Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

A supramolecular assembly: aquatris(pentafluorophenyl)borane as its mixed dimethyl sulfone and water solvate, $(H_2O)B(C_6F_5)_3{\cdot}Me_2SO_2{\cdot}H_2O$

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Received 20 June 2003
Accepted 24 June 2003
Online 23 August 2003

Key indicators

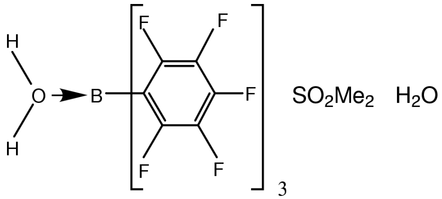
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.095
Data-to-parameter ratio = 13.6

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

The title compound, $C_{18}H_2BF_{15}O{\cdot}H_2O{\cdot}C_2H_6O_2$, obtained by crystallization of a product formed from a reaction mixture containing $B(C_6F_5)_3$ and Me_2SO_2 (and H_2O) in hexane, was characterized in the solid state as a supramolecular assembly containing water adducts of tris(pentafluorophenyl)borane, $(H_2O)B(C_6F_5)_3$, linked together by a network of hydrogen bonds involving one additional H_2O and one additional Me_2SO_2 molecule per adduct molecule.

Comment

There has been much recent interest in the chemistry of $B(C_6F_5)$ owing to its Lewis acidic properties and its potential as a catalyst for organic transformations (Piers & Chivers, 1997). We have recently explored the chemistry of phosphoryl donors (Beckett *et al.*, 2000, 2001) towards $B(C_6F_5)_3$ and have now turned our attention to sulfuryl donors. In an attempt to obtain the Me_2SO_2 adduct of $B(C_6F_5)$, we inadvertently prepared the title compound $(H_2O)B(C_6F_5)_3{\cdot}Me_2SO_2{\cdot}H_2O$, (I), as a consequence of adventitious water in our reaction system. Compound (I) was characterized by melting point, elemental analysis, IR and NMR (^{1}H and ^{11}B) spectroscopy, and in the solid state by a single-crystal X-ray diffraction study.



(I)

The structure of (I) may be conveniently described as a water adduct of $B(C_6F_5)_3$, *i.e.* $(H_2O)B(C_6F_5)_3$, linked to other adducts by a network of hydrogen bonds involving one additional H_2O and one Me_2SO_2 molecule per adduct molecule. Previous crystallographic studies on structures which contain the $B(C_6F_5)_3$ motif include $[(C_6F_5)_3B(OH)B(C_6F_5)_3]^-$ (Danopoulos *et al.*, 1998), $[WO\{OBC_6F_5)_3\}_3]^{2-}$ (Barrada *et al.*, 1999), $[Zr(Cp^*)_2\{OB(C_6F_5)_3\}]$ (Siedle *et al.*, 1993), and the adducts $B(C_6F_5)_3{\cdot}L$, with L = Et_3PO (Beckett *et al.*, 2000), Ph_3PO (Beckett *et al.*, 2001), PPh_3 (Jacobson *et al.*, 1999), RCN (R = Me, 4-MeC_6H_4, 4-$NO_2C_6H_4$) (Jacobson *et al.*, 1999), RNC (R = ^{t}Bu, $CMe_2CH_2{}^t$Bu) (Jacobson *et al.*, 1999), H_2O (Doerrer & Green, 1999; Danopoulos *et al.*, 1998), PhC(X)O (X = OEt, N^{i}Pr$_2$, H, Me) (Parks *et al.*, 1998), PH_3 (Bradley *et al.*, 1991), and P^{t}BuH$_2$ (Bradley *et al.*, 1996). The structure of the $B(C_6F_5)$ moiety in (I) is that of a three-bladed propeller and is geometrically comparable with the structures outlined above.

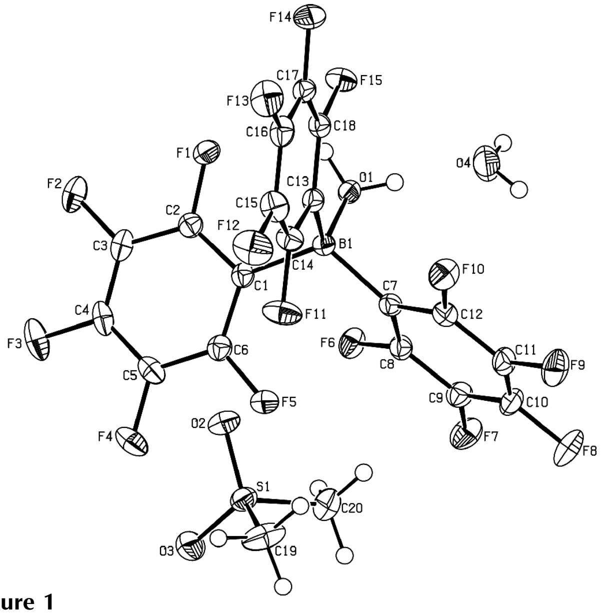


Figure 1
The asymmetric unit, with the atom numbering scheme and ??% probability displacement ellipsoids.

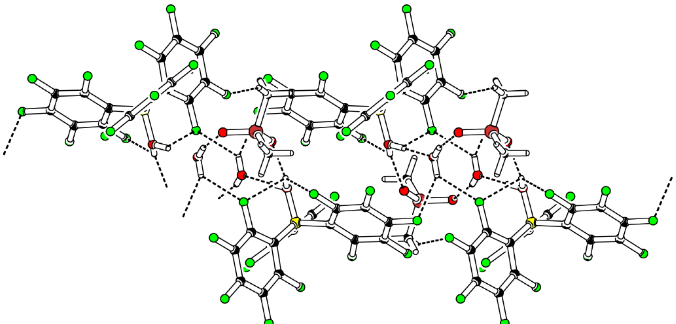


Figure 2
View showing the inter- and intramolecular interactions forming a supramolecular assembly.

