Crystal structure of bis(pentamethylcyclopentadienyl)tri(pyridine)barium, $Ba(C_5H_5N)_3(C_{10}H_{15})_2$

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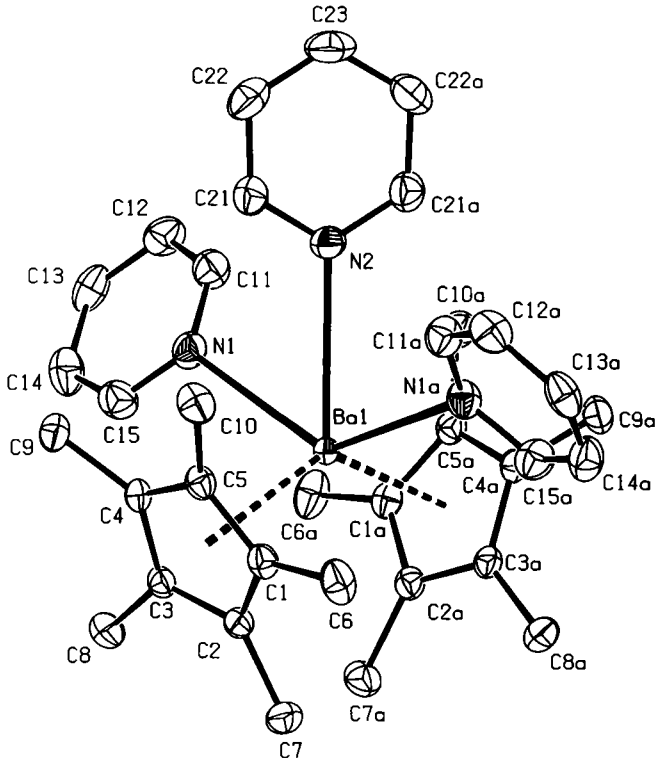

Abstract

$C_{35}H_{45}BaN_3$, orthorhombic, *Pbcn* (no. 60),
$a = 15.7141(5)$ Å, $b = 12.8573(4)$ Å, $c = 15.7388(5)$ Å,
$V = 3179.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.020$, $wR_{ref}(F^2) = 0.058$,
$T = 153$ K.

Source of material

The title compound was prepared analogously to the similar compounds [1,2]. All manipulations were performed using conventional Schlenk techniques under an atmosphere of purified argon. Solvents were dried, distilled, and degassed before use. 1.46 g (2.42 mmol) barium bis(hexamethyldisilazide) was dissolved in 25 ml toluene. To this solution 0.66 g (4.85 mmol) pentamethylcyclopentadiene in 4 ml toluene was added. The white precipitate formed was collected, washed with *n*-hexane and redissolved in a small amount of pyridine. This solution was overlayered with *n*-hexane and cooled down to –30 °C for three days to give small yellow blocks of $[BaCp^*_2py_3]$ (yield 15–20 %).

Discussion

The title compound forms molecules with five ligands (two C_5Me_5 anions and three pyridine units) coordinated to the central barium cation, which is located on the twofold rotation axis of the space group *Pbcn*. The Ba—N bond lengths are 297.5(2) pm and 300.0(1) pm, the Ba—C distances are between 304.1(1) pm and 305.1(1) pm. The C—C bond lengths within the five-membered ring are 141.6(2) pm.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.24 × 0.34 × 0.36 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.75 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART CCD, ω
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	75507, 4644
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3592
$N(param)_{refined}$:	183
Programs:	SHELXS-97 [3], SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(6A)	8*d*	0.6339	0.3591	0.0722	0.063
H(6B)	8*d*	0.5912	0.3731	–0.0194	0.063
H(6C)	8*d*	0.6141	0.2593	0.0147	0.063
H(7A)	8*d*	0.5570	0.4976	0.1692	0.056
H(7B)	8*d*	0.4640	0.5482	0.1672	0.056
H(7C)	8*d*	0.5185	0.5400	0.0816	0.056
H(8A)	8*d*	0.2602	0.4036	0.1692	0.052
H(8B)	8*d*	0.3199	0.5036	0.1588	0.052
H(8C)	8*d*	0.3259	0.4339	0.2429	0.052
H(9A)	8*d*	0.2481	0.2379	0.1377	0.051
H(9B)	8*d*	0.3033	0.1335	0.1320	0.051
H(9C)	8*d*	0.2734	0.1912	0.0469	0.051
H(10A)	8*d*	0.4418	0.0788	0.0469	0.057
H(10B)	8*d*	0.5230	0.1296	0.0016	0.057
H(10C)	8*d*	0.4305	0.1424	–0.0398	0.057
H(11)	8*d*	0.3875	0.0228	0.3811	0.046
H(12)	8*d*	0.2767	–0.0111	0.4738	0.050
H(13)	8*d*	0.1574	0.0978	0.4747	0.049
H(14)	8*d*	0.1538	0.2370	0.3789	0.052
H(15)	8*d*	0.2687	0.2657	0.2906	0.048
H(21)	8*d*	0.3954	–0.0163	0.1828	0.047
H(22)	8*d*	0.3927	–0.1963	0.1799	0.058
H(23)	4*c*	½	–0.2893	¼	0.063

