Crystal structure of bis(1-ethyl-3-methylimidazol-2-ylidene)silver(I) tetracyanoborate, $[(C_6H_{10}N_2)_2Ag][B(CN)_4]$

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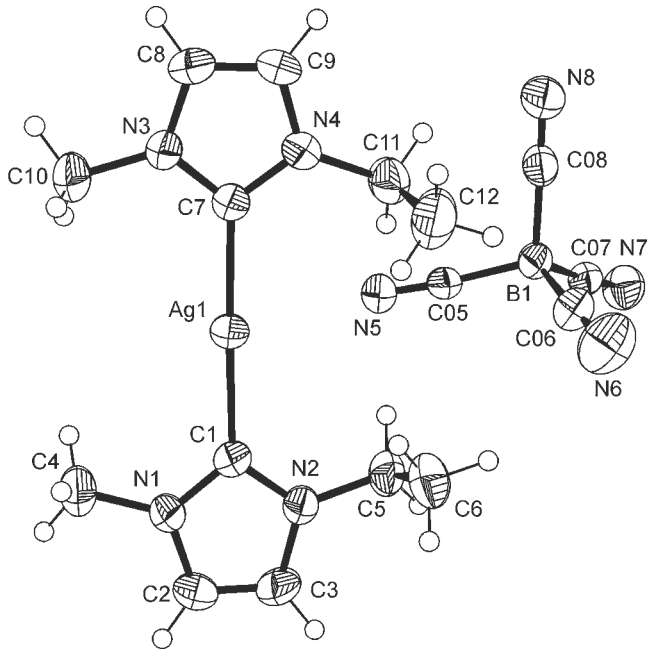

Abstract

$C_{16}H_{20}AgBN_8$, monoclinic, $P12_1/c1$ (no. 14), a = 11.2198(4) Å, b = 12.3987(4) Å, c = 15.4397(5) Å, β = 109.701(2)°, V = 2022.1 Å^3, Z = 4, $R_{gt(F)}$ = 0.032, $wR_{ref}(F^2)$ = 0.089, T = 233 K.

Source of material

The title compound was synthesized by stirring the commercial room temperature ionic liquid 1-ethyl-3-methylimidazolium tetracyanoborate and silver oxide in methanol for 96 h at room temperature. The colorless precipitate was collected by filtration, washed with methanol and diethyl ether, and dried (mp. 100 - 104 °C). ^{1}H NMR, ^{13}C NMR and IR spectroscopic data are available in the CIF file.

Discussion

The moderately coordinating tetracyanoborate anion is a relatively recent invention [1]. N-Heterocyclic carbene (NHC)-silver complexes are valuable carbene transfer reagents for the conversion to other metal NHC systems [2-5]. To our knowledge, the title compound is the first tetracyanoborate of an N-heterocyclic carbene-silver complex. Predictably, when an imidazolium salt with a weakly coordinating anion is used to generate the carbene, the resulting cationic silver complex is of the biscarbene type [2].

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The central carbene-metal bond lengths C1—Ag and C7—Ag are 2.082(3) and 2.079(3) Å, respectively, with a pertinent angle of 176.7(2)°. The N–C–N 'carbene angles' are 104.6° and 104.9°, which is unusually large [6,7]. In the crystal structure, the silver atoms are located in a glide plane perpendicular to [010]. The tetracyanoborate anions exhibit a slightly distorted tetrahedral shape and are packed along a two-fold screw axis in the direction of the crystallographic axis [010]. The B–C–N angles range from 174° to 179°. Weak interionic contacts were observed: C2–H^{i}···N5 (H···A distance 2.65 Å, D···A distance 3.460(5) Å, D–H···A angle 145°), C3–H^{ii}···N7 (2.69 Å, 3.521(5) Å, 148°), C8–H^{iii}···N5 (2.62 Å, 3.480(5) Å, 152°), and C9–H^{iv}···N7 (2.64 Å, 3.398(5) Å, 138°), respectively; symmetry codes: (i) $-x,1-y,1-z$; (ii) $1-x,-\frac{1}{2}+y,\frac{3}{2}-z$; (iii) $-x,-y,1-z$; (iv) $1-x,\frac{1}{2}+y,\frac{3}{2}-z$.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.03 × 0.1 × 0.3 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.13 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ/ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11635, 3562
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2512
$N(param)_{refined}$:	239
Programs:	SHELXS-97, SHELXL-97 [8]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	–0.0285	0.6559	0.3620	0.065
H(3)	4*e*	0.1988	0.6462	0.4492	0.064
H(4A)	4*e*	–0.1646	0.4368	0.2579	0.090
H(4B)	4*e*	–0.2109	0.5198	0.3178	0.090
H(4C)	4*e*	–0.1805	0.3990	0.3514	0.090
H(5A)	4*e*	0.2926	0.3721	0.5290	0.059
H(5B)	4*e*	0.3470	0.4912	0.5414	0.059
H(6A)	4*e*	0.4542	0.3888	0.4656	0.091
H(6B)	4*e*	0.3751	0.4787	0.3974	0.091
H(6C)	4*e*	0.3250	0.3583	0.3879	0.091
H(8)	4*e*	–0.0468	–0.1503	0.3466	0.066
H(9)	4*e*	0.1796	–0.1495	0.4363	0.067
H(10A)	4*e*	–0.1883	0.1102	0.3427	0.093
H(10B)	4*e*	–0.2233	–0.0087	0.3053	0.093
H(10C)	4*e*	–0.1713	0.0747	0.2490	0.093
H(11A)	4*e*	0.3329	–0.0012	0.5372	0.066
H(11B)	4*e*	0.2826	0.1191	0.5286	0.066
H(12A)	4*e*	0.4468	0.1040	0.4686	0.112
H(12B)	4*e*	0.3202	0.1386	0.3901	0.112
H(12C)	4*e*	0.3677	0.0176	0.3965	0.112

