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Key indicators

Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(C-C) = 0.004$ Å
 R factor = 0.023
 wR factor = 0.039
Data-to-parameter ratio = 14.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.An indium(III) complex with pyridine-
2,6-dicarboxylate

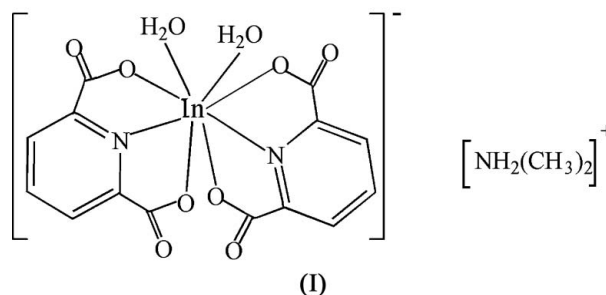
In the title compound, dimethylammonium diaquabis(pyridine-2,6-dicarboxylato- $\kappa^3 O^2, N, O^6$)indate(III), $(C_2H_8N)[In(C_7H_3NO_4)_2(H_2O)_2]$, the central In^{III} ion is eight-coordinated by two pyridine-2,6-dicarboxylate ligands and two water molecules. The water molecules, the dimethylammonium ion and the carboxylate O atoms interact through hydrogen bonds, forming a three-dimensional supramolecular structure.

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Comment

Development of supramolecular synthons is an important aspect of crystal engineering as it generates new structures with novel architectures and topologies which, in turn, lead to new materials and properties (Zhang & Chen, 2005). Crystals built from metal ions and organic acids have received considerable attention in the planned assembly of supramolecular architectures (Che *et al.*, 2006). Recently, aromatic di- or poly(carboxylic acids) have been investigated considerably in the area of solid state and materials science (Ma *et al.*, 2003). For instance, pyridine-2,6-dicarboxylate (pdc) was employed in the preparation of metal-based functional solids with interesting structures (Zhao *et al.*, 2003). In this work, we present a new compound, $[NH_2(CH_3)_2][In(pdc)_2(H_2O)_2]$, (I).



Compound (I) was obtained under solvothermal conditions at 413 K. The compound, once formed, is insoluble in most solvents. The structure of compound (I) can be described as a three-dimensional hydrogen-bonded supramolecular structure. The asymmetric unit of (I) is shown in Fig. 1. The central In^{III} ion is eight-coordinated by four carboxylate O atoms, two N atoms from two pdc ligands and two water molecules (Fig. 1). Selected bond lengths and angles are given in Table 1.

The dimethylammonium ion in (I) is believed to be the result of decomposition of the DMF (*N,N'*-dimethylformamide) solvent under solvothermal conditions. This phenomenon has been commonly observed in the literature (Mahon *et al.*, 2005). When the reaction was carried out in different solvents, such as ethanol, tetrahydrofuran and

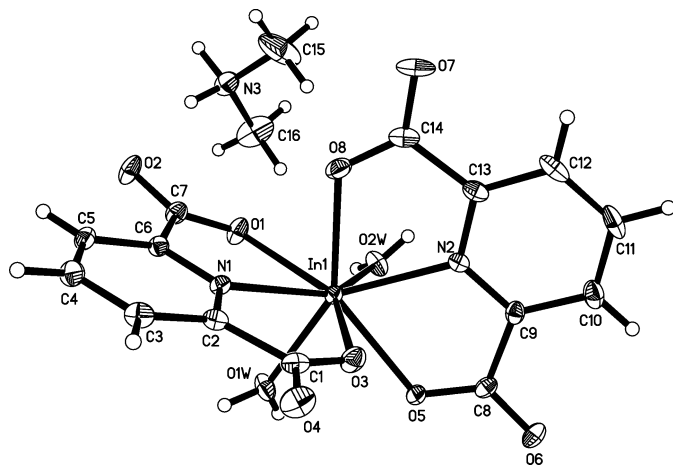


Figure 1
View of the asymmetric unit of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

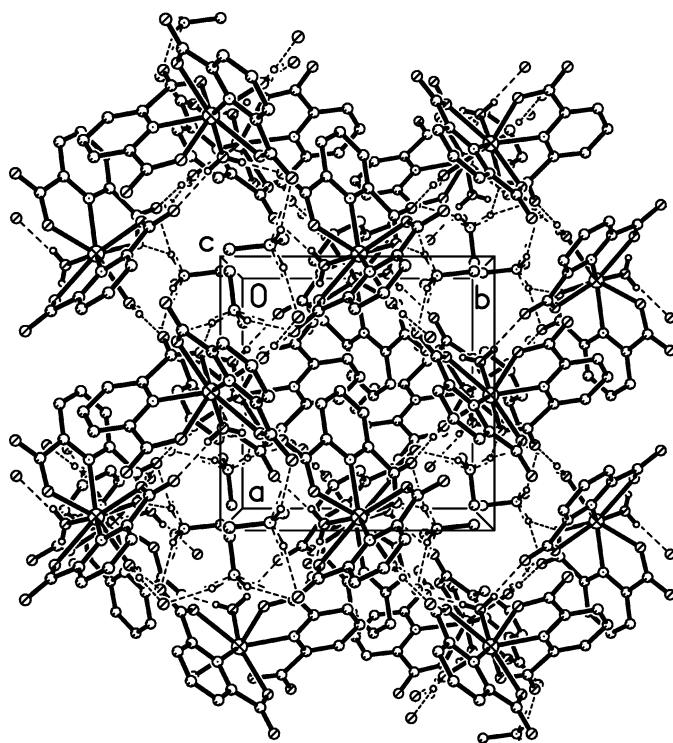


Figure 2
Packing of (I), viewed along the *c* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

ethylene glycol, compound (I) was not obtained. This implies that DMF decomposition plays an important role in the formation of (I).

