

Poly[hemi(ethylenediammonium) [di- μ -oxalato-indium(III)] dihydrate]

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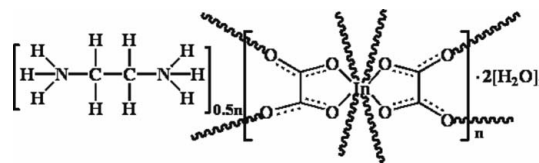
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; disorder in solvent or counterion; R factor = 0.018; wR factor = 0.046; data-to-parameter ratio = 10.5.

In title compound, $\{(\text{C}_2\text{H}_{10}\text{N}_2)_{0.5}[\text{In}(\text{C}_2\text{O}_4)_2] \cdot 2\text{H}_2\text{O}\}_n$, the unique In^{III} ion is coordinated by eight O atoms from four oxalate ligands in a distorted square-antiprismatic environment. The doubly bis-chelating oxalate ligands act as bridging ligands connecting symmetry-related In^{III} ions and forming a three-dimensional open framework structure. Ethylenediammonium cations and water molecules occupy the voids within the structure. The unique ethylenediammonium cation and one water molecule both lie on a twofold rotation axis. One of the other two water molecules residing on general crystallographic sites was refined as disordered with half occupancy. In the crystal structure, cations and water molecules are linked to the anionic framework *via* intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds.

Related literature

For background information on open-framework materials, see: Fang *et al.* (2004); Li *et al.* (2008); Serre *et al.* (2006); Sun *et al.* (2006). For related materials containing the oxalate ligand, see: Audebrand *et al.* (2001, 2004); Kokunov *et al.* (2004); Stock *et al.* (2000); Chakrabarti & Natarajan (2002); Evans & Lin (2001); Vaidhyanathan *et al.* (2001); Gavilan *et al.* (2007); Bataille *et al.* (2000); Trombe *et al.* (2001); Yuan *et al.* (2004). For indium oxalates, see: Audebrand *et al.* (2003); Bulc *et al.* (1983); Bulc & Golič (1983); Chen *et al.* (2003); Huang & Lii (1998); Jeanneau *et al.* (2003); Yang *et al.* (2005); For the bond-valence method, see: Brown (1996). For bond distances and angles for bridging bidentate oxalate groups, see: Hann (1957).



Experimental

Crystal data

$(\text{C}_2\text{H}_{10}\text{N}_2)_{0.5}[\text{In}(\text{C}_2\text{O}_4)_2] \cdot 2\text{H}_2\text{O}$
 $M_r = 357.95$
 Orthorhombic, *Fdd2*
 $a = 15.8498$ (4) Å
 $b = 31.1643$ (8) Å
 $c = 8.6618$ (2) Å

$V = 4278.48$ (18) Å³
 $Z = 16$
 Mo $K\alpha$ radiation
 $\mu = 2.26$ mm⁻¹
 $T = 293$ K
 $0.40 \times 0.38 \times 0.38$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
 $T_{\text{min}} = 0.426$, $T_{\text{max}} = 0.467$
 (expected range = 0.387–0.424)

7189 measured reflections
 1679 independent reflections
 1673 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.018$
 $wR(F^2) = 0.046$
 $S = 1.06$
 1679 reflections
 160 parameters
 13 restraints

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.45$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.71$ e Å⁻³
 Absolute structure: Flack (1983),
 668 Friedel pairs
 Flack parameter: 0.00 (3)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1C···OW1 ⁱ	0.89	2.35	2.880 (8)	118
N1—H1B···O7 ⁱⁱ	0.89	2.47	2.956 (5)	115
N1—H1B···OW3	0.89	2.21	2.825 (8)	126
N1—H1C···O4 ⁱ	0.89	2.44	3.140 (6)	136
N1—H1C···O5 ⁱⁱⁱ	0.89	2.38	3.166 (5)	147
OW1—HW1A···O2 ^{iv}	0.85	2.04	2.889 (5)	180
OW2—HW2B···O1 ⁱⁱⁱ	0.85	2.46	3.241 (15)	153
OW2—HW2A···O3	0.85	2.39	3.12 (3)	145
OW3—HW3B···O8 ^v	0.85	2.19	2.870 (6)	137
OW3—HW3A···O7	0.85	2.26	2.971 (7)	141
OW3—HW3A···O3 ⁱⁱ	0.85	2.40	2.962 (6)	124

Symmetry codes: (i) $x, y, z - 1$; (ii) $x + \frac{1}{4}, -y + \frac{3}{4}, z - \frac{1}{4}$; (iii) $-x, -y + \frac{1}{2}, z - \frac{1}{2}$; (iv) $-x + \frac{1}{2}, -y + \frac{1}{2}, z$; (v) $x - \frac{1}{4}, -y + \frac{3}{4}, z - \frac{3}{4}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2779).