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}
Ag(1)	4*e*	0.06089(3)	0.25053(2)	0.40862(2)	0.0394(2)	0.0289(2)	0.0622(2)	0.0010(1)	0.0113(2)	–0.0011(1)
C(1)	4*e*	0.0691(3)	0.4183(3)	0.4098(2)	0.038(2)	0.034(2)	0.045(2)	–0.003(2)	0.012(2)	–0.001(2)
C(2)	4*e*	0.0193(4)	0.5930(3)	0.3829(3)	0.055(2)	0.032(2)	0.073(3)	0.006(2)	0.017(2)	0.003(2)
C(3)	4*e*	0.1426(4)	0.5877(3)	0.4307(3)	0.054(2)	0.033(2)	0.071(3)	–0.009(2)	0.018(2)	–0.007(2)
C(4)	4*e*	–0.1563(3)	0.4585(3)	0.3200(3)	0.037(2)	0.065(3)	0.069(3)	0.002(2)	0.005(2)	–0.001(2)
C(5)	4*e*	0.2992(3)	0.4381(3)	0.4960(3)	0.035(2)	0.058(2)	0.046(2)	–0.003(2)	0.003(2)	0.001(2)
C(6)	4*e*	0.3696(4)	0.4138(4)	0.4310(3)	0.044(2)	0.072(3)	0.064(3)	0.012(2)	0.016(2)	–0.004(2)
C(7)	4*e*	0.0602(3)	0.0829(3)	0.4035(2)	0.036(2)	0.034(2)	0.050(2)	0.001(2)	0.012(2)	–0.000(2)
C(8)	4*e*	0.0035(4)	–0.0894(3)	0.3703(3)	0.054(2)	0.032(2)	0.078(3)	–0.005(2)	0.020(2)	0.001(2)
C(9)	4*e*	0.1263(4)	–0.0890(3)	0.4195(3)	0.051(2)	0.031(2)	0.079(3)	0.006(2)	0.013(2)	0.010(2)
C(10)	4*e*	–0.1656(3)	0.0511(3)	0.3101(3)	0.033(2)	0.058(3)	0.087(3)	0.003(2)	0.010(2)	–0.002(2)
C(11)	4*e*	0.2883(4)	0.0547(3)	0.4934(3)	0.046(2)	0.055(2)	0.053(2)	0.003(2)	0.001(2)	–0.000(2)
C(12)	4*e*	0.3622(4)	0.0811(4)	0.4318(3)	0.049(2)	0.095(4)	0.076(3)	–0.009(2)	0.014(2)	0.007(3)
N(1)	4*e*	–0.0246(3)	0.4884(2)	0.3698(2)	0.034(2)	0.037(2)	0.053(2)	0.004(1)	0.011(1)	0.001(1)
N(2)	4*e*	0.1721(3)	0.4800(2)	0.4479(2)	0.034(2)	0.039(2)	0.046(2)	–0.002(1)	0.010(1)	–0.002(1)
N(3)	4*e*	–0.0357(3)	0.0163(2)	0.3607(2)	0.032(2)	0.038(2)	0.056(2)	–0.001(1)	0.012(2)	0.001(1)
N(4)	4*e*	0.1603(3)	0.0169(2)	0.4414(2)	0.041(2)	0.037(2)	0.049(2)	0.002(1)	0.009(2)	0.006(1)
B(1)	4*e*	0.5066(4)	0.2010(3)	0.7433(3)	0.035(2)	0.038(2)	0.044(2)	–0.003(2)	0.008(2)	–0.004(2)
N(5)	4*e*	0.2585(3)	0.2595(2)	0.6925(3)	0.037(2)	0.046(2)	0.082(3)	–0.001(1)	0.009(2)	0.003(2)
C(05)	4*e*	0.3631(3)	0.2397(2)	0.7147(2)	0.038(2)	0.030(2)	0.045(2)	–0.001(2)	0.009(2)	0.000(2)
N(6)	4*e*	0.6219(5)	0.2662(3)	0.6233(3)	0.089(3)	0.108(4)	0.072(3)	–0.018(3)	0.044(3)	–0.002(2)
C(06)	4*e*	0.5703(4)	0.2406(3)	0.6721(3)	0.046(2)	0.053(2)	0.049(2)	–0.007(2)	0.016(2)	–0.003(2)
N(7)	4*e*	0.6537(4)	0.2695(3)	0.9132(3)	0.056(2)	0.065(2)	0.048(2)	–0.007(2)	0.004(2)	–0.002(2)
C(07)	4*e*	0.5889(3)	0.2447(3)	0.8426(3)	0.036(2)	0.038(2)	0.043(2)	–0.002(2)	0.012(2)	0.007(2)
N(8)	4*e*	0.5071(3)	–0.0206(2)	0.7507(2)	0.057(2)	0.050(2)	0.083(3)	0.003(2)	0.019(2)	–0.008(2)
C(08)	4*e*	0.5077(3)	0.0709(3)	0.7472(2)	0.036(2)	0.048(2)	0.048(2)	0.003(2)	0.010(2)	–0.006(2)

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