Acta Crystallographica Section C
**Crystal Structure
Communications**

ISSN 0108-2701

[Hydroxytris(pentafluorophenyl)borato-*O*][*N*-isopropyl-2-(isopropylamino)-troponiminato-*N*]methylindium(**III**)

Ilia A. Guzei *et al.*

Electronic paper
This paper is published electronically. It meets the data-validation criteria for publication in Acta Crystallographica Section C. The submission has been checked by a Section C Co-editor though the text in the 'Comments' section is the responsibility of the authors.

Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

[Hydroxytris(pentafluorophenyl)borato-*O*][*N*-isopropyl-2-(isopropylamino)troponiminato-*N*]methyl-

Ilia A. Guzei,[a]* Fabien Delpech[b] and Richard F. Jordan[b]

[a]Department of Chemistry, Iowa State University of Science and Technology, Ames, Iowa 50011, USA, and [b]Department of Chemistry, The University of Chicago, 5735 South Ellis Avenue, Chicago, IL 60637, USA
Correspondence e-mail: iguzei@iastate.edu

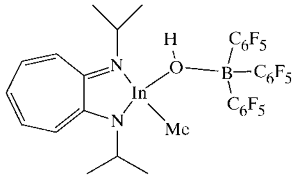
Received 13 June 2000
Accepted 10 July 2000

Data validation number: IUC0000184

The title complex, $[In(CH_3)(C_{18}HBF_{15}O)(C_{13}H_{19}N_2)]$, crystallizes as an ion pair linked by a μ-hydroxo bridge. There are two independent molecules in the asymmetric unit that exhibit essentially identical metric parameters, but different conformations.

Comment

Cationic group 13 complexes have been investigated because of their potential applications as catalysts for olefin polymerization, alkyne oligomerization and other reactions, and as general Lewis-acid catalysts (Bochmann & Dawson, 1996; Coles & Jordan, 1997; Atwood, 1998; Korolev *et al.*, 1999; Radzewich *et al.*, 1999; Dagorne *et al.*, 2000). In the course of our studies of cationic indium complexes containing *N,N'*-diisopropylaminotroponiminate (^{i}Pr$_2$–ATI) ligands (Delpech *et al.*, 2000), we isolated and structurally characterized the title compound, (I).



(I)

The molecular structure of (I) consists of an ion pair in which the $\{^i\mathrm{Pr}_2\text{–ATI}\}\mathrm{In(Me)}^+$ cation is associated with the $B(C_6F_5)_3(OH)^-$ anion through an In—O(H)—B bridge. The asymmetric unit of (I) contains two independent molecules with statistically equivalent metrical parameters but different conformations. The molecules differ in the positions of the methyl substituents of the isopropyl groups at atoms N2 and N2*A*, and in the conformation of the In—O(H)—$B(C_6F_5)_3$ unit. The latter difference is best illustrated by the torsion angles for the two molecules: B1—O1—In1—C8 42.3 (3)° and B2—O2—In2—C8*A* 27.8 (3)°. In the following discussion, the bond lengths and angles of (I) are reported as average values for the two molecules.

The In atom possesses a severely distorted tetrahedral geometry. The N—In—N bite angle [77.92 (6)°] is in the expected range for ^{i}Pr$_2$–ATI complexes (Korolev *et al.*, 1999; Ihara *et al.*, 1998). The O—In—N [average 104 (2)°] and O—In—C angles [average 105.6 (2)°] are similar, while the C—In—N angles are larger [average 130 (1)°]. The In—C [2.114 (3) Å] and In—N [2.103 (6) Å] bond lengths, and the N—In—N bite angle in (I) are similar to those in the closely related complex $[\{^i\mathrm{Pr}_2\text{–ATI}\}\mathrm{In(Me)(NMe_2Ph)}]^+$ [In—C 2.121 (2) Å; In—N(ATI) average 2.108 (4) Å; N—In—N 78.18 (6)°; Delpech *et al.*, 2000]. The In—O bond length [2.186 (9) Å] in (I) is similar to the In—μ-OH distances in $[\{InC(SiMe_3)_3\}_4(\mu_4\text{–}O)(\mu\text{-}OH)_6]$ [2.17 (2) Å; Al-Juaid *et al.*, 1986].

The five- and seven-membered ring of the $\{^i\mathrm{Pr}_2\text{–ATI}\}$In units are planar to within 0.08 Å in the In1 molecule and to within 0.01 Å in the In2 molecule. The isopropyl groups adopt orientations that minimize steric interactions with the closest H atoms on the seven-membered rings; consequently, the methyl groups point toward the In centre. The isopropyl group on atom N2 (but not N2*A* in the second molecule) is disordered over two positions in a 72/28 ratio and was refined isotropically with an idealized geometry. The C—N bond distances in the InN_2C_2 chelate ring of (I) [average 1.339 (6) Å] are intermediate between the normal C*sp*^{2}—N single-bond (1.47 Å) and C═N double-bond distances (1.27 Å), which is consistent with significant delocalization of the π-electrons within the ATI unit, as observed in related compounds (Ihara *et al.*, 1998; Korolev *et al.*, 1999).

