

Crystal structure of tetramethylammonium triphenyl-(9-triptycyl)borate, $C_4H_{12}N^+C_{38}H_{28}B^-$

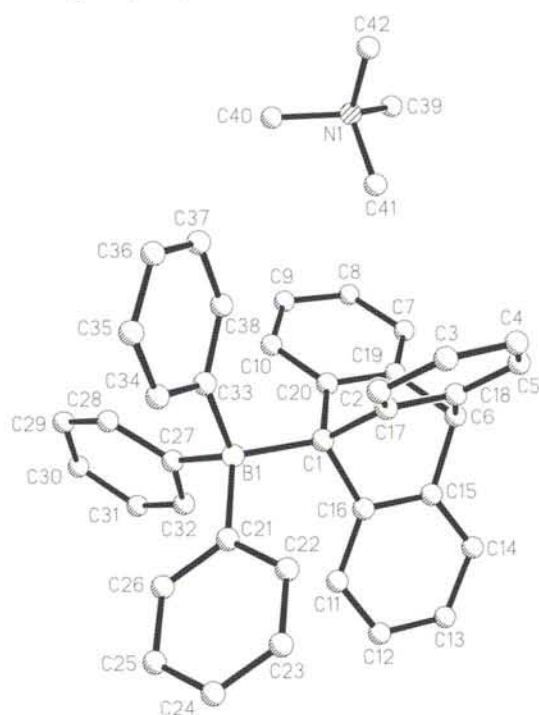
K. Peters, E.-M. Peters

Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, D-70506 Stuttgart, Germany

T. Panitzsch and W. Tochtermann

Christian-Albrechts-Universität zu Kiel, Institut für Organische Chemie, Olshausenstraße 40, D-24098 Kiel, Germany

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Abstract

$C_{42}H_{40}BN$, monoclinic, $C1c1$ (No. 9), $a = 17.171(2)$ Å, $b = 9.658(1)$ Å, $c = 19.742(2)$ Å, $\beta = 104.63(1)^\circ$, $V = 3167.8$ Å³, $Z = 4$, $R_g(F) = 0.064$, $R_w(F) = 0.059$, $T = 293$ K.

Source of material

The title compound was prepared as described in [1].

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.65 x 0.65 x 0.55 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	0.70 cm ⁻¹
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3989, 3760
Criterion for F_{obs} , $N(hkl)_{gt}$:	$F_{obs} > 3 \sigma(F_{obs})$, 3180
$N(param)_{refined}$:	395
Program:	SHELXTL-plus [2]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4a	0.5566(4)	0.3654(4)	0.8958(3)	0.08
H(3)	4a	0.6038(4)	0.5694(4)	0.9571(4)	0.08
H(4)	4a	0.6345(4)	0.5781(4)	1.0784(3)	0.08
H(5)	4a	0.6090(4)	0.3819(4)	1.1412(3)	0.08
H(6)	4a	0.5590(4)	0.1389(4)	1.1402(3)	0.08
H(7)	4a	0.6595(4)	-0.0552(5)	1.1650(3)	0.08
H(8)	4a	0.7382(4)	-0.2171(5)	1.1230(4)	0.08
H(9)	4a	0.7217(4)	-0.2404(5)	1.0027(4)	0.08
H(10)	4a	0.6288(4)	-0.1052(4)	0.9238(3)	0.08
H(11)	4a	0.3579(4)	0.0365(4)	0.8950(3)	0.08
H(12)	4a	0.2582(4)	0.0013(5)	0.9557(4)	0.08
H(13)	4a	0.2849(4)	0.0296(5)	1.0751(4)	0.08
H(14)	4a	0.4164(4)	0.0922(5)	1.1397(4)	0.08
H(22)	4a	0.4218(4)	0.3334(5)	0.8787(4)	0.08
H(23)	4a	0.3016(4)	0.4295(5)	0.8163(4)	0.08
H(24)	4a	0.2215(4)	0.3153(6)	0.7186(4)	0.08
H(25)	4a	0.2670(4)	0.1057(6)	0.6838(4)	0.08
H(26)	4a	0.3901(4)	0.0126(5)	0.7432(3)	0.08
H(28)	4a	0.5799(5)	-0.0653(5)	0.7743(4)	0.08
H(29)	4a	0.5864(5)	-0.2993(6)	0.7494(4)	0.08
H(30)	4a	0.5003(5)	-0.4580(5)	0.7842(4)	0.08
H(31)	4a	0.4222(5)	-0.3808(5)	0.8573(4)	0.08
H(32)	4a	0.4243(4)	-0.1476(5)	0.8896(4)	0.08
H(34)	4a	0.5090(4)	0.2361(5)	0.7414(3)	0.08
H(35)	4a	0.6077(5)	0.3516(6)	0.7017(4)	0.08
H(36)	4a	0.7410(5)	0.3490(6)	0.7658(4)	0.08
H(37)	4a	0.7746(4)	0.2276(6)	0.8709(4)	0.08
H(38)	4a	0.6771(4)	0.1126(5)	0.9111(4)	0.08
H(39A)	4a	0.8656(5)	-0.0402(8)	0.1322(4)	0.08
H(39B)	4a	0.9004(5)	0.0991(8)	0.1682(4)	0.08
H(39C)	4a	0.9562(5)	-0.0024(8)	0.1400(4)	0.08
H(40A)	4a	0.8672(5)	0.0540(7)	-0.0378(4)	0.08
H(40B)	4a	0.8437(5)	-0.0669(7)	0.0062(4)	0.08
H(40C)	4a	0.9346(5)	-0.0325(7)	0.0131(4)	0.08
H(41A)	4a	0.7826(4)	0.2114(5)	0.0089(4)	0.08
H(41B)	4a	0.7978(4)	0.2304(5)	0.0901(4)	0.08
H(41C)	4a	0.7601(4)	0.0924(5)	0.0545(4)	0.08
H(42A)	4a	0.9253(5)	0.2658(6)	0.0201(5)	0.08
H(42B)	4a	0.9922(5)	0.1816(6)	0.0727(5)	0.08
H(42C)	4a	0.9365(5)	0.2831(6)	0.1009(5)	0.08