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Poly[hemi(ethylenediammonium) [di- μ -oxalato-indium(III)] dihydrate]

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Comment

The synthesis of open-framework materials has emerged as an important area of research because of their potential applications in separation processes, ion exchange and catalysis. In the past few years, there has been considerable effort in designing open-framework structures formed by metal organic carboxylates because of its interesting structural features and the quality for apt design (Fang *et al.*, 2004; Li *et al.*, 2008; Serre *et al.*, 2006; Sun *et al.*, 2006) of which the oxalate ligand plays a major role in the assembly of metal-organic porous frameworks. Many metal oxalate structures are reported such as tin (Audebrand *et al.*, 2001; Kokunov *et al.*, 2004; Stock *et al.*, 2000), zinc (Chakrabarti & Natarajan, 2002; Rvans & Lin, 2001; Vaidhyanathan *et al.*, 2001), zirconium (Audebrand *et al.*, 2004; Gavilan *et al.*, 2007), rare earth (Bataille *et al.*, 2000; Trombe *et al.*, 2001; Yuan *et al.*, 2004). The structures of these compounds vary from monomers, dimmers, chains, layered honeycomb networks to three dimensional frameworks. In this paper, we selected indium and synthesized the three-dimensional indium oxalate compound $[(C_2N_2H_{10})_{0.5}In(C_2O_4)_2 \cdot 2H_2O]_n$ (Fig. 1). Although many indium oxalates have been reported (Audebrand *et al.*, 2003; Bulc *et al.*, 1983; Chen *et al.*, 2003; Huang & Lii, 1998; Jeanneau *et al.*, 2003; Yang *et al.*, 2005), relatively a few of them are three dimensional open frameworks (Chen *et al.*, 2003; Huang & Lii, 1998; Yang *et al.*, 2005).

In the title structure, the In ion is coordinated by eight O atoms from four tetradentate oxalate groups, forming a distorted square antiprismatic arrangement (Fig. 2) in which atoms O1, O2, O5A and O6A (Symmetry code A: $-x, 0.5 - y, 1/2 + z$) are approximately in the same plane with a deviation of ca. 0.01 Å, while the other plane (formed by atoms O3, O4, O7B and O8B; Symmetry code B: $x - 1/4, 0.75 - y, 1/4 + z$) is significantly distorted, with a deviation of ca. 0.24 Å. The eight In—O bond distances vary between 2.168 (3) and 2.423 (3) Å (average 2.279 Å), which agrees well with the value 2.265 Å calculated for an eightfold coordinated indium atom with the bond valence method using the program VALENCE (Brown, 1996).

The indium ions are linked by the oxygen atoms of oxalate, giving rise to a three-dimensional interdependent porous framework (Fig. 3). The protonated ethylenediammonium and water molecules occupy the voids, interacting with oxalate anions through N—H \cdots O and O—H \cdots O hydrogen bonds. Without water molecules and cations, the framework exhibits voids possessing approximate dimensions of 6.9 \times 14.5 Å along the crystallographic *c* axis and an analysis of the void shows that ca 44% of the space is empty. Thus, the ethylenediammonium and water molecules assigned to these cavities act as not only charge-compensating cations but also organic templates. The bond distances and angles for the bridging bidentate oxalate groups are in good agreement with the mean values reported by Hann (1957) for oxalate compounds, *i.e.*, 1.24 and 1.52 Å, 118 and 123° for the C1—O1 and C1—C2 bond lengths and O1—C1—C2 and O1—C1—O5 angles, respectively.

