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fac-[Bis(2-pyridylmethyl)amine-κ^3N]tribromoindium(III)

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Key indicators

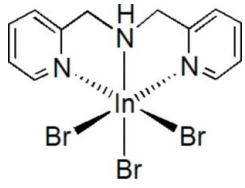
Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.012 Å
R factor = 0.053
wR factor = 0.125
Data-to-parameter ratio = 20.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, *fac*-[$InBr_3${$NH(CH_2C_5H_4N)_2$}], the In^{III} atom is coordinated by three bromide ligands and a tridentate bis(2-pyridylmethyl)amine ligand. A mirror plane passes through the In atom, one Br atom and central NH group. The molecule exhibits facial octahedral stereochemistry, with In—Br bond lengths of 2.5682 (13) and 2.6060 (9) Å, and In—N bond lengths of 2.311 (6) and 2.328 (9) Å.

Comment

A limited number of crystal structures have been reported to date for complexes of indium(III) halides of the type InX_3L, where L is a tridentate N,N',N''-donor ligand, *viz*. [$InCl_3L$] [L = 2,6-bis(1-phenyliminoethyl)pyridine (Abram *et al.*, 1997), 2-[2-(pyridylamino)phenylazo]pyridine (Das *et al.*, 2003) and 2,6-di-2-pyridylpyridine (Butcher *et al.*, 2003)] and [$InBr_3L$] (L = 1,4,7-trimethyl-1,4,7-triazacyclononane; Willey *et al.*, 2001). We now report the isolation and structural characterization of the title compound, (I).



(I)

The molecular structure of (I) reveals that the In^{III} atom has three Br ligands with a facial geometry and a tridentate bis(2-pyridylmethyl)amine (bpa) ligand (Fig. 1). A mirror plane passes through In1, Br1, N1 and H1N. Only one crystal structure has been reported to date for a six-coordinate complex formulated as MX_3(bpa) (X = halogen); [$FeCl_3$(bpa)] (Viswanathan *et al.*, 1996). The In—N bond distances in (I) (Table 1) are similar to those observed in [$InBr_3(C_5H_5N)_3$] [2.32 (2) Å; Small & Worrall, 1982] and in [$InBr_3${–N(Me)—CH_2–}$_3$] [2.338 (7), 2.355 (8) and 2.360 (7) Å; Willey *et al.*, 2001]. The In—Br bond *trans* to the bridging amino group [2.5682 (13) Å] is shorter than that *trans* to the pyridyl ligand [2.6060 (9) Å]. The former is shorter than the In—Br bond distances of 2.5974 (11), 2.6053 (11) and 2.6054 (11) Å in [$InBr_3${–N(Me)—CH_2–}$_3$], whereas the latter distance is slightly longer than that of 2.593 (3) Å in [$InBr_3(C_5H_5N)_3$]. The constriction of the N—In—N bond angles allows relaxation of the Br—In—Br bond angles in (I), as observed previously (Willey *et al.*, 2001).

Experimental

A solution of bis(2-pyridylmethyl)amine (0.448 g, 2.25 mmol) in diethyl ether (10 ml) was added dropwise to a solution of $InBr_3$

(0.785 g, 2.21 mmol) in diethyl ether (10 ml). The resulting white suspension was stirred at ambient temperature for 1 h, then the solution was filtered. The white solid was washed repeatedly with diethyl ether (10 ml × 5 times) and dried *in vacuo* to give (I) (yield 1.167 g, 2.10 mmol, 95%). White crystals of (I) suitable for X-ray diffraction study were obtained by cooling a methanol solution at 253 K over a period of few days.

Crystal data

$[InBr_3(C_{12}H_{13}N_3)]$	$Z = 4$
$M_r = 553.80$	$D_x = 2.319$ Mg m^{-3}
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation
$a = 13.995$ (2) Å	$\mu = 9.04$ mm^{-1}
$b = 14.227$ (2) Å	$T = 203$ (2) K
$c = 7.9682$ (12) Å	Plate, colorless
$V = 1586.6$ (4) Å^3	0.12 × 0.06 × 0.02 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer	14768 measured reflections 1877 independent reflections
ω scans	1802 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (Jacobson, 1998)	$R_{int} = 0.056$
$T_{min} = 0.410$, $T_{max} = 0.840$	$\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 3P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.053$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.125$	$\Delta\rho_{max} = 0.84$ e Å^{-3}
$S = 1.01$	$\Delta\rho_{min} = -0.69$ e Å^{-3}
1877 reflections	
93 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

In1—N1	2.328 (9)	In1—Br1	2.5682 (13)
In1—N2	2.311 (6)	In1—Br2	2.6060 (9)
N1—In1—N2	74.1 (2)	Br1—In1—Br2^{i}	100.20 (3)
N2—In1—N2^{i}	75.5 (3)	Br2—In1—Br2^{i}	98.84 (4)

Symmetry code: (i) $x, -y+\frac{1}{2}, z$.

The amine H was located in a difference Fourier map and its positional parameters were refined, with $U_{iso}(H) = 1.2U_{eq}(N)$. All other H atoms were positioned geometrically, with methylene C—H = 0.98 Å and aromatic C—H = 0.94 Å, and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CrystalClear* (Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

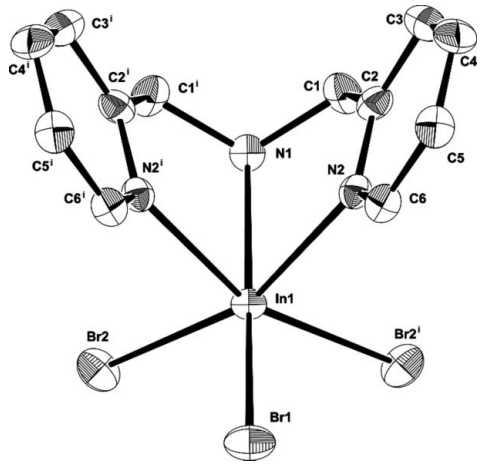


Figure 1
The molecule structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity [symmetry code: (i) $x, -y+\frac{1}{2}, z$].

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