Crystal structure of *bis*-(1-(2-(diethylamino)ethyl)-3-methylimidazolin-2-ylidene) di-silver(I) *bis*-(triflimide), $Ag_2[C_{10}H_{19}N_3]_2[C_2F_6NO_4S_2]_2$, $C_{24}H_{38}Ag_2F_{12}N_8O_8S_4$

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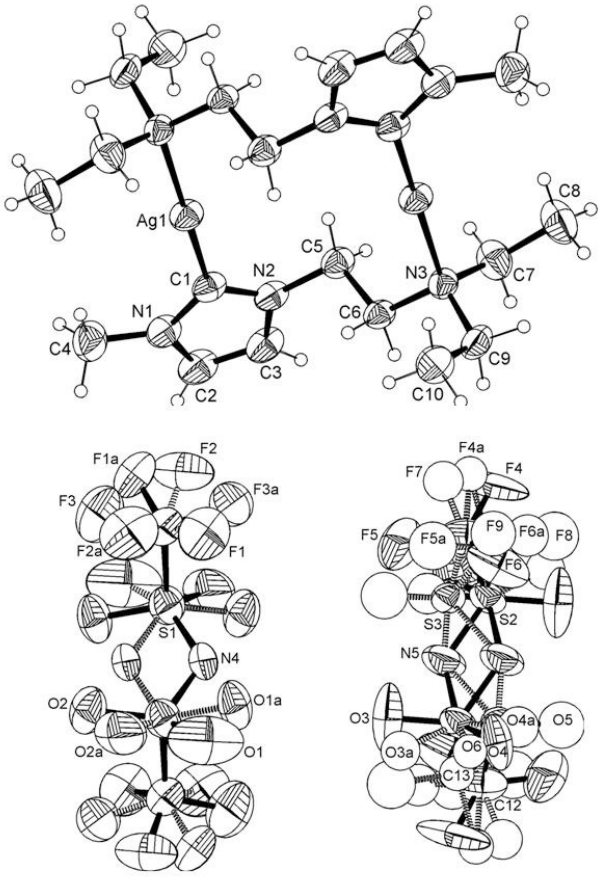
Received January 18, 2012, accepted July 06, 2012, available online July 26, 2012, CCDC no. 1267/3798



Abstract

$C_{24}H_{38}Ag_2F_{12}N_8O_8S_4$, triclinic, $P\bar{1}$ (no. 2), a = 9.4438(4) Å, b = 9.8561(3) Å, c = 12.0065(4) Å, α = 101.606(2)°, β = 93.366(2)°, γ = 92.789(2)°, V = 1090.7 Å^3, Z = 1, $R_{gt}(F)$ = 0.0347, $wR_{ref}(F^2)$ = 0.0837, T = 233 K.

Table 1. Data collection and handling.

Crystal:	colourless prisms, size 0.08×0.11×0.12 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.90 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6375, 3813
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3284
$N(param)_{refined}$:	371
Programs:	SHELXS-97 [14]

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Source of material

The title compound was synthesized by stirring 1-(2-(diethylamino)ethyl)-3-methylimidazolium triflimide [1] and silver oxide in methanol. After filtration, single crystals were grown from the solution at -20°C.

Experimental details

In the present case, the asymmetric unit contains one half molecule of the dicationic Ag complex and two half units of the triflimide anions. The two triflimide molecules are located near an inversion centre and are disordered in a way which is not consistent with the symmetry operation of the inversion. For the first anion, we have a 1:1 disorder with split atom positions for O1, O2, F1, F2, F3 and O1A, O2A, F1A, F2A, F3A. The nitrogen atom N4 lies near an inversion centre (occupancy 0.5). The complete anion is generated in two ways: S1–C11–O1–O2–F1–F2–F3–N4 connected with the inverted coordinates of S1–C11–O1A–O2A–F1A–F2A–F3A, or S1–C11–O1A–O2A–F1A–F2A–F3A connected with the inverted coordinates of N4–S1–C11–O1–O2–F1–F2–F3. The second anion shows even more disorder: one identical to the first anion, the second due to additional splitting of the S- and C-positions. Thus, four positions are possible. The occupancies are 0.75 for S2 and C12; 0.5 for N5, O3, O4, F4, F5, and F6; and 0.25 for O3A, O4A, F4A, F5A, F6A, S3, C13, O5, O6, F7, F8, and F9. With the exception of S3, the atoms of the minor component were refined isotropically. Cation and anions are shown separately for clarity.