Molecules of (I) are held together through hydrogen bonds, forming a three-dimensional structure (Fig. 2). Hydrogen-bonding details are listed in Table 2. There are six H atoms available for hydrogen-bonding interactions in the asymmetric unit, from two water molecules and one dimethylammonium ion. The carboxylate O atoms of pdc are also involved in hydrogen bonds and play the role of acceptor.

Experimental

InCl₃·6H₂O (0.385 g, 1.0 mmol) and H₂pdc (0.247 g, 1.5 mmol) were dissolved in DMF (10 ml). The solution was heated in a 25 ml Teflon-lined reaction vessel at 413 K for 120 h and then cooled to room temperature over a period of 16 h. Colourless crystals of (I) were obtained in a yield of 77%.

Crystal data

(C₂H₈N)[In(C₇H₃NO₄)₂(H₂O)₂]
M_r = 527.15
 Tetragonal, *P*4₁
a = 9.7681 (14) Å
c = 19.632 (4) Å
V = 1873.2 (5) Å³
Z = 4
D_x = 1.869 Mg m⁻³

Mo Kα radiation
 Cell parameters from 18141 reflections
 θ = 2.1–27.5°
 μ = 1.33 mm⁻¹
T = 292 (2) K
 Block, colourless
 0.41 × 0.37 × 0.34 mm

Data collection

Rigaku R-Axis RAPID
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
T_{min} = 0.546, *T_{max}* = 0.637
 18141 measured reflections

4295 independent reflections
 3739 reflections with *I* > 2σ(*I*)
R_{int} = 0.040
 θ_{max} = 27.5°
h = -12 → 11
k = -12 → 12
l = -25 → 25

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.024
wR (*F*²) = 0.039
S = 0.91
 4295 reflections
 295 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0188P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 $\Delta\rho_{max}$ = 0.27 e Å⁻³
 $\Delta\rho_{min}$ = -0.44 e Å⁻³
 Absolute structure: Flack (1983),
 with 2080 Friedel pairs
 Flack parameter: -0.012 (15)

Table 1

Selected geometric parameters (Å, °).

N1–In1	2.291 (2)	O5–In1	2.3420 (19)
N2–In1	2.266 (2)	O8–In1	2.281 (2)
O1–In1	2.364 (2)	O1W–In1	2.212 (3)
O3–In1	2.291 (2)	O2W–In1	2.212 (2)
O1W–In1–O2W	93.02 (10)	N1–In1–O3	69.44 (8)
O1W–In1–N2	141.69 (9)	O1W–In1–O5	72.49 (9)
O2W–In1–N2	78.13 (9)	O2W–In1–O5	77.00 (9)
O1W–In1–O8	147.92 (9)	N2–In1–O5	69.20 (7)
O2W–In1–O8	93.05 (9)	O8–In1–O5	139.49 (8)
N2–In1–O8	70.33 (8)	N1–In1–O5	133.36 (8)
O1W–In1–N1	79.11 (9)	O3–In1–O5	76.27 (8)
O2W–In1–N1	141.60 (9)	O1W–In1–O1	76.95 (9)
N2–In1–N1	129.52 (8)	O2W–In1–O1	73.05 (9)
O8–In1–N1	76.65 (8)	N2–In1–O1	132.87 (8)
O1W–In1–O3	93.75 (10)	O8–In1–O1	74.81 (8)
O2W–In1–O3	148.95 (9)	N1–In1–O1	68.55 (8)
N2–In1–O3	77.84 (8)	O3–In1–O1	137.96 (8)
O8–In1–O3	97.07 (8)	O5–In1–O1	135.46 (8)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—HW12 \cdots O2 ⁱ	0.74 (3)	1.96 (3)	2.698 (3)	171 (3)
O1W—HW11 \cdots O6 ⁱⁱ	0.84 (4)	1.88 (2)	2.721 (3)	177 (4)
O2W—HW21 \cdots O4 ⁱⁱⁱ	0.77 (4)	1.90 (4)	2.661 (3)	169 (4)
N3—H31 \cdots O2	0.83 (4)	2.07 (4)	2.859 (4)	158 (4)
N3—H32 \cdots O6 ^{iv}	0.99 (4)	1.92 (4)	2.884 (4)	167 (3)

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

All H atoms on C atoms were positioned geometrically and refined as riding atoms, with $C-H = 0.93 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The H atoms of the water molecules and those attached to N atoms were located in difference Fourier maps and refined freely. O—H and N—H distances are given in Table 2.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997);

program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXTL*

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