Experimental

A mixture of Ti(SO₄)₂ (0.2 g, 0.84 mmol), H₂C₂O₄·2H₂O (1.0 g, 7.93 mmol), InCl₃·4H₂O (2 ml, 0.5 mol/L) and H₂N(CH₂)₂NH₂ (0.2 ml, CR) in H₂O (5.0 ml) was sealed in a 20 ml stainless-steel reactor with Teflon liner and heated

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at 393 K for 2 days under autogenously pressure. Colorless crystals were isolated after the reaction solution was cooled gradually and washed with water.

Refinement

H atoms bonded to C and N atoms were included in calculated positions with C-H = 0.97 and N-H = 0.89 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{N})$. The H atoms bonded to O atoms were either included in calculated positions [O-H = 0.85 Å] based on 'as found' locations or based on the most efficient H-bonding location and with $U_{\text{iso}}(\text{H}) = 1.0-1.2U_{\text{eq}}(\text{C})$.

Figures

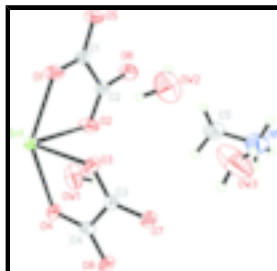


Fig. 1. The asymmetric unit showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

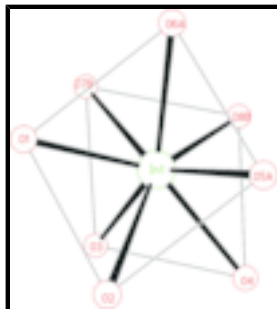


Fig. 2. The distorted square antiprismatic environment of Indium. Symmetry codes A: $-x, 0.5 - y, 1/2 + z$; B: $x - 1/4, 0.75 - y, 1/4 + z$.

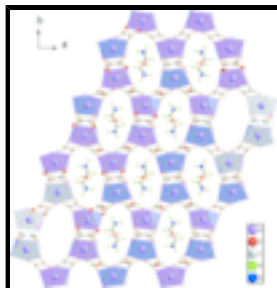


Fig. 3. Part of the crystal structure viewed crystal along the c axis, the ethylenediammonium and water molecules reside in the voids.

Poly[hemi(ethylenediammonium) [di- μ -oxalato-indium(III)] dihydrate]

Crystal data

$(\text{C}_2\text{H}_{10}\text{N}_2)_{0.5}[\text{In}(\text{C}_2\text{O}_4)_2] \cdot 2\text{H}_2\text{O}$

$M_r = 357.95$

Orthorhombic, $Fdd2$

Hall symbol: $F 2 -2d$

$F_{000} = 2800$

$D_x = 2.223 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 7228 reflections

$a = 15.8498 (4) \text{ \AA}$	$\theta = 2.6\text{--}27.9^\circ$
$b = 31.1643 (8) \text{ \AA}$	$\mu = 2.26 \text{ mm}^{-1}$
$c = 8.6618 (2) \text{ \AA}$	$T = 293 \text{ K}$
$V = 4278.48 (18) \text{ \AA}^3$	Block, colourless
$Z = 16$	$0.4 \times 0.38 \times 0.38 \text{ mm}$

Data collection

Bruker SMART CCD diffractometer	1679 independent reflections
Radiation source: fine-focus sealed tube	1673 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.025$
$T = 293 \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.6^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -18 \rightarrow 18$
$T_{\text{min}} = 0.426$, $T_{\text{max}} = 0.467$	$k = -36 \rightarrow 36$
7189 measured reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.018$	$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2 + 7.4478P]$
$wR(F^2) = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\text{max}} = 0.001$
1679 reflections	$\Delta\rho_{\text{max}} = 0.45 \text{ e \AA}^{-3}$
160 parameters	$\Delta\rho_{\text{min}} = -0.71 \text{ e \AA}^{-3}$
13 restraints	Extinction correction: SHELXTL (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00087 (5)
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 668 Friedel pairs
	Flack parameter: 0.00 (3)

