Crystal structure of 1,2-vinyl-bis(triphenylphosphonium) bis(boron tetrafluoride), $[\text{Ph}_3\text{P}-\text{CH}=\text{CH}-\text{PPh}_3][\text{BF}_4]_2$

R. Blachnik^{*†}, H. Reuter[†], G. Roth^{††}, T. Wiest[†] and H. Eickmeier[†]

[†] Universität Osnabrück, Institut für Chemie, Barbarastr. 7, D-49076 Osnabrück, Germany

^{††} Institut für Kristallographie, RWTH, Jägerstr. 17-19, D-52066 Aachen, Germany

Received December 1, 2000, CCDC-No. 1267/571

Abstract

$\text{C}_{38}\text{H}_{32}\text{B}_2\text{F}_8\text{P}_2$, triclinic, $P\bar{1}$ (No. 2), $a = 8.130(2)$ Å, $b = 10.473(2)$ Å, $c = 11.143(2)$ Å, $\alpha = 68.22(3)^\circ$, $\beta = 79.16(3)^\circ$, $\gamma = 87.87(3)^\circ$, $V = 864.8$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.073$, $wR_{\text{ref}}(F^2) = 0.220$, $T = 293$ K.

Source of material

AgBF_4 and $[\text{Ph}_3\text{P}-\text{HC}=\text{CH}-\text{Ph}_3\text{P}][\text{Br}_2]$ were reacted in dry ethanolic solution in a molar ratio of 2:1. Single crystals of $[\text{Ph}_3\text{P}-\text{HC}=\text{CH}-\text{PPh}_3][\text{BF}_4]_2$ appeared in the solution after filtration of AgBr and after slow evaporation of ethanol.

Experimental details

Although the anisotropic displacement parameters of the fluorine atoms in the BF_4 ion indicate that this ion is disordered, it was not possible to refine the structure model using split positions. Therefore only one common fluorine - boron distance was refined and the value of the bond angles restricted to 109.45° .

Table 1. Data collection and handling.

Crystal:	white block, size $0.10 \times 0.13 \times 0.25$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	1.97 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 185 exposures, $\Delta\phi = 1.8^\circ$
$2\theta_{\text{max}}$:	48°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	7712, 2544
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1893
$N(\text{param})_{\text{refined}}$:	228
Program:	SHELXS-97 [1]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	2i	0.5102	0.8860	0.4661	0.053(4)
H(11)	2i	0.9140	0.7822	0.7202	0.053
H(12)	2i	1.0940	0.6074	0.7105	0.053
H(13)	2i	1.0090	0.4359	0.6508	0.053
H(14)	2i	0.7525	0.4403	0.5943	0.053
H(15)	2i	0.5738	0.6173	0.5953	0.053
H(21)	2i	0.2753	0.9484	0.7578	0.053
H(22)	2i	0.0367	0.8558	0.9172	0.053
H(23)	2i	0.0210	0.6265	1.0488	0.053
H(24)	2i	0.2351	0.4856	1.0179	0.053
H(25)	2i	0.4721	0.5738	0.8602	0.053
H(31)	2i	0.5746	0.8893	0.9126	0.053
H(32)	2i	0.7325	1.0270	0.9746	0.053
H(33)	2i	0.9301	1.1829	0.8214	0.053
H(34)	2i	0.9842	1.1976	0.6055	0.053
H(35)	2i	0.8265	1.0655	0.5402	0.053