Discussion

Silver *N*-heterocyclic carbene (NHC) complexes have received much attention, and their chemistry has been reviewed [2-4]. The silver compounds are particularly well-suited precursors for other metal NHC systems by transmetallation. Thus, they bear potential in catalysis [5], for their optical properties [6-8], and as pharmaceuticals [9,10]. Bidentate amino-carbene ligands [11] often yield cyclic dinuclear metal complexes with interesting structural properties.

The central metal–ligand bonds C1–Ag1 and N3–Ag1 are 2.073 and 2.178 Å long, respectively, and deviate only slightly from linearity with a C1–Ag1–N3 angle of 172.6°. The N1–C1–N2 'carbene angle' of 104.7° in this silver complex is in line with that observed in other imidazolin-2-ylidene-metal complexes (mean value 104.0° from 4185 related structures in the CSD), which is significantly smaller than the typical value in imidazolium salts (mean value 108.7° from 1202 cations in the CSD). The triflimide was selected as a weakly coordinating anion in order to lend lipophilic properties to the resulting salt. Nonetheless, the triflimide ions accept several weak hydrogen bonds from the

imidazolium cation and the alkyl backbone of the ligand (H···acceptor and donor···acceptor distance, donor–H···acceptor angle): C8–H···O1^{i} (2.41 and 3.23 Å, 143°), C9–H···O1^{i} (2.23 and 3.15 Å, 156°), C5–H···O4^{ii} (2.47 and 3.33 Å, 147°), C3–H···O2^{iii} (2.48 and 3.27 Å, 142°), C4–H···O3^{iv} (2.56 and 3.21 Å, 125°). Only one significant hydrogen···fluorine interaction, C6–H···F6^{iv} (2.44 and 3.06 Å, 121°), was identified. A short Ag1···O4 contact (3.071 Å) is also observed. Dinuclear silver complexes are sometimes stabilized by argentophilic attraction [12, 13], but here the distance between the silver atoms (Ag···Ag 4.478 Å) is safely beyond the range of any interaction.

Symmetry codes: **i**: 1+x, y, -1+z; **ii**: 1-x,-y,-z; **iii**: x, y, -1+z; **iv**: 1-x, 1-y, -z.

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(2)	2*i*		–0.0664	0.2701	–0.0216	0.101
H(3)	2*i*		0.0015	0.0841	–0.1731	0.093
H(4A)	2*i*		0.2057	0.3304	0.2219	0.146
H(4B)	2*i*		0.0378	0.3120	0.2189	0.146
H(4C)	2*i*		0.1105	0.4257	0.1608	0.146
H(5A)	2*i*		0.1949	–0.1062	–0.1754	0.078
H(5B)	2*i*		0.3213	–0.0726	–0.0788	0.078
H(6A)	2*i*		0.2820	0.0668	–0.2633	0.069
H(6B)	2*i*		0.3962	0.1225	–0.1605	0.069
H(7A)	2*i*		0.3192	–0.1416	–0.4021	0.102
H(7B)	2*i*		0.3167	–0.2200	–0.2994	0.102
H(8A)	2*i*		0.4244	–0.3522	–0.4527	0.160
H(8B)	2*i*		0.5357	–0.3108	–0.3457	0.160
H(8C)	2*i*		0.5416	–0.2299	–0.4469	0.160
H(9A)	2*i*		0.5858	–0.0222	–0.3905	0.091
H(9B)	2*i*		0.4573	0.0755	–0.3841	0.091
H(10A)	2*i*		0.6691	0.2051	–0.3234	0.137
H(10B)	2*i*		0.6957	0.1203	–0.2259	0.137
H(10C)	2*i*		0.5678	0.2190	–0.2216	0.137
O(3A)	2*i*	0.25	0.742(2)	0.404(2)	–0.019(2)	0.108(6)
O(4A)	2*i*	0.25	0.529(2)	0.322(3)	0.059(2)	0.117(7)
F(4A)	2*i*	0.25	0.782(2)	0.452(2)	0.222(2)	0.086(4)
F(5A)	2*i*	0.25	0.622(3)	0.585(3)	0.238(2)	0.161(9)
F(6A)	2*i*	0.25	0.778(2)	0.619(2)	0.150(2)	0.123(5)
C(13)	2*i*	0.25	0.699(5)	0.511(5)	0.131(3)	0.09(1)
O(5)	2*i*	0.25	0.467(2)	0.422(2)	0.157(2)	0.152(6)
O(6)	2*i*	0.25	0.636(2)	0.291(2)	0.015(1)	0.081(5)
F(7)	2*i*	0.25	0.732(2)	0.454(2)	0.246(1)	0.112(6)
F(8)	2*i*	0.25	0.812(2)	0.505(2)	0.061(2)	0.164(6)
F(9)	2*i*	0.25	0.732(3)	0.633(3)	0.201(2)	0.141(8)