Special details

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

supplementary materials

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
In1	0.013694 (15)	0.314689 (7)	0.33266 (6)	0.01510 (11)	
O1	-0.06449 (17)	0.27864 (9)	0.1683 (4)	0.0277 (6)	
O2	0.10165 (19)	0.26861 (9)	0.1750 (4)	0.0290 (7)	
O3	0.04152 (19)	0.35822 (9)	0.1416 (3)	0.0241 (6)	
O4	0.14920 (17)	0.34173 (8)	0.3713 (3)	0.0220 (6)	
C1	-0.0334 (2)	0.25113 (11)	0.0817 (7)	0.0194 (7)	
C2	0.0620 (2)	0.24552 (11)	0.0823 (7)	0.0204 (7)	
C3	0.1128 (2)	0.37559 (10)	0.1357 (4)	0.0165 (7)	
C4	0.1740 (2)	0.36645 (11)	0.2696 (5)	0.0180 (8)	
O5	-0.07692 (18)	0.22769 (9)	-0.0061 (3)	0.0259 (7)	
O6	0.09025 (18)	0.21856 (9)	-0.0112 (4)	0.0302 (7)	
O7	0.13838 (16)	0.39927 (8)	0.0302 (3)	0.0223 (6)	
O8	0.24530 (18)	0.38498 (9)	0.2625 (4)	0.0231 (6)	
C5	0.2294 (3)	0.2715 (2)	-0.2277 (8)	0.0535 (16)	
H5C	0.1687	0.2676	-0.2275	0.064*	
H5A	0.2444	0.2864	-0.1332	0.064*	
N1	0.2527 (3)	0.29883 (17)	-0.3608 (6)	0.0502 (12)	
H1A	0.3035	0.2913	-0.3949	0.075*	
H1B	0.2534	0.3262	-0.3316	0.075*	
H1C	0.2150	0.2954	-0.4360	0.075*	
OW1	0.2500	0.2500	0.3570 (7)	0.0504 (16)	
HW1A	0.2938	0.2445	0.3040	0.050*	
OW2	0.0119 (6)	0.3117 (3)	-0.173 (3)	0.069 (2)	0.50
HW2B	0.0188	0.2848	-0.1847	0.083*	0.50
HW2A	0.0010	0.3173	-0.0793	0.083*	0.50
OW3	0.1418 (3)	0.36723 (19)	-0.2928 (7)	0.1000 (17)	
HW3B	0.1123	0.3788	-0.3632	0.120*	
HW3A	0.1542	0.3857	-0.2244	0.120*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
In1	0.01446 (16)	0.01545 (14)	0.01540 (15)	-0.00012 (8)	-0.00093 (14)	0.00069 (11)
O1	0.0185 (13)	0.0268 (13)	0.0378 (17)	-0.0013 (11)	0.0018 (14)	-0.0157 (13)
O2	0.0224 (15)	0.0289 (15)	0.0358 (17)	0.0008 (12)	-0.0078 (14)	-0.0104 (13)
O3	0.0187 (14)	0.0299 (14)	0.0237 (15)	-0.0058 (12)	-0.0031 (12)	0.0086 (11)
O4	0.0264 (15)	0.0220 (12)	0.0176 (15)	-0.0037 (10)	-0.0021 (11)	0.0065 (10)
C1	0.0209 (18)	0.0154 (15)	0.0218 (17)	-0.0017 (14)	0.001 (2)	0.0002 (16)
C2	0.0220 (19)	0.0194 (16)	0.0199 (17)	0.0002 (14)	0.000 (2)	-0.0018 (17)
C3	0.0177 (19)	0.0139 (15)	0.0179 (17)	0.0038 (13)	0.0005 (15)	0.0001 (14)
C4	0.019 (2)	0.0132 (15)	0.0214 (17)	0.0010 (14)	-0.0015 (16)	-0.0011 (14)
O5	0.0193 (15)	0.0251 (13)	0.0333 (17)	-0.0004 (11)	-0.0037 (13)	-0.0135 (13)
O6	0.0213 (15)	0.0324 (15)	0.0369 (19)	0.0024 (12)	0.0032 (14)	-0.0131 (14)
O7	0.0224 (13)	0.0229 (12)	0.0217 (14)	-0.0042 (10)	-0.0032 (11)	0.0066 (10)

O8	0.0180 (13)	0.0273 (15)	0.0239 (15)	-0.0057 (11)	-0.0039 (13)	0.0084 (12)
C5	0.031 (3)	0.080 (4)	0.049 (4)	-0.020 (2)	0.006 (3)	-0.016 (3)
N1	0.028 (2)	0.074 (3)	0.049 (3)	-0.006 (2)	-0.011 (2)	-0.001 (2)
OW1	0.026 (2)	0.086 (4)	0.039 (4)	0.024 (2)	0.000	0.000
OW2	0.084 (4)	0.067 (4)	0.055 (4)	0.013 (4)	-0.002 (5)	-0.006 (4)
OW3	0.083 (3)	0.146 (4)	0.072 (3)	0.016 (3)	-0.030 (3)	-0.045 (3)