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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}
Ba(1)	4c	½	0.232078(8)	¼	0.01726(6)	0.01932(7)	0.02109(7)	0	–0.00074(4)	0
C(1)	8d	0.50704(7)	0.3203(1)	0.07085(9)	0.0247(7)	0.0341(9)	0.0236(6)	0.0013(5)	0.0005(5)	0.0063(6)
C(2)	8d	0.46688(9)	0.4002(1)	0.11809(8)	0.0255(6)	0.0264(7)	0.0269(6)	–0.0015(6)	–0.0042(5)	0.0058(5)
C(3)	8d	0.38395(7)	0.3662(1)	0.13976(7)	0.0230(6)	0.0265(6)	0.0240(6)	0.0020(5)	–0.0037(5)	0.0024(5)
C(4)	8d	0.37295(8)	0.26486(9)	0.10622(8)	0.0213(6)	0.0286(7)	0.0234(6)	0.0004(5)	–0.0050(5)	0.0000(5)
C(5)	8d	0.44892(9)	0.2366(1)	0.06368(8)	0.0268(7)	0.0308(7)	0.0232(7)	0.0036(5)	–0.0035(5)	–0.0016(5)
C(6)	8d	0.59420(9)	0.3287(1)	0.03111(9)	0.0283(7)	0.060(1)	0.0378(8)	0.0036(7)	0.0050(6)	0.0167(7)
C(7)	8d	0.50493(9)	0.5058(1)	0.1356(1)	0.0409(8)	0.0292(8)	0.0415(9)	–0.0077(6)	–0.0077(6)	0.0077(7)
C(8)	8d	0.31660(8)	0.4327(1)	0.18131(9)	0.0311(7)	0.0319(7)	0.0415(8)	0.0065(6)	0.0001(6)	0.0002(6)
C(9)	8d	0.29242(8)	0.2013(1)	0.10566(9)	0.0258(7)	0.0391(8)	0.0363(8)	–0.0034(6)	–0.0061(6)	–0.0029(7)
C(10)	8d	0.4622(1)	0.1383(1)	0.01377(9)	0.0412(8)	0.0406(8)	0.0317(7)	0.0091(7)	–0.0023(6)	–0.0087(6)
N(1)	8d	0.33941(7)	0.14734(9)	0.32642(7)	0.0291(6)	0.0380(7)	0.0345(6)	–0.0043(5)	0.0021(5)	0.0048(5)
C(11)	8d	0.33944(9)	0.0677(1)	0.38065(9)	0.0333(8)	0.0423(9)	0.0403(8)	–0.0009(6)	0.0030(6)	0.0064(7)
C(12)	8d	0.2735(1)	0.0467(1)	0.43634(9)	0.0459(9)	0.0434(8)	0.0367(8)	–0.0110(7)	0.0054(7)	0.0086(7)
C(13)	8d	0.20341(9)	0.1102(1)	0.43685(9)	0.0334(8)	0.058(1)	0.0309(8)	–0.0147(7)	0.0053(6)	–0.0087(7)
C(14)	8d	0.20169(9)	0.1919(1)	0.3812(1)	0.0314(8)	0.058(1)	0.0415(9)	0.0070(7)	–0.0014(7)	–0.0100(8)
C(15)	8d	0.2706(1)	0.2079(1)	0.3282(1)	0.0425(9)	0.0407(8)	0.0379(8)	0.0017(7)	–0.0008(7)	0.0066(7)
N(2)	4c	½	0.0007(2)	¼	0.0287(9)	0.0268(9)	0.053(1)	0	0.0030(7)	0
C(21)	8d	0.43939(9)	–0.0535(1)	0.2109(1)	0.0305(7)	0.0412(9)	0.0468(9)	–0.0013(6)	–0.0012(7)	0.0047(7)
C(22)	8d	0.4370(1)	–0.1608(1)	0.2090(1)	0.0441(9)	0.043(1)	0.057(1)	–0.0124(8)	0.0013(8)	–0.0127(8)
C(23)	4c	½	–0.2154(2)	¼	0.061(2)	0.025(1)	0.073(2)	0	0.012(1)	0

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