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	2*i*		0.38689(3)	0.10541(3)	0.14917(2)	0.0529(2)	0.0832(2)	0.0639(2)	0.0156(1)	0.0015(1)	0.0255(1)
N(1)	2*i*		0.1161(3)	0.2325(3)	0.0702(3)	0.054(2)	0.095(2)	0.072(2)	0.023(2)	0.015(1)	0.034(2)
N(2)	2*i*		0.1759(3)	0.0710(3)	–0.0621(2)	0.046(2)	0.085(2)	0.065(2)	0.013(1)	0.010(1)	0.035(2)
N(3)	2*i*		0.4529(3)	–0.0492(3)	–0.2670(2)	0.054(2)	0.069(2)	0.051(2)	0.011(1)	0.002(1)	0.014(1)
C(1)	2*i*		0.2166(3)	0.1411(4)	0.0441(3)	0.048(2)	0.081(2)	0.066(2)	0.011(2)	0.013(2)	0.033(2)
C(2)	2*i*		0.0142(4)	0.2181(5)	–0.0194(4)	0.053(2)	0.126(4)	0.088(3)	0.034(2)	0.013(2)	0.050(3)
C(3)	2*i*		0.0509(4)	0.1173(5)	–0.1018(3)	0.048(2)	0.120(3)	0.072(2)	0.018(2)	0.003(2)	0.040(2)
C(4)	2*i*		0.1176(5)	0.3336(5)	0.1768(4)	0.094(3)	0.122(4)	0.081(3)	0.041(3)	0.022(2)	0.020(3)
C(5)	2*i*		0.2585(4)	–0.0318(4)	–0.1293(3)	0.058(2)	0.077(2)	0.065(2)	0.009(2)	0.008(2)	0.025(2)
C(6)	2*i*		0.3466(3)	0.0383(3)	–0.2066(3)	0.051(2)	0.070(2)	0.056(2)	0.011(2)	0.003(2)	0.024(2)
C(7)	2*i*		0.3791(5)	–0.1727(4)	–0.3439(4)	0.078(3)	0.086(3)	0.081(3)	0.012(2)	–0.013(2)	–0.003(2)
C(8)	2*i*		0.4792(6)	–0.2757(5)	–0.4026(4)	0.114(4)	0.089(3)	0.103(4)	0.018(3)	0.000(3)	–0.013(3)
C(9)	2*i*		0.5287(4)	0.0380(5)	–0.3380(3)	0.077(3)	0.105(3)	0.055(2)	0.024(2)	0.018(2)	0.031(2)
C(10)	2*i*		0.6236(5)	0.1559(5)	–0.2714(4)	0.091(3)	0.089(3)	0.099(3)	–0.003(2)	0.035(3)	0.030(3)
S(1)	2*i*		–0.0755(1)	–0.0992(1)	0.5438(1)	0.0726(7)	0.0925(7)	0.0962(8)	0.0129(6)	0.0038(6)	0.0188(6)
C(11)	2*i*		–0.0236(7)	–0.2629(6)	0.4615(7)	0.104(5)	0.090(4)	0.127(5)	–0.008(3)	–0.024(4)	0.016(4)
N(4)	2*i*	0.5	–0.0585(9)	0.0010(8)	0.4587(8)	0.096(6)	0.086(5)	0.144(7)	0.005(4)	–0.060(5)	0.033(5)
O(1)	2*i*	0.5	–0.220(1)	–0.130(1)	0.555(2)	0.069(5)	0.150(9)	0.49(3)	0.019(5)	0.11(1)	0.07(1)