Geometric parameters (Å, °)

In1—O5 ⁱ	2.168 (3)	C4—O8	1.270 (5)
In1—O3	2.185 (3)	O5—In1 ⁱⁱⁱ	2.168 (3)
In1—O1	2.196 (3)	O6—In1 ⁱⁱⁱ	2.370 (3)
In1—O8 ⁱⁱ	2.230 (3)	O7—In1 ^{iv}	2.327 (3)
In1—O7 ⁱⁱ	2.327 (3)	O8—In1 ^{iv}	2.230 (3)
In1—O4	2.331 (3)	C5—N1	1.480 (8)
In1—O6 ⁱ	2.370 (3)	C5—C5 ^v	1.492 (12)
In1—O2	2.423 (3)	C5—H5C	0.9700
O1—C1	1.242 (5)	C5—H5A	0.9700
O2—C2	1.248 (6)	N1—H1A	0.8900
O3—C3	1.253 (5)	N1—H1B	0.8900
O4—C4	1.235 (5)	N1—H1C	0.8900
C1—O5	1.260 (6)	OW1—HW1A	0.8500
C1—C2	1.522 (6)	OW2—HW2B	0.8502
C2—O6	1.250 (6)	OW2—HW2A	0.8500
C3—O7	1.243 (4)	OW3—HW3B	0.8498
C3—C4	1.539 (5)	OW3—HW3A	0.8500
O5 ⁱ —In1—O3	140.63 (11)	C4—O4—In1	114.7 (2)
O5 ⁱ —In1—O1	111.52 (10)	O1—C1—O5	123.2 (4)
O3—In1—O1	86.60 (12)	O1—C1—C2	118.2 (4)
O5 ⁱ —In1—O8 ⁱⁱ	92.22 (11)	O5—C1—C2	118.7 (4)
O3—In1—O8 ⁱⁱ	96.82 (11)	O2—C2—O6	128.6 (4)
O1—In1—O8 ⁱⁱ	137.63 (10)	O2—C2—C1	115.8 (4)
O5 ⁱ —In1—O7 ⁱⁱ	144.54 (11)	O6—C2—C1	115.5 (4)
O3—In1—O7 ⁱⁱ	74.02 (11)	O7—C3—O3	125.5 (4)
O1—In1—O7 ⁱⁱ	68.82 (10)	O7—C3—C4	117.2 (3)
O8 ⁱⁱ —In1—O7 ⁱⁱ	71.65 (10)	O3—C3—C4	117.3 (3)
O5 ⁱ —In1—O4	72.66 (9)	O4—C4—O8	127.0 (4)
O3—In1—O4	72.43 (9)	O4—C4—C3	116.8 (3)
O1—In1—O4	142.91 (10)	O8—C4—C3	116.1 (3)
O8 ⁱⁱ —In1—O4	76.46 (10)	C1—O5—In1 ⁱⁱⁱ	119.2 (3)
O7 ⁱⁱ —In1—O4	129.79 (9)	C2—O6—In1 ⁱⁱⁱ	114.5 (3)
O5 ⁱ —In1—O6 ⁱ	71.78 (10)	C3—O7—In1 ^{iv}	115.0 (2)
O3—In1—O6 ⁱ	147.56 (11)	C4—O8—In1 ^{iv}	118.0 (3)
O1—In1—O6 ⁱ	75.75 (11)	N1—C5—C5 ^v	114.0 (4)

supplementary materials

O8 ⁱⁱ —In1—O6 ⁱ	79.52 (11)	N1—C5—H5C	108.7
O7 ⁱⁱ —In1—O6 ⁱ	74.28 (10)	C5 ^v —C5—H5C	108.7
O4—In1—O6 ⁱ	135.78 (10)	N1—C5—H5A	108.7
O5 ⁱ —In1—O2	74.70 (10)	C5 ^v —C5—H5A	108.7
O3—In1—O2	79.93 (11)	H5C—C5—H5A	107.6
O1—In1—O2	69.90 (11)	C5—N1—H1A	109.5
O8 ⁱⁱ —In1—O2	152.36 (10)	C5—N1—H1B	109.5
O7 ⁱⁱ —In1—O2	131.86 (10)	H1A—N1—H1B	109.5
O4—In1—O2	76.41 (9)	C5—N1—H1C	109.5
O6 ⁱ —In1—O2	117.54 (10)	H1A—N1—H1C	109.5
C1—O1—In1	121.4 (3)	H1B—N1—H1C	109.5
C2—O2—In1	114.5 (3)	HW2B—OW2—HW2A	109.8
C3—O3—In1	118.7 (2)	HW3B—OW3—HW3A	109.8

