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Crystal structure of (2,6-diisopropyl-phenyl)[*N*-tris(pentafluorophenyl)-boronyl-pyridin-2-yl]amine, $(C_6F_5)_3B(C_5H_4N)(C_{12}H_{18}N)$

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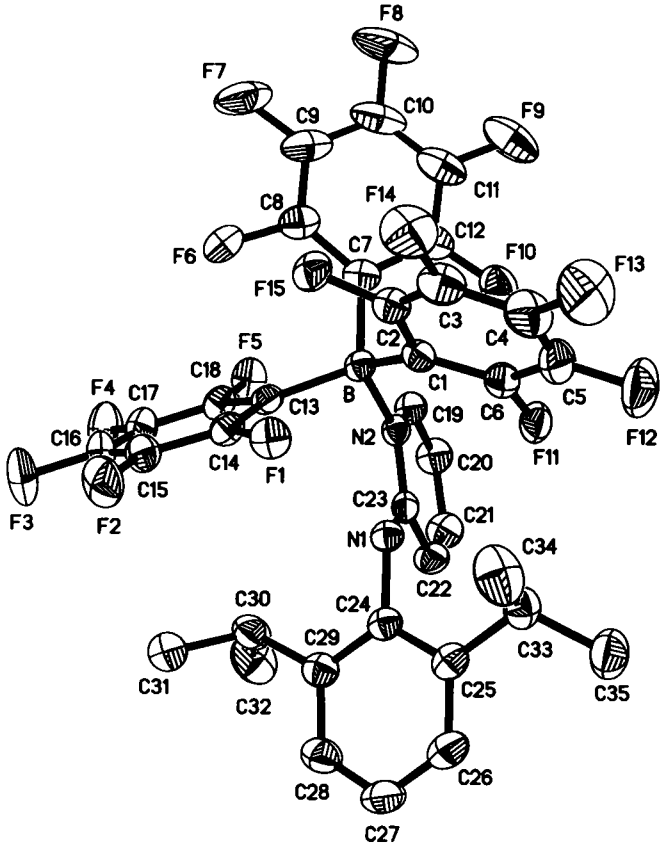

Abstract

$C_{35}H_{22}BF_{15}N_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.800(1) Å, b = 12.022(1) Å, c = 14.326(1) Å, α = 99.486(5)°, β = 102.673(5)°, γ = 94.591(5)°, V = 1612.3 Å^3, Z = 2, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.124, T = 193 K.

Source of material

To a stirred solution of 0.256 g (0.5 mmol) $B(C_6F_5)_3$ in 10 mL bromobenzene a solution of 0.366 g (0.5 mmol) $[(Ap)_2Ta(CH_3)_3]$ [1] (Ap = (2,6-diisopropyl-phenyl)-pyridin-2-yl-amine) in 10 mL bromobenzene was added at room temperature. A color change from yellow to orange was observed. The reaction mixture was stirred for half an hour. Volume was reduced to 3 mL and the reaction mixture was then stored at –25 °C overnight to afford colorless crystals of the title compound suitable for X-ray crystal structure analysis.

Experimental details

The H atom bonded to the N atom was refined freely due to its importance in the hydrogen bonding.