The $[B(C_6F_5)_3(OH)]^-$ anion exhibits a slightly distorted tetrahedral geometry. The B—O bond distance [1.532 (4) Å] is similar to the B—O bond lengths in related compounds, *e.g.* $[(C_5Me_5)_2Ta(OH)Me][B(C_6F_5)_3OH]$ [1.490 (10) Å; Schaefer *et al.*, 1993], $V\{OB(C_6F_5)_3\}(acac)_2$ [1.527 (2) Å], $MoO\{OB(C_6F_5)_3\}(acac)_2$ [1.521 (3) Å], $Ti\{OB(C_6F_5)_3\}(acac)_2$ [1.496 (3) Å; Galsworthy *et al.*, 1997] and the aqua adduct $B(C_6F_5)_3(H_2O)$–dioxane [1.565 (3) Å; Janiak *et al.*, 1998].

Experimental

A solution of $[\{(^i\mathrm{Pr})_2\text{–ATI}\}\mathrm{In(Me)(Nme_2Ph)}][\mathrm{MeB(C_6F_5)_3}]$ in C_6D_5Cl was exposed to air. Hexane was added and the mixture was maintained at 296 K whereupon yellow crystals of (I) formed.

Crystal data

$[In(CH_3)(C_{18}HBF_{15}O)(C_{13}H_{19}N_2)]$
M_r = 862.15
Monoclinic, $P2_1/c$
a = 19.6683 (16) Å
b = 18.7739 (16) Å
c = 19.8049 (17) Å
β = 117.201 (1)°
V = 6504.2 (9) Å^3
Z = 8

D_x = 1.761 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6872 reflections
θ = 2–27°
μ = 0.844 mm^{-1}
T = 183 (2) K
Block, yellow
0.43 × 0.40 × 0.36 mm

Data collection

Bruker CCD-1000 area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Blessing, 1995)
$T_{\min} = 0.713$, $T_{\max} = 0.751$
41 003 measured reflections
13 220 independent reflections

9101 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.029$
$\theta_{\max} = 26.37°$
$h = -24 \rightarrow 21$
$k = 0 \rightarrow 23$
$l = 0 \rightarrow 24$
Intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.075$
$S = 0.966$
13220 reflections
947 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0350P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.68$ e Å^{-3}
$\Delta\rho_{\min} = -0.68$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

In1—N1	2.099 (2)	In2—N1*A*	2.101 (2)
In1—N2	2.103 (2)	In2—C1*A*	2.113 (3)
In1—C1	2.113 (3)	In2—O2	2.1811 (17)
In1—O1	2.1938 (17)	B1—O1	1.528 (3)
In2—N2*A*	2.098 (2)	B2—O2	1.531 (3)
N1—In1—N2	77.96 (8)	N2*A*—In2—C1*A*	129.74 (11)
N1—In1—C1	129.58 (11)	N1*A*—In2—C1*A*	129.81 (11)
N2—In1—C1	131.59 (12)	N2*A*—In2—O2	106.02 (8)
N1—In1—O1	106.05 (8)	N1*A*—In2—O2	103.15 (8)
N2—In1—O1	101.37 (8)	C1*A*—In2—O2	105.68 (10)
C1—In1—O1	105.45 (10)	B1—O1—In1	140.47 (16)
N2*A*—In2—N1*A*	77.99 (9)	B2—O2—In2	139.92 (16)

The disordered C12, C13, C14, C12′, C13′ and C14′ atoms were refined isotropically. Some restraints were used to ensure the disordered isopropyl groups have idealized geometries.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Al-Juaid, S. S., Buttrus, N. H., Eaborn, C., Hitchcock, P. B., Roberts, A. T. L., Smith, J. D. & Sullivan, A. C. (1986). *J. Chem Soc. Chem. Commun.* pp. 908–909.

Atwood, D. A. (1998). *Coord. Chem. Rev.* **176**, 407–430.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Bochmann, M. & Dawson, D. M. (1996). *Angew. Chem. Int. Ed. Engl.* **35**, 2226–2228.

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Coles, M. P. & Jordan, R. F. (1997). *J. Am. Chem. Soc.* **119**, 8125–8126.

Dagorne, S, Guzei, I. A., Coles, M. P. & Jordan, R. F. (2000). *J. Am. Chem. Soc.* **122**, 274–289.

Delpech, F., Guzei, I. A. & Jordan, R. F. (2000). In preparation.

Galsworthy, J. R., Green, M. L. H., Muller, M. & Prout, K. (1997). *J. Chem. Soc. Dalton Trans.* pp. 1309–1313.

Ihara, E., Young, V. G. Jr. & Jordan, R. F. (1998). *J. Am. Chem. Soc.* **120**, 8277–8278.

Janiak, C., Braun, L., Scharmann, T. G. & Girgsdies, F. (1998). *Acta Cryst.* C**54**, 1722–1724.

Korolev, A. V., Guzei, I. A. & Jordan, R. F. (1999). *J. Am. Chem. Soc.* **121**, 11606–11607.

Radzewich, C. E., Guzei, I. A. & Jordan, R. F. (1999). *J. Am. Chem. Soc.* **121**, 8673–8674.

Schaefer, W. P., Quan, R. W. & Bercaw, J. E. (1993). *Acta Cryst.* C**49**, 878–881.

Sheldrick, G. M. (1997). *SHELXTL Reference Manual.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

e328 Ilia A. Guzei *et al.* • [In(CH$_3$)(C$_{18}$HBF$_{15}$O)(C$_{13}$H$_{19}$N$_2$)] *Acta Cryst.* (2000). C**56**, e327–e328