* Correspondence author (e-mail: bl@chnik.de)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
B(1)	2i	0.3593(5)	0.2916(3)	0.7577(4)	0.042(4)	0.052(4)	0.072(4)	0.006(3)	−0.021(3)	−0.014(3)
F(11)	2i	0.435(1)	0.2318(7)	0.6781(6)	0.45(2)	0.246(9)	0.205(8)	0.23(1)	−0.18(1)	−0.179(8)
F(12)	2i	0.2149(7)	0.2266(7)	0.8218(8)	0.122(8)	0.30(1)	0.38(2)	−0.102(8)	−0.045(9)	0.05(1)
F(13)	2i	0.3328(7)	0.4207(4)	0.6884(5)	0.138(5)	0.079(3)	0.169(5)	0.029(3)	−0.083(4)	−0.041(3)
F(14)	2i	0.4547(7)	0.2873(5)	0.8425(4)	0.145(6)	0.174(6)	0.098(4)	0.030(5)	−0.056(4)	−0.030(4)
C(1)	2i	0.5183(5)	0.9345(4)	0.5200(4)	0.031(3)	0.032(2)	0.025(2)	0.003(2)	−0.008(2)	−0.012(2)
P(1)	2i	0.5836(1)	0.8457(1)	0.67506(9)	0.0291(7)	0.0242(6)	0.0226(6)	0.0009(4)	−0.0067(4)	−0.0067(4)
C(10)	2i	0.7262(6)	0.7160(4)	0.6607(4)	0.033(3)	0.028(2)	0.025(2)	0.002(2)	−0.006(2)	−0.005(2)
C(11)	2i	0.8814(6)	0.7138(4)	0.6943(4)	0.036(3)	0.035(2)	0.031(2)	0.001(2)	−0.011(2)	−0.011(2)
C(12)	2i	0.9885(7)	0.6089(5)	0.6892(5)	0.033(3)	0.047(3)	0.043(2)	0.012(2)	−0.012(2)	−0.013(2)
C(13)	2i	0.9381(7)	0.5072(5)	0.6526(5)	0.049(4)	0.038(3)	0.050(3)	0.015(2)	−0.005(2)	−0.016(2)
C(14)	2i	0.7849(8)	0.5099(5)	0.6188(5)	0.059(4)	0.043(3)	0.065(3)	0.007(2)	−0.012(3)	−0.033(3)
C(15)	2i	0.6770(7)	0.6146(5)	0.6205(5)	0.044(3)	0.040(3)	0.053(3)	0.007(2)	−0.018(2)	−0.025(2)
C(20)	2i	0.3973(6)	0.7704(4)	0.7937(4)	0.030(3)	0.035(2)	0.026(2)	−0.003(2)	−0.009(2)	−0.008(2)
C(21)	2i	0.2675(6)	0.8552(5)	0.8102(4)	0.040(3)	0.041(3)	0.036(2)	0.000(2)	−0.005(2)	−0.014(2)
C(22)	2i	0.1250(7)	0.7997(6)	0.9061(5)	0.032(3)	0.066(3)	0.045(3)	0.001(2)	−0.004(2)	−0.028(3)
C(23)	2i	0.1151(7)	0.6629(6)	0.9837(5)	0.042(4)	0.077(4)	0.035(2)	−0.018(3)	0.001(2)	−0.015(3)
C(24)	2i	0.2439(8)	0.5788(6)	0.9657(5)	0.052(4)	0.050(3)	0.048(3)	−0.012(3)	−0.005(3)	0.005(2)
C(25)	2i	0.3854(7)	0.6311(5)	0.8716(5)	0.046(3)	0.039(3)	0.043(3)	−0.001(2)	−0.009(2)	−0.003(2)
C(30)	2i	0.6852(6)	0.9638(4)	0.7202(4)	0.032(3)	0.027(2)	0.030(2)	0.003(2)	−0.008(2)	−0.011(2)
C(31)	2i	0.6559(6)	0.9516(5)	0.8506(4)	0.036(3)	0.041(2)	0.035(2)	0.000(2)	−0.006(2)	−0.018(2)
C(32)	2i	0.7505(7)	1.0344(5)	0.8873(5)	0.056(4)	0.060(3)	0.043(3)	0.001(3)	−0.011(2)	−0.033(2)
C(33)	2i	0.8697(8)	1.1267(5)	0.7960(5)	0.060(4)	0.046(3)	0.062(3)	−0.006(3)	−0.020(3)	−0.031(3)
C(34)	2i	0.9006(7)	1.1369(5)	0.6664(5)	0.051(4)	0.040(3)	0.053(3)	−0.013(2)	−0.009(2)	−0.014(2)
C(35)	2i	0.8078(6)	1.0572(4)	0.6278(4)	0.045(3)	0.036(2)	0.032(2)	−0.003(2)	−0.006(2)	−0.012(2)

Reference

- Sheldrick, G. M.: Program Package SHELXTL, Release 5.1, Bruker Analytical X-Ray Instruments Inc, Madison (WI 53719), USA 1997.