delta\rho_{\min} = -0.35$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sc1	0.64421 (2)	0.20102 (2)	0.65362 (2)	0.00619 (2)
O1	0.77577 (9)	0.34667 (7)	0.50248 (7)	0.01145 (8)
H1A	0.730 (3)	0.425 (2)	0.469 (2)	0.037 (4)*
H1B	0.814 (2)	0.297 (2)	0.4330 (19)	0.031 (4)*
O2	0.66804 (8)	−0.04815 (7)	0.53126 (6)	0.00944 (8)
H2	0.755 (3)	−0.084 (2)	0.562 (2)	0.042 (4)*
O3	0.97436 (9)	0.22378 (8)	0.82658 (7)	0.01256 (9)
H3A	1.025 (3)	0.136 (2)	0.832 (2)	0.036 (4)*
H3B	1.042 (2)	0.306 (2)	0.919 (2)	0.030 (4)*
O4	0.54719 (9)	0.11936 (8)	0.84788 (7)	0.01370 (9)
H4A	0.639 (2)	0.131 (2)	0.951 (2)	0.032 (4)*
H4B	0.425 (3)	0.048 (2)	0.829 (2)	0.037 (4)*
O5	0.77362 (8)	0.48678 (7)	0.86375 (7)	0.01144 (8)
O6	0.45946 (9)	0.42130 (7)	0.64820 (6)	0.01136 (8)
O7	0.57811 (11)	0.68156 (9)	0.85879 (8)	0.02282 (13)
N1	0.60275 (10)	0.53719 (8)	0.79388 (8)	0.01116 (9)
Cl1	0.09630 (3)	−0.14716 (2)	0.78189 (2)	0.01058 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sc1	0.00664 (4)	0.00485 (4)	0.00490 (4)	0.00172 (3)	0.00048 (3)	0.00005 (3)
O1	0.0153 (2)	0.0105 (2)	0.0118 (2)	0.00636 (17)	0.00710 (17)	0.00454 (16)
O2	0.00739 (17)	0.00762 (18)	0.00909 (18)	0.00341 (14)	−0.00061 (14)	−0.00082 (14)
O3	0.01049 (19)	0.00957 (19)	0.0107 (2)	0.00455 (16)	−0.00204 (16)	−0.00219 (16)
O4	0.0119 (2)	0.0173 (2)	0.00861 (19)	0.00022 (17)	0.00226 (16)	0.00464 (17)
O5	0.00943 (18)	0.00930 (19)	0.01084 (19)	0.00451 (15)	−0.00074 (15)	−0.00048 (15)
O6	0.01149 (19)	0.00946 (19)	0.00762 (18)	0.00380 (15)	−0.00085 (15)	−0.00164 (15)
O7	0.0229 (3)	0.0165 (3)	0.0196 (3)	0.0137 (2)	−0.0006 (2)	−0.0075 (2)
N1	0.0113 (2)	0.0094 (2)	0.0092 (2)	0.00476 (17)	0.00089 (17)	−0.00115 (17)
Cl1	0.01037 (6)	0.01118 (6)	0.01048 (6)	0.00477 (5)	0.00374 (5)	0.00283 (5)

Geometric parameters (\AA , $^\circ$)

Sc1—O2	2.0542 (5)	O2—Sc1 ⁱ	2.0569 (5)
Sc1—O2 ⁱ	2.0569 (5)	O2—H2	0.692 (16)
Sc1—O4	2.1238 (6)	O3—H3A	0.815 (16)
Sc1—O1	2.1399 (5)	O3—H3B	0.811 (15)
Sc1—O3	2.1482 (5)	O4—H4A	0.859 (15)

Sc1—O6	2.2910 (5)	O4—H4B	0.824 (16)
Sc1—O5	2.3140 (5)	O5—N1	1.2752 (8)
Sc1—Sc1 ⁱ	3.3085 (3)	O6—N1	1.2837 (8)
O1—H1A	0.782 (16)	O7—N1	1.2068 (8)
O1—H1B	0.787 (15)		
O2—Sc1—O2 ⁱ	72.83 (2)	O4—Sc1—Sc1 ⁱ	95.494 (17)
O2—Sc1—O4	99.79 (2)	O1—Sc1—Sc1 ⁱ	97.895 (16)
O2 ⁱ —Sc1—O4	89.10 (2)	O3—Sc1—Sc1 ⁱ	117.158 (16)
O2—Sc1—O1	92.12 (2)	O6—Sc1—Sc1 ⁱ	114.024 (14)
O2 ⁱ —Sc1—O1	100.60 (2)	O5—Sc1—Sc1 ⁱ	168.016 (14)
O4—Sc1—O1	166.48 (2)	Sc1—O1—H1A	121.1 (11)
O2—Sc1—O3	81.36 (2)	Sc1—O1—H1B	122.4 (10)
O2 ⁱ —Sc1—O3	152.16 (2)	H1A—O1—H1B	107.7 (15)
O4—Sc1—O3	85.12 (2)	Sc1—O2—Sc1 ⁱ	107.17 (2)
O1—Sc1—O3	90.41 (2)	Sc1—O2—H2	125.6 (14)
O2—Sc1—O6	148.997 (19)	Sc1 ⁱ —O2—H2	125.0 (14)
O2 ⁱ —Sc1—O6	78.391 (19)	Sc1—O3—H3A	124.5 (11)
O4—Sc1—O6	90.93 (2)	Sc1—O3—H3B	122.1 (10)
O1—Sc1—O6	81.93 (2)	H3A—O3—H3B	108.8 (14)
O3—Sc1—O6	128.81 (2)	Sc1—O4—H4A	124.1 (10)
O2—Sc1—O5	154.887 (19)	Sc1—O4—H4B	125.0 (11)
O2 ⁱ —Sc1—O5	131.997 (19)	H4A—O4—H4B	108.9 (14)
O4—Sc1—O5	79.56 (2)	N1—O5—Sc1	94.62 (4)
O1—Sc1—O5	86.94 (2)	N1—O6—Sc1	95.45 (4)
O3—Sc1—O5	73.561 (19)	O7—N1—O5	122.93 (6)
O6—Sc1—O5	55.610 (18)	O7—N1—O6	122.90 (6)
O2—Sc1—Sc1 ⁱ	36.441 (14)	O5—N1—O6	114.17 (5)
O2 ⁱ —Sc1—Sc1 ⁱ	36.388 (14)		
Sc1—O5—N1—O7	176.54 (7)	Sc1—O6—N1—O7	-176.50 (7)
Sc1—O5—N1—O6	-3.65 (6)	Sc1—O6—N1—O5	3.69 (6)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots O6 ⁱⁱ	0.782 (16)	2.051 (16)	2.8175 (7)	166.6 (16)
O1—H1B \cdots C11 ⁱ	0.787 (15)	2.300 (15)	3.0856 (6)	176.6 (14)
O2—H2 \cdots C11 ⁱⁱⁱ	0.692 (16)	2.617 (16)	3.2749 (5)	159.7 (17)
O3—H3A \cdots C11 ⁱⁱⁱ	0.815 (16)	2.305 (16)	3.1017 (6)	165.9 (15)
O3—H3B \cdots O5 ^{iv}	0.811 (15)	1.989 (15)	2.7977 (7)	175.0 (14)
O4—H4A \cdots C11 ^v	0.859 (15)	2.316 (15)	3.1722 (6)	174.5 (13)
O4—H4B \cdots C11	0.824 (16)	2.242 (16)	3.0658 (6)	179.0 (15)

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