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

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	4a	0.5234(4)	0.1046(4)	0.9506(3)	0.027(2)	0.023(2)	0.035(2)	-0.001(2)	0.009(2)	-0.003(2)
C(2)	4a	0.5672(4)	0.3677(4)	0.9459(3)	0.032(2)	0.026(2)	0.040(2)	-0.001(2)	0.009(2)	0.001(2)
C(3)	4a	0.5959(4)	0.4884(4)	0.9827(4)	0.034(2)	0.025(2)	0.050(3)	-0.003(2)	0.012(2)	0.001(2)
C(4)	4a	0.6130(4)	0.4950(4)	1.0541(3)	0.037(2)	0.031(2)	0.053(3)	-0.008(2)	0.012(2)	-0.011(2)
C(5)	4a	0.5985(4)	0.3790(4)	1.0911(3)	0.036(2)	0.033(2)	0.036(2)	-0.003(2)	0.008(2)	-0.003(2)
C(6)	4a	0.5499(4)	0.1298(4)	1.0904(3)	0.043(2)	0.031(2)	0.028(2)	-0.000(2)	0.009(2)	-0.000(2)
C(7)	4a	0.6539(4)	-0.0647(5)	1.1156(3)	0.043(3)	0.034(2)	0.040(2)	-0.005(2)	0.001(2)	0.008(2)
C(8)	4a	0.6999(4)	-0.1606(5)	1.0910(4)	0.042(3)	0.027(2)	0.065(3)	0.002(2)	-0.006(2)	0.009(2)
C(9)	4a	0.6898(4)	-0.1741(5)	1.0200(4)	0.037(2)	0.025(2)	0.062(3)	0.000(2)	0.000(2)	-0.003(2)
C(10)	4a	0.6349(4)	-0.0936(4)	0.9732(3)	0.034(2)	0.026(2)	0.050(3)	-0.001(2)	0.007(2)	-0.001(2)
C(11)	4a	0.3708(4)	0.0465(4)	0.9450(3)	0.033(2)	0.026(2)	0.054(3)	0.002(2)	0.014(2)	-0.000(2)
C(12)	4a	0.3115(4)	0.0269(5)	0.9814(4)	0.036(2)	0.030(2)	0.071(4)	0.003(2)	0.022(2)	0.005(2)
C(13)	4a	0.3270(4)	0.0430(5)	1.0517(4)	0.047(3)	0.034(2)	0.071(4)	0.005(2)	0.036(3)	0.011(2)
C(14)	4a	0.4046(4)	0.0790(5)	1.0899(4)	0.054(3)	0.030(2)	0.053(3)	0.003(2)	0.027(2)	0.002(2)
C(15)	4a	0.4639(4)	0.0954(4)	1.0540(3)	0.042(2)	0.022(2)	0.044(2)	0.000(2)	0.019(2)	0.002(2)
C(16)	4a	0.4494(4)	0.0810(4)	0.9822(3)	0.032(2)	0.017(2)	0.041(2)	0.002(2)	0.015(2)	0.002(2)
C(17)	4a	0.5542(4)	0.2502(4)	0.9830(3)	0.024(2)	0.023(2)	0.038(2)	0.000(2)	0.009(2)	0.000(2)
C(18)	4a	0.5691(4)	0.2606(4)	1.0552(3)	0.028(2)	0.026(2)	0.036(2)	0.001(2)	0.007(2)	0.001(2)
C(19)	4a	0.6006(4)	0.0165(4)	1.0695(3)	0.035(2)	0.028(2)	0.036(2)	-0.004(2)	0.008(2)	0.003(2)
C(20)	4a	0.5883(4)	0.0037(4)	0.9967(3)	0.027(2)	0.024(2)	0.043(3)	-0.004(2)	0.006(2)	0.000(2)
B(1)	4a	0.5074	0.0878(5)	0.8614	0.036(3)	0.025(2)	0.032(2)	0.000(2)	0.007(2)	-0.001(2)
C(21)	4a	0.4208(4)	0.1589(4)	0.8193(3)	0.032(2)	0.033(2)	0.034(2)	-0.002(2)	0.010(2)	0.002(2)
C(22)	4a	0.3908(4)	0.2852(5)	0.8383(4)	0.039(3)	0.032(2)	0.063(3)	0.003(2)	0.007(2)	-0.004(2)