Symmetry codes: (i) $-x, -y+1/2, z+1/2$; (ii) $x-1/4, -y+3/4, z+1/4$; (iii) $-x, -y+1/2, z-1/2$; (iv) $x+1/4, -y+3/4, z-1/4$; (v) $-x+1/2, -y+1/2, z$.

Hydrogen-bond geometry ($\text{\AA}, \text{\circ}$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1C \cdots OW1 ^{vi}	0.89	2.35	2.880 (8)	118
N1—H1B \cdots O7 ^{iv}	0.89	2.47	2.956 (5)	115
N1—H1B \cdots OW3	0.89	2.21	2.825 (8)	126
N1—H1C \cdots O4 ^{vi}	0.89	2.44	3.140 (6)	136
N1—H1C \cdots O5 ⁱⁱⁱ	0.89	2.38	3.166 (5)	147
OW1—HW1A \cdots O2 ^v	0.85	2.04	2.889 (5)	180
OW2—HW2B \cdots O1 ⁱⁱⁱ	0.85	2.46	3.241 (15)	153
OW2—HW2A \cdots O3	0.85	2.39	3.12 (3)	145
OW3—HW3B \cdots O8 ^{vii}	0.85	2.19	2.870 (6)	137
OW3—HW3A \cdots O7	0.85	2.26	2.971 (7)	141
OW3—HW3A \cdots O3 ^{iv}	0.85	2.40	2.962 (6)	124

Symmetry codes: (vi) $x, y, z-1$; (iv) $x+1/4, -y+3/4, z-1/4$; (iii) $-x, -y+1/2, z-1/2$; (v) $-x+1/2, -y+1/2, z$; (vii) $x-1/4, -y+3/4, z-3/4$.

