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Bis(pentafluorophenyl)boron Chloride

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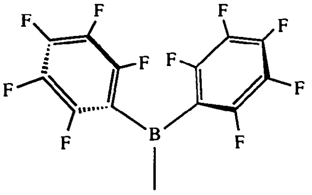
Abstract

The title compound, $(C_6F_5)_2BCl$, crystallizes upon slow sublimation at room temperature. It is a monomer in the solid state, which accounts for its volatility. The two perfluorinated phenyl rings are twisted by 35.5 (2) and 35.6 (2)° out of the plane containing Cl, B, C1 and C7, thereby alleviating steric interactions between *ortho* fluoro groups on opposing aryl rings.

Comment

Bis(pentafluorophenyl)boron chloride, (I), was first reported by Chambers & Chivers (1965) and the compound was synthesized using their methodology. The compound is a useful starting material for production of co-catalysts employed in Ziegler–Natta olefin-polymerization catalyst systems.



(I)

Compound (I) is monomeric in the solid state, accounting for its volatility. It is probably stable in the monomeric state because the three substituents on the boron are capable of π bonding with the unhybridized empty p orbital on the B atom. Although the planes of the perfluorinated phenyl rings are twisted out of the plane containing Cl, B, C1 and C7 by 35.5 (2) and 35.6 (2)°, short B—C1 and B—C7 bond lengths [compared with distances of 1.664–1.666 Å in complexes containing pyramidalized B-atom centres with C_6F_5 groups (Yang, Stern & Marks, 1994)] attest to the presence of π bonding between the B atom and the π system of the aryl rings. Steric repulsions between *ortho* fluoro groups F5 and F10 [F···F distance 2.956 (3) Å] on opposing aryl rings prevent the molecule from being completely planar.

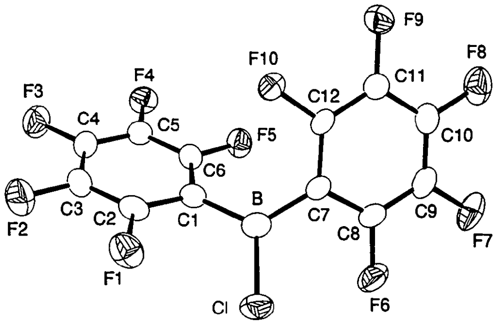


Fig. 1. A view of $(C_6F_5)_2BCl$, (I), with our numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Experimental

Ultrapure samples of (I) were obtained by double sublimation at room temperature and crystals were obtained *via* slow sublimation at room temperature under a static vacuum.

Crystal data

$C_{12}BClF_{10}$	Mo $K\alpha$ radiation
$M_r = 380.38$	$\lambda = 0.7107$ Å
Monoclinic	Cell parameters from 24 reflections
$P2_1/n$	$\theta = 17.00$–$20.50°$
$a = 14.454\ (2)$ Å	$\mu = 0.41\ \text{mm}^{-1}$
$b = 6.3050\ (6)$ Å	$T = 294\ (1)$ K
$c = 15.085\ (3)$ Å	Rectangular cube
$\beta = 112.03\ (2)°$	$0.70 \times 0.30 \times 0.20$ mm
$V = 1274.4\ (4)\ \text{Å}^3$	Colourless
$Z = 4$	
$D_x = 1.983\ \text{Mg m}^{-3}$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.013$
ω scans	$\theta_{\max} = 22.43°$
Absorption correction: ψ scans	$h = -15 \rightarrow 14$
$T_{\min} = 0.855$, $T_{\max} = 0.997$	$k = 0 \rightarrow 6$
1728 measured reflections	$l = 0 \rightarrow 16$
1656 independent reflections	3 standard reflections monitored every 100 reflections
1114 observed reflections $[I > 2.5\sigma(I)]$	intensity decay: 1.0%