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Discussion

Recently, we described the synthesis and structure of trialkyl tantalum complexes stabilized by aminopyridinato ligands [1]. The reaction of $[(Ap)_2Ta(CH_3)_3]$ with $B(C_6F_5)_3$ leads to the title compound as a by-product. One $B(C_6F_5)_3$ unit coordinated to the pyridine nitrogen (d(B—N2) = 1.619 Å). The pyridine-N atom (N2) is three-coordinated with bond angles of ∠C23–N2–C19 = 118.6°, ∠C23–N2–B = 121.1° and ∠C19–N2–B = 120.1°. The sum of these angles is 359.8° and thus indicative of a planar coordination. The boron center shows a pseudo tetrahedral coordination environment. The distortion is caused by small C1–B–C7 (102.1°) and N2–B–C13 (102.7°) angles. The phenyl substituent at the amino-N atom is twisted to the pyridine ring, forming a dihedral angle of 103.6°. The three pentafluorophenyl substituents at the boron center are twisted with regard to the pyridine ring, and the dihedral angles are 109.8°, 62.0° and 87.8°, respectively.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.4 × 0.5 × 0.6 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	1.53 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{max}$:	51.52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21557, 6077
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4913
$N(param)_{refined}$:	482
Programs:	SIR97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(19)	2*i*	0.5462	0.3116	0.4798	0.038
H(20)	2*i*	0.7585	0.3067	0.4363	0.046
H(21)	2*i*	0.7604	0.2229	0.2763	0.048
H(22)	2*i*	0.5511	0.1494	0.1659	0.041
H(26)	2*i*	0.2410	−0.1118	−0.0777	0.049
H(27)	2*i*	0.2788	0.0221	−0.1719	0.053
H(28)	2*i*	0.3160	0.2135	−0.1039	0.050
H(30)	2*i*	0.3217	0.3355	0.1427	0.053
H(31A)	2*i*	0.2275	0.4645	0.0447	0.085
H(31B)	2*i*	0.1204	0.3506	0.0232	0.085
H(31C)	2*i*	0.2154	0.3714	−0.0515	0.085
H(32A)	2*i*	0.4836	0.4604	0.0968	0.097
H(32B)	2*i*	0.4827	0.3676	0.0027	0.097
H(32C)	2*i*	0.5467	0.3435	0.1092	0.097
H(33)	2*i*	0.2897	−0.0574	0.1796	0.046
H(34A)	2*i*	0.0658	−0.1635	0.1475	0.095
H(34B)	2*i*	0.0291	−0.1331	0.0408	0.095
H(34C)	2*i*	0.0531	−0.0347	0.1348	0.095
H(35A)	2*i*	0.2726	−0.2545	0.1227	0.085
H(35B)	2*i*	0.3958	−0.1886	0.0884	0.085
H(35C)	2*i*	0.2478	−0.2342	0.0129	0.085
H(100)	2*i*	0.227(3)	0.142(2)	0.212(2)	0.037(6)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*i*	0.1827(2)	0.1258(2)	0.3722(1)	0.028(1)	0.0339(9)	0.0266(8)	0.0040(7)	0.0062(7)	0.0083(7)
C(2)	2*i*	0.0461(2)	0.1216(2)	0.3857(1)	0.029(1)	0.038(1)	0.0316(9)	0.0029(8)	0.0071(7)	0.0072(8)