O(2)	2*i*	0.5	0.0190(9)	–0.080(1)	0.6436(6)	0.119(6)	0.143(7)	0.062(4)	–0.017(5)	–0.007(4)	0.025(4)
F(1)	2*i*	0.5	0.113(1)	–0.261(1)	0.445(1)	0.109(7)	0.161(8)	0.151(8)	0.035(6)	0.041(7)	–0.008(6)
F(2)	2*i*	0.5	–0.059(2)	–0.362(1)	0.512(1)	0.23(2)	0.088(6)	0.19(1)	0.009(9)	0.05(1)	0.031(7)
F(3)	2*i*	0.5	–0.092(3)	–0.292(2)	0.362(2)	0.49(3)	0.18(1)	0.19(2)	0.02(2)	–0.21(2)	–0.05(1)
O(1A)	2*i*	0.5	–0.2003(7)	–0.0704(9)	0.4839(6)	0.055(4)	0.154(7)	0.115(5)	0.013(4)	0.004(3)	0.060(5)
O(2A)	2*i*	0.5	–0.097(2)	–0.114(1)	0.6543(7)	0.28(2)	0.119(7)	0.080(5)	–0.002(9)	–0.005(8)	0.035(5)
F(1A)	2*i*	0.5	–0.128(1)	–0.356(1)	0.457(1)	0.139(8)	0.132(8)	0.19(1)	–0.043(6)	–0.065(7)	0.015(7)
F(2A)	2*i*	0.5	–0.003(2)	–0.261(1)	0.365(1)	0.18(1)	0.163(9)	0.13(1)	0.038(9)	0.06(1)	–0.028(8)
F(3A)	2*i*	0.5	0.076(2)	–0.307(1)	0.526(2)	0.15(1)	0.110(6)	0.30(2)	0.042(7)	–0.09(1)	0.008(8)
S(2)	2*i*	0.75	0.6152(2)	0.4249(2)	0.0268(2)	0.065(1)	0.070(1)	0.129(2)	0.0210(8)	0.003(1)	0.034(1)
C(12)	2*i*	0.75	0.681(2)	0.513(1)	0.176(1)	0.14(1)	0.065(5)	0.15(1)	0.029(6)	–0.04(1)	0.016(8)
N(5)	2*i*	0.5	0.4541(9)	0.457(1)	0.015(1)	0.073(6)	0.132(8)	0.21(1)	0.015(5)	–0.018(6)	0.091(7)
O(3)	2*i*	0.5	0.712(2)	0.458(1)	–0.051(1)	0.23(1)	0.140(9)	0.20(1)	–0.033(9)	0.14(1)	0.033(8)
O(4)	2*i*	0.5	0.583(1)	0.2855(9)	0.035(1)	0.118(8)	0.071(5)	0.18(1)	–0.038(5)	0.062(7)	0.005(5)
F(4)	2*i*	0.5	0.797(1)	0.457(2)	0.182(1)	0.142(9)	0.28(1)	0.18(1)	0.132(9)	–0.091(9)	–0.055(9)
F(5)	2*i*	0.5	0.573(2)	0.503(2)	0.2421(8)	0.22(1)	0.24(1)	0.109(6)	0.02(1)	0.043(7)	0.052(7)
F(6)	2*i*	0.5	0.690(1)	0.6487(7)	0.162(2)	0.125(7)	0.051(3)	0.28(1)	0.018(4)	–0.051(8)	–0.020(5)
S(3)	2*i*		0.5565(7)	0.4219(5)	0.0741(5)	0.077(4)	0.056(3)	0.093(4)	0.009(3)	–0.001(3)	0.029(2)

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