Refinement

Refinement on F	$\Delta\rho_{\max} = 0.18\ \text{e Å}^{-3}$
$R = 0.035$	$\Delta\rho_{\min} = -0.27\ \text{e Å}^{-3}$
$wR = 0.033$	Extinction correction: none
$S = 2.11$	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)
1114 reflections	
217 parameters	
$w = 1/[\sigma^2(F) + 0.00004F^2]$	
$(\Delta/\sigma)_{\max} < 0.001$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl	0.36753 (10)	0.3953 (2)	0.08929 (8)	0.0858 (10)
F1	0.24531 (18)	0.3210 (4)	0.20646 (18)	0.0754 (17)
F2	0.22348 (17)	0.3670 (4)	0.37220 (17)	0.079 (2)
F3	0.38091 (18)	0.3157 (4)	0.53851 (17)	0.081 (2)
F4	0.56387 (17)	0.2257 (4)	0.53708 (16)	0.0662 (18)
F5	0.58901 (16)	0.1856 (4)	0.37246 (15)	0.0635 (17)
F6	0.55941 (19)	0.2997 (4)	0.07093 (17)	0.082 (2)
F7	0.69580 (18)	0.0316 (4)	0.05978 (17)	0.090 (2)
F8	0.72461 (18)	−0.3485 (5)	0.15166 (18)	0.085 (2)
F9	0.61288 (16)	−0.4556 (4)	0.25379 (17)	0.0677 (17)
F10	0.47725 (16)	−0.1879 (3)	0.26776 (15)	0.0592 (16)
C1	0.4186 (3)	0.2534 (6)	0.2802 (3)	0.047 (3)
C2	0.3275 (3)	0.2990 (6)	0.2870 (3)	0.053 (3)
C3	0.3136 (3)	0.3211 (6)	0.3714 (3)	0.054 (3)
C4	0.3930 (3)	0.2952 (7)	0.4553 (3)	0.055 (3)
C5	0.4847 (3)	0.2513 (7)	0.4544 (3)	0.050 (3)
C6	0.4959 (3)	0.2299 (6)	0.3683 (3)	0.049 (3)
C7	0.5109 (3)	0.0706 (7)	0.1701 (3)	0.047 (3)
C8	0.5695 (3)	0.1131 (8)	0.1171 (3)	0.055 (3)
C9	0.6407 (3)	−0.0220 (9)	0.1106 (3)	0.059 (3)
C10	0.6555 (3)	−0.2134 (8)	0.1566 (3)	0.058 (3)
C11	0.5991 (3)	−0.2671 (7)	0.2095 (3)	0.050 (3)
C12	0.5297 (3)	−0.1242 (7)	0.2151 (3)	0.047 (3)
B	0.4354 (4)	0.2293 (8)	0.1840 (3)	0.054 (4)

Table 2. *Selected geometric parameters* (Å, °)

Cl—B	1.746 (5)	C1—B	1.566 (6)
F1—C2	1.351 (5)	C2—C3	1.367 (6)
F2—C3	1.339 (5)	C3—C4	1.363 (6)
F3—C4	1.337 (5)	C4—C5	1.358 (6)
F4—C5	1.349 (5)	C5—C6	1.376 (6)
F5—C6	1.353 (5)	C7—C8	1.392 (6)
F6—C8	1.347 (5)	C7—C12	1.380 (6)
F7—C9	1.340 (5)	C7—B	1.551 (7)
F8—C10	1.336 (5)	C8—C9	1.367 (7)
F9—C11	1.341 (5)	C9—C10	1.368 (8)
F10—C12	1.348 (4)	C10—C11	1.379 (6)
C1—C2	1.390 (6)	C11—C12	1.375 (6)
C1—C6	1.386 (6)		
C2—C1—C6	113.4 (4)	C12—C7—B	121.7 (4)
C2—C1—B	124.7 (4)	F6—C8—C7	119.6 (4)
C6—C1—B	121.9 (4)	F6—C8—C9	116.5 (4)
F1—C2—C1	119.5 (4)	C7—C8—C9	123.9 (4)
F1—C2—C3	116.1 (4)	F7—C9—C8	120.6 (5)
C1—C2—C3	124.3 (4)	F7—C9—C10	120.0 (4)
F2—C3—C2	120.9 (4)	C8—C9—C10	119.5 (4)
F2—C3—C4	120.0 (4)	F8—C10—C9	120.8 (4)
C2—C3—C4	119.1 (4)	F8—C10—C11	119.5 (5)
F3—C4—C3	120.0 (4)	C9—C10—C11	119.7 (4)
F3—C4—C5	120.0 (4)	F9—C11—C10	119.8 (4)
C3—C4—C5	120.0 (4)	F9—C11—C12	121.6 (4)
F4—C5—C4	120.6 (4)	C10—C11—C12	118.6 (4)
F4—C5—C6	120.1 (4)	F10—C12—C7	120.3 (4)
C4—C5—C6	119.4 (4)	F10—C12—C11	115.2 (4)
F5—C6—C1	119.8 (4)	C7—C12—C11	124.5 (4)
F5—C6—C5	116.3 (4)	Cl—B—C1	117.7 (3)
C1—C6—C5	123.8 (4)	Cl—B—C7	119.0 (3)
C8—C7—C12	113.8 (4)	C1—B—C7	123.3 (4)
C8—C7—B	124.3 (4)		

The structure was solved by direct methods. All non-H atoms were located *via E* map inspection and were refined anisotropically.

Data collection: *Enraf–Nonius CAD-4 Software* (Enraf–Nonius, 1992). Cell refinement: *SET4* (Boer & Duisenberg, 1984); *CELDIM* (Enraf–Nonius, 1992). Data reduction: *NRCVAX94 DATRD2* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX94 SOLVER*. Program(s) used to refine structure: *NRCVAX94*. Molecular graphics: *NRCVAX94*; *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *NRCVAX94*.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BK1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Amino-8-methyl-2-quinolone Monohydrate

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Abstract

The quinolone ring system in $C_{10}H_{10}N_2O.H_2O$ is essentially planar. The hydrogen-bonding scheme involves O—H···O, N—H···O, N—H···N and C—H···O interactions.

Comment

Since the observation of laser action from organic compounds, many classes of dyes have been shown to

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