Fig. 1

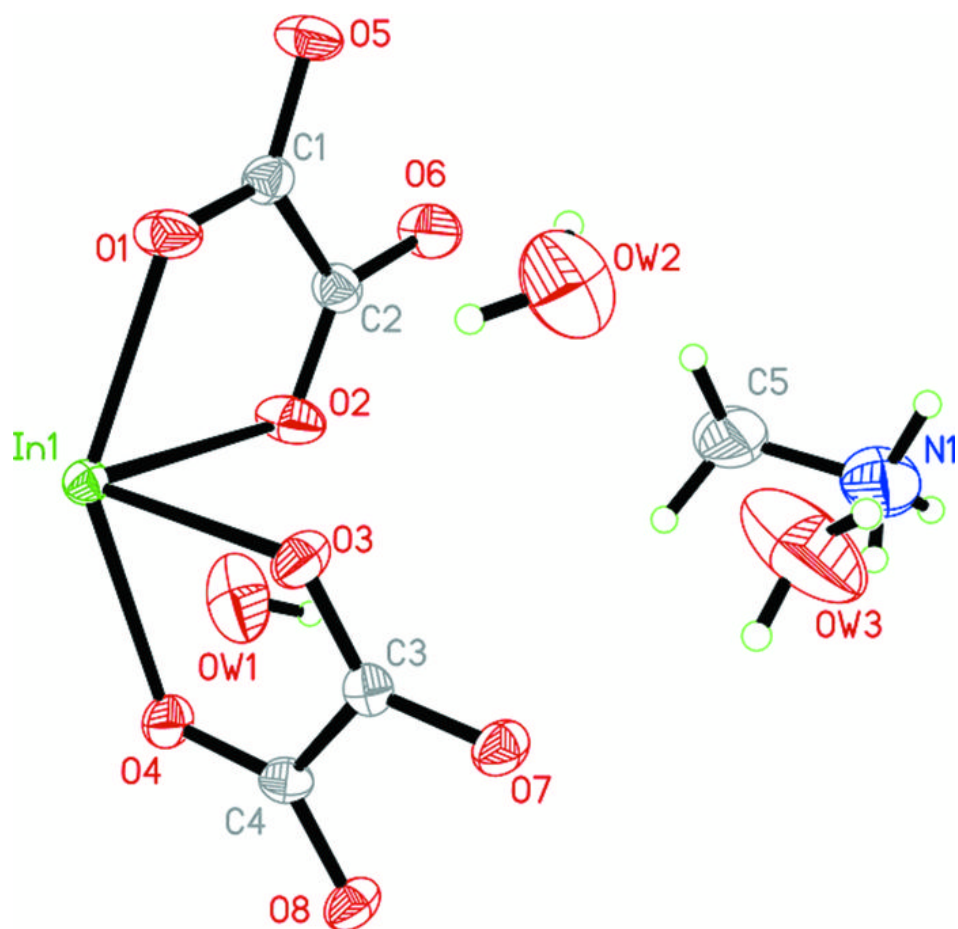


Fig. 2

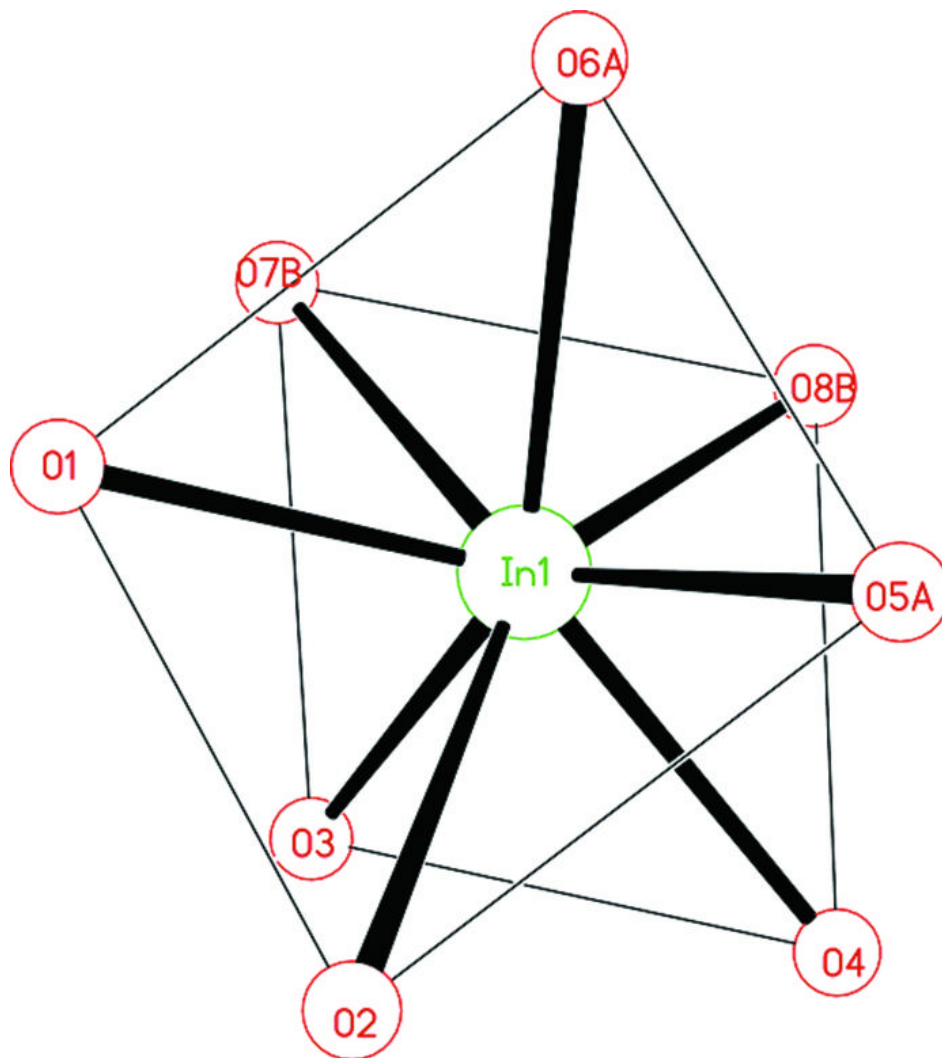


Fig. 3