C(3)	2*i*	−0.0369(2)	0.0240(2)	0.3843(2)	0.033(1)	0.051(1)	0.047(1)	−0.0049(9)	0.0147(9)	0.0113(9)
C(4)	2*i*	0.0182(3)	−0.0777(2)	0.3728(2)	0.056(2)	0.040(1)	0.059(1)	−0.013(1)	0.020(1)	0.012(1)
C(5)	2*i*	0.1537(3)	−0.0790(2)	0.3636(2)	0.063(2)	0.030(1)	0.057(1)	0.007(1)	0.023(1)	0.0134(9)
C(6)	2*i*	0.2328(2)	0.0206(2)	0.3631(1)	0.037(1)	0.036(1)	0.038(1)	0.0073(8)	0.0143(8)	0.0107(8)
C(7)	2*i*	0.3016(2)	0.2986(2)	0.5027(1)	0.0242(9)	0.041(1)	0.0282(9)	−0.0021(8)	0.0053(7)	0.0042(8)
C(8)	2*i*	0.2416(2)	0.3875(2)	0.5460(1)	0.039(1)	0.041(1)	0.037(1)	−0.0013(9)	0.0128(8)	0.0019(8)
C(9)	2*i*	0.2511(3)	0.4153(2)	0.6443(2)	0.061(2)	0.046(1)	0.044(1)	−0.016(1)	0.029(1)	−0.008(1)
C(10)	2*i*	0.3234(3)	0.3525(2)	0.7060(2)	0.070(2)	0.064(2)	0.027(1)	−0.029(1)	0.015(1)	0.001(1)
C(11)	2*i*	0.3834(3)	0.2622(2)	0.6678(2)	0.047(1)	0.072(2)	0.033(1)	−0.013(1)	0.0025(9)	0.017(1)
C(12)	2*i*	0.3716(2)	0.2374(2)	0.5694(1)	0.032(1)	0.054(1)	0.034(1)	−0.0016(9)	0.0056(8)	0.0110(9)
C(13)	2*i*	0.2037(2)	0.3395(1)	0.3190(1)	0.0263(9)	0.0292(9)	0.0261(8)	0.0060(7)	0.0083(7)	0.0027(7)
C(14)	2*i*	0.0801(2)	0.3221(2)	0.2479(1)	0.0260(9)	0.0305(9)	0.0318(9)	0.0042(7)	0.0076(7)	0.0025(7)
C(15)	2*i*	0.0251(2)	0.4050(2)	0.1991(1)	0.033(1)	0.047(1)	0.034(1)	0.0145(9)	0.0014(8)	0.0079(8)
C(16)	2*i*	0.1008(2)	0.5107(2)	0.2174(2)	0.048(1)	0.037(1)	0.050(1)	0.0200(9)	0.009(1)	0.0166(9)
C(17)	2*i*	0.2283(2)	0.5316(2)	0.2836(2)	0.042(1)	0.0268(9)	0.053(1)	0.0075(8)	0.0120(9)	0.0080(8)
C(18)	2*i*	0.2759(2)	0.4477(2)	0.3324(1)	0.028(1)	0.0315(9)	0.0363(9)	0.0065(7)	0.0072(8)	0.0027(7)
C(19)	2*i*	0.5468(2)	0.2783(2)	0.4150(1)	0.026(1)	0.036(1)	0.0320(9)	0.0050(7)	0.0045(7)	0.0060(7)
C(20)	2*i*	0.6732(2)	0.2757(2)	0.3901(2)	0.021(1)	0.049(1)	0.043(1)	0.0042(8)	0.0031(8)	0.0071(9)
C(21)	2*i*	0.6738(2)	0.2266(2)	0.2955(2)	0.024(1)	0.054(1)	0.046(1)	0.0089(8)	0.0118(8)	0.0118(9)
C(22)	2*i*	0.5505(2)	0.1836(2)	0.2305(1)	0.029(1)	0.043(1)	0.0341(9)	0.0086(8)	0.0116(8)	0.0074(8)
C(23)	2*i*	0.4216(2)	0.1896(1)	0.2584(1)	0.0267(9)	0.0240(8)	0.0310(9)	0.0058(7)	0.0076(7)	0.0084(7)
C(24)	2*i*	0.2904(2)	0.1151(2)	0.0898(1)	0.0237(9)	0.0334(9)	0.0294(9)	0.0041(7)	0.0061(7)	0.0032(7)
C(25)	2*i*	0.2658(2)	−0.0013(2)	0.0499(1)	0.025(1)	0.035(1)	0.036(1)	0.0039(7)	0.0060(7)	0.0016(8)
C(26)	2*i*	0.2597(2)	−0.0335(2)	−0.0486(2)	0.041(1)	0.040(1)	0.038(1)	0.0066(9)	0.0081(9)	−0.0041(8)
C(27)	2*i*	0.2803(2)	0.0459(2)	−0.1051(1)	0.047(1)	0.055(1)	0.030(1)	0.010(1)	0.0115(9)	−0.0002(9)
C(28)	2*i*	0.3031(2)	0.1595(2)	−0.0642(1)	0.042(1)	0.051(1)	0.035(1)	0.0057(9)	0.0135(9)	0.0120(9)