A view of (I), showing the atomic numbering scheme, is shown in Fig. 1. Bond lengths and angles (Table 1) for the $(H_2O)B(C_6F_5)_3$ component of (I) are not significantly different from those found within this moiety in the structures of $(H_2O)B(C_6F_5)_3$ (Doerrer & Green, 1999) and $(H_2O)B(C_6F_5)_3{\cdot}2H_2O$ (Danopoulos *et al.*, 1998). These earlier structures which contain the $(H_2O)B(C_6F_5)_3$ fragment display hydrogen-bond interactions. Hydrogen-bond interactions and weakly interacting short contacts are apparent in (I) (Table 2) and these are responsible for its supramolecular architecture (Fig. 2). Two dimethyl sulfone and two water molecules associate as a tetramer, which binds two aquatris(pentafluorophenyl)borane moieties through the water ligand and fluoride groups.

Experimental

To a stirred solution of $B(B_6F_5)_3$ (0.25 g, 0.48 mmol) in hexane was added Me_2SO_2 (0.12 g, 0.48 mmol). The product, $(C_6F_5)_3B(H_2O){\cdot}Me_2SO_2{\cdot}H_2O$, precipitated out of the reaction mixture as a white solid (0.21; 67%). M.p. 336 K. NMR (δ/p.p.m.; $CDCl_3$/RT): ^{1}H (250 MHz): 2.98 (*s*, 6H), 5.97 (*br*, *s*, 4H); ^{11}B 80.2 MHz: −0.6; IR (KBr disc, ν_{max}/cm^{-1}): 3,500 (*br*), 3020 (*w*) 2940 (*w*), 1648 (*s*), 1519 (*s*) 1460 (*s*), 1279 (*s*), 1144 (*s*), 1098 (*s*), 977 (*s*), 935 (*m*), 764 (*m*), 680 (*m*). Elemental analysis required for $C_{20}H_{10}BF_{15}SO_4$: C 37.4, H 1.6%; found: C 37.7, H 1.0%. A single crystal suitable for X-ray diffraction was grown by diffusion of hexane into a $CHCl_3$ solution of the adduct.

Crystal data

$C_{18}H_2BF_{15}O{\cdot}H_2O{\cdot}C_2H_6O_2S$
M_r = 642.15
Triclinic, $P\overline{1}$
a = 10.357 (2) Å
b = 10.397 (2) Å
c = 11.671 (2) Å
α = 77.76 (3)°
β = 71.14 (3)°
γ = 83.95 (3)°
V = 1161.4 (4) Å^3
Z = 2
D_x = 1.836 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 28129 reflections
θ = 1–27.5°
μ = 0.29 mm^{-1}
T = 150 (2) K
Block, colourless
0.5 × 0.3 × 0.1 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.796, T_{max} = 0.924
21780 measured reflections
5282 independent reflections
4488 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 27.5°
h = −13 → 13
k = −13 → 13
l = −14 → 15

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.03
5282 reflections
389 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.3645P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.007
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.053 (3)

Table 1
Selected geometric parameters (Å, °).

C19—S1	1.7545 (18)	O3—S1	1.4447 (12)
C20—S1	1.7550 (19)	C1—B1	1.640 (2)
O1—B1	1.5651 (19)	C7—B1	1.631 (2)
O2—S1	1.4449 (12)	C13—B1	1.638 (2)
O1—B1—C7	100.50 (11)	O3—S1—O2	116.69 (7)
O1—B1—C13	111.53 (11)	O3—S1—C19	108.63 (9)
C7—B1—C13	115.45 (11)	O2—S1—C19	108.23 (8)
O1—B1—C1	105.60 (11)	O3—S1—C20	108.27 (9)
C7—B1—C1	115.70 (12)	O2—S1—C20	109.15 (8)
C13—B1—C1	107.52 (11)	C19—S1—C20	105.27 (10)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O4	0.85 (2)	1.69 (2)	2.5391 (18)	179 (2)
O1—H1*A*···F1	0.87 (3)	2.19 (2)	2.7046 (16)	117.0 (19)
O4—H4*B*···O3^{i}	0.83 (3)	1.99 (3)	2.807 (2)	169 (3)
O4—H4*B*···S1^{i}	0.83 (3)	2.94 (3)	3.6522 (17)	146 (3)
O1—H1*A*···O2^{ii}	0.87 (3)	1.95 (2)	2.7291 (17)	148 (2)
O4—H4*A*···F3^{iii}	0.80 (5)	2.24 (5)	2.950 (2)	147 (5)
O4—H4*A*···F15^{iv}	0.80 (5)	2.50 (5)	3.089 (2)	131 (5)

Symmetry codes: (i) 1 − *x*, 2 − *y*, 1 − *z*; (ii) *x*, *y*, *z* − 1; (iii) 1 − *x*, 1 − *y*, −*z*; (iv) 1 − *x*, 2 − *y*, −*z*.

Acta Cryst. (2003). E**59**, o1354–o1356 Simon J. Coles *et al.* • $C_{18}H_2BF_{15}O{\cdot}H_2O{\cdot}C_2H_6O_2S$ **o1355**

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002); software used to prepare material for publication: *SHELXL*97.

The authors thank the EPSRC for funding crystallographic facilities.

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o1356 Simon J. Coles *et al.* • $C_{18}H_2BF_{15}O \cdot H_2O \cdot C_2H_6O_2S$ *Acta Cryst.* (2003). E**59**, o1354–o1356