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Tetrahydrothiophene tris(pentafluorophenyl)borane

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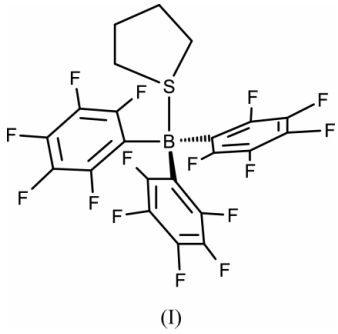
Key indicators

Single-crystal X-ray study
T = 230 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.083
Data-to-parameter ratio = 11.4

In the title compound, $(C_4H_8S)B(C_6F_5)_3$, a comparatively long boron–sulfur distance of 2.0843 (16) Å is found, most probably due to the sterically demanding pentafluorophenyl-substituents. Bond distances and angles of the boron–aryl fragment are comparable to those found for uncoordinated $MeB(C_6F_5)_3^-$ anions.

Comment

As a part of our investigations on the displacement of methylborate anions from zirconocene contact ion pairs, we have investigated the coordination of Lewis bases to tris-(pentafluorophenyl)borane for comparison. Single crystals of a Lewis base–Lewis acid adduct of tetrahydrothiophene and tris(pentafluorophenyl)borane were obtained by slow evaporation of a toluene solution containing the Lewis base and the Lewis acid in a ratio of approximately 1:1.



(I)

Bond distances and angles of the boron–aryl fragment [$d_{B,C}$ = 1.624 (2)–1.628 (2) Å, $\alpha_{C,B,S}$ = 101.67 (9)–106.45 (10)°] are comparable to those found for uncoordinated methylborate anions $MeB(C_6F_5)_3^-$ [$d_{B,C}$ = 1.60–1.73 Å, $\alpha_{C,B,Me}$ = 101–116° (CSD; Allen & Kennard, 1993): BIQREY, BIQREY, CACVEH, CACVOR, CACWAE, CACXIN, CEWFAL, CEWFOZ, FEQDAG, GEGYUM, GEHFAA, GUCXAD, HIMJOC, HIWQIN, HOHYUY, KICYEA, NUWWIL, NUWWOR, POQVEW, POWTUQ, POWVAY, POZWIK, QAPGET, QAPGIX, QAPGOD, QAWZIX, QEMWUA, REZBIH, RIDYOS, RIDYUY, RIRLEJ, TOPBUV, WEMLAB, WEYHAJ, WILQAJ, WILQEN, WORJOC, WORJUI, WORKAP, YEKLEF, YUVPAG, ZAFQAY]. The boron–sulfur distance of 2.0843 (16) Å is the longest observed so far for the coordination of a thioether to a Lewis acidic boron centre [$d_{B,S}$ = 1.82–2.07 Å, 54 structures in the CSD (Allen & Kennard, 1993): BEGYIV, BUPBOD, CAFNUS, CHMSDB, CONBEM, DIMBEG10, DIMBOQ10, DONXIN, FEBROT, FIJNIV, FIJNOB, FIJPAP, GALZEY, GAMBUR,

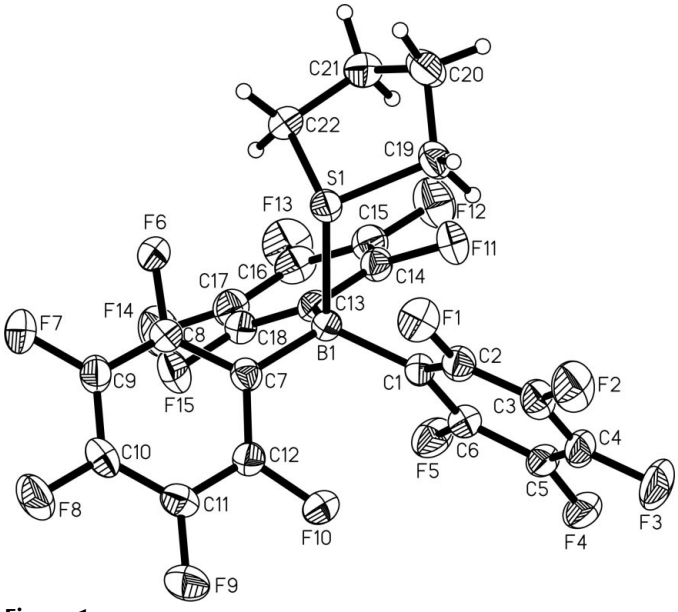


Figure 1
View of the title compound showing the labelling of non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

GAMGOQ, GAMHIL, GAMHUX, GICDIF, GICDOL, JESWIN, KOMZAN, MOMSHB, NAQPIE, NEDLAJ, NITWUI, NITXAP, NORNEN, NUHCOI, NUHCUO, NUHDAV, PIBXED, PIBXIH, PIJTUX, PUCNAC, PUCNEG, RUHBOR, SOGMAC, TAJBEL, TAJBIP, TOJXUL, TOJYAS, TOJYEW, VEMVIS, VUYHIG, WARGAX, WARGEB, WARGIF, WICHAR, WICHEV, YAKCOC, YEDHUK, ZEJCAS, ZEKQEL, ZONVAZ], most probably due to the sterically demanding pentafluorophenyl substituents.

Experimental

A toluene solution containing $B(C_6F_5)_3$ and C_4H_8S in an approximately 1:1 ratio was slowly evaporated under an argon atmosphere over three weeks to yield colourless crystals of the adduct complex (I).

Crystal data

$C_{22}H_8BF_{15}S$
M_r = 600.15
Triclinic, $P\overline{1}$
a = 9.5892 (8) Å
b = 10.8264 (10) Å
c = 11.3959 (9) Å
α = 111.904 (6)°
β = 96.814 (7)°
γ = 100.727 (7)°
V = 1055.29 (16) Å^3
Z = 2
D_x = 1.889 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 42 reflections
θ = 11.3–12.4°
μ = 0.30 mm^{-1}
T = 230 K
Prism, colourless
0.6 × 0.5 × 0.3 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
7694 measured reflections
4359 independent reflections
3938 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.0°
$h = -10 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$
3 standard reflections
every 97 reflections
intensity decay: 1.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.083
S = 1.05
4359 reflections
384 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0271P)^2 + 0.4601P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C19	1.8261 (15)	B1—C13	1.624 (2)
S1—C22	1.8286 (18)	B1—C7	1.628 (2)
S1—B1	2.0843 (16)	B1—C1	1.628 (2)
C13—B1—C7	115.42 (12)	C13—B1—S1	106.45 (10)
C13—B1—C1	113.86 (12)	C7—B1—S1	101.67 (9)
C7—B1—C1	112.10 (12)	C1—B1—S1	105.93 (9)

A relatively low data completeness (94.6%) was caused by the presence of a cooling device. The C—H bond lengths are 0.94 (2)–1.02 (2) Å.

Data collection: *XSCANS* (Siemens, 1992); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Siemens, 1991); software used to prepare material for publication: *SHELXL*97.

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