C(29)	2*i*	0.3076(2)	0.1974(2)	0.0333(1)	0.032(1)	0.039(1)	0.0330(9)	0.0037(8)	0.0080(8)	0.0068(8)
C(30)	2*i*	0.3282(3)	0.3242(2)	0.0732(2)	0.065(2)	0.035(1)	0.033(1)	0.002(1)	0.012(1)	0.0098(8)
C(31)	2*i*	0.2125(3)	0.3830(2)	0.0174(2)	0.082(2)	0.050(1)	0.046(1)	0.023(1)	0.022(1)	0.014(1)
C(32)	2*i*	0.4734(3)	0.3788(2)	0.0702(2)	0.075(2)	0.046(1)	0.068(2)	−0.015(1)	0.008(1)	0.020(1)
C(33)	2*i*	0.2387(2)	−0.0893(2)	0.1107(2)	0.038(1)	0.032(1)	0.041(1)	0.0021(8)	0.0028(8)	0.0050(8)
C(34)	2*i*	0.0829(3)	−0.1067(2)	0.1082(2)	0.045(2)	0.065(2)	0.086(2)	−0.001(1)	0.017(1)	0.033(1)
C(35)	2*i*	0.2936(3)	−0.2017(2)	0.0811(2)	0.072(2)	0.038(1)	0.056(1)	0.012(1)	0.003(1)	0.006(1)
N(1)	2*i*	0.2989(2)	0.1507(1)	0.1924(1)	0.0230(8)	0.0300(8)	0.0284(7)	0.0026(6)	0.0080(6)	0.0050(6)
N(2)	2*i*	0.4215(2)	0.2358(1)	0.3517(1)	0.0221(8)	0.0271(7)	0.0288(7)	0.0050(6)	0.0056(6)	0.0067(6)
F(1)	2*i*	0.0038(1)	0.21800(9)	0.21913(8)	0.0289(6)	0.0380(6)	0.0380(6)	−0.0015(4)	−0.0001(4)	0.0043(5)
F(2)	2*i*	−0.0974(1)	0.3816(1)	0.13266(9)	0.0411(7)	0.0644(8)	0.0500(7)	0.0156(6)	−0.0106(6)	0.0131(6)
F(3)	2*i*	0.0518(2)	0.5913(1)	0.1694(1)	0.073(1)	0.0500(8)	0.083(1)	0.0259(7)	0.0017(8)	0.0335(7)
F(4)	2*i*	0.3065(2)	0.6325(1)	0.3004(1)	0.0597(9)	0.0285(6)	0.092(1)	0.0033(6)	0.0087(8)	0.0169(6)
F(5)	2*i*	0.3994(1)	0.47634(9)	0.39963(8)	0.0308(6)	0.0326(6)	0.0493(6)	−0.0014(4)	−0.0007(5)	0.0026(5)
F(6)	2*i*	0.1639(2)	0.4529(1)	0.49159(9)	0.0615(8)	0.0471(7)	0.0490(7)	0.0202(6)	0.0236(6)	0.0025(6)
F(7)	2*i*	0.1876(2)	0.5021(1)	0.6802(1)	0.116(1)	0.0560(9)	0.0637(9)	−0.0010(8)	0.0586(9)	−0.0110(7)
F(8)	2*i*	0.3309(2)	0.3772(2)	0.80146(9)	0.135(2)	0.086(1)	0.0301(7)	−0.034(1)	0.0285(8)	−0.0011(7)
F(9)	2*i*	0.4510(2)	0.1978(2)	0.7270(1)	0.081(1)	0.105(1)	0.0422(7)	−0.0031(9)	−0.0023(7)	0.0367(8)
F(10)	2*i*	0.4312(1)	0.1462(1)	0.53679(9)	0.0473(8)	0.0639(8)	0.0465(7)	0.0198(6)	0.0078(6)	0.0241(6)
F(11)	2*i*	0.3658(1)	0.01109(9)	0.35488(9)	0.0416(7)	0.0386(6)	0.0560(7)	0.0172(5)	0.0208(6)	0.0182(5)
F(12)	2*i*	0.2090(2)	−0.1775(1)	0.3546(1)	0.094(1)	0.0306(7)	0.110(1)	0.0161(7)	0.047(1)	0.0217(7)
F(13)	2*i*	−0.0601(2)	−0.1748(1)	0.3722(1)	0.082(1)	0.0471(8)	0.113(1)	−0.0214(8)	0.039(1)	0.0189(8)
F(14)	2*i*	−0.1692(1)	0.0262(1)	0.3953(1)	0.0338(7)	0.0726(9)	0.0765(9)	−0.0067(6)	0.0218(6)	0.0214(7)
F(15)	2*i*	−0.0094(1)	0.2201(1)	0.40364(8)	0.0270(6)	0.0448(6)	0.0475(6)	0.0095(5)	0.0137(5)	0.0095(5)
B	2*i*	0.2753(2)	0.2498(2)	0.3851(1)	0.021(1)	0.031(1)	0.0272(9)	0.0056(8)	0.0047(7)	0.0039(8)

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