C(23)	4a	0.3189(4)	0.3426(5)	0.8015(4)	0.045(3)	0.038(3)	0.081(4)	0.011(2)	0.003(3)	0.008(3)
C(24)	4a	0.2719(4)	0.2766(6)	0.7440(4)	0.036(3)	0.057(3)	0.066(3)	0.006(2)	-0.003(2)	0.015(3)
C(25)	4a	0.2992(4)	0.1537(6)	0.7237(4)	0.042(3)	0.057(3)	0.049(3)	-0.005(3)	0.001(2)	0.005(3)
C(26)	4a	0.3724(4)	0.0973(5)	0.7599(3)	0.040(2)	0.044(2)	0.038(2)	-0.000(2)	0.004(2)	0.001(2)
C(27)	4a	0.5041(4)	-0.0780(4)	0.8376(3)	0.032(2)	0.029(2)	0.035(2)	-0.001(2)	0.006(2)	-0.004(2)
C(28)	4a	0.5487(5)	-0.1292(5)	0.7938(4)	0.080(4)	0.036(3)	0.055(3)	-0.005(3)	0.035(3)	-0.003(2)
C(29)	4a	0.5507(5)	-0.2685(6)	0.7766(4)	0.118(6)	0.040(3)	0.063(4)	0.001(3)	0.052(4)	-0.012(3)
C(30)	4a	0.5018(5)	-0.3628(5)	0.7984(4)	0.092(4)	0.029(2)	0.051(3)	0.002(3)	0.020(3)	-0.008(2)
C(31)	4a	0.4559(5)	-0.3170(5)	0.8404(4)	0.066(3)	0.029(2)	0.080(4)	-0.011(2)	0.025(3)	-0.005(2)
C(32)	4a	0.4574(4)	-0.1766(5)	0.8596(4)	0.054(3)	0.033(2)	0.072(3)	-0.002(2)	0.032(3)	-0.007(2)
C(33)	4a	0.5800(4)	0.1639(4)	0.8327(3)	0.038(2)	0.026(2)	0.035(2)	0.003(2)	0.011(2)	-0.003(2)
C(34)	4a	0.5635(4)	0.2343(5)	0.7696(3)	0.048(3)	0.046(3)	0.042(3)	0.004(2)	0.014(2)	0.010(2)
C(35)	4a	0.6225(5)	0.3027(6)	0.7454(4)	0.068(4)	0.062(3)	0.054(3)	-0.001(3)	0.026(3)	0.023(3)
C(36)	4a	0.7004(5)	0.3013(6)	0.7825(4)	0.059(4)	0.055(3)	0.071(4)	-0.012(3)	0.034(3)	0.003(3)
C(37)	4a	0.7197(4)	0.2306(6)	0.8439(4)	0.041(3)	0.060(3)	0.068(4)	-0.007(3)	0.018(3)	0.007(3)
C(38)	4a	0.6611(4)	0.1629(5)	0.8679(4)	0.040(3)	0.046(3)	0.048(3)	-0.000(2)	0.013(2)	0.013(2)
N(1)	4a	0.8807(4)	0.1046(4)	0.0636(3)	0.039(2)	0.040(2)	0.056(2)	0.000(2)	0.010(2)	-0.002(2)
C(39)	4a	0.9026(5)	0.0341(8)	0.1319(4)	0.070(4)	0.088(5)	0.075(4)	0.004(4)	0.005(3)	0.018(4)
C(40)	4a	0.8816(5)	0.0061(7)	0.0062(4)	0.073(4)	0.073(4)	0.078(4)	0.007(3)	0.021(3)	-0.019(3)
C(41)	4a	0.7978(4)	0.1652(5)	0.0534(4)	0.042(3)	0.047(3)	0.084(4)	0.004(2)	0.015(3)	-0.006(3)
C(42)	4a	0.9388(5)	0.2189(6)	0.0644(5)	0.053(4)	0.051(3)	0.116(6)	-0.015(3)	0.016(4)	0.005(3)

References

1. Wittig, G.; Tochtermann, W.: Reaktionen am Brückenkopf des Triptycens. *Justus Liebigs Ann. Chem.* **660** (1962) 23-33.
2. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.