Crystal structure of 3,3′-trimethylene-2,2′-biquinolinium tetrabromoaurate(III), $[C_{21}H_{16}N_2H]^+[AuBr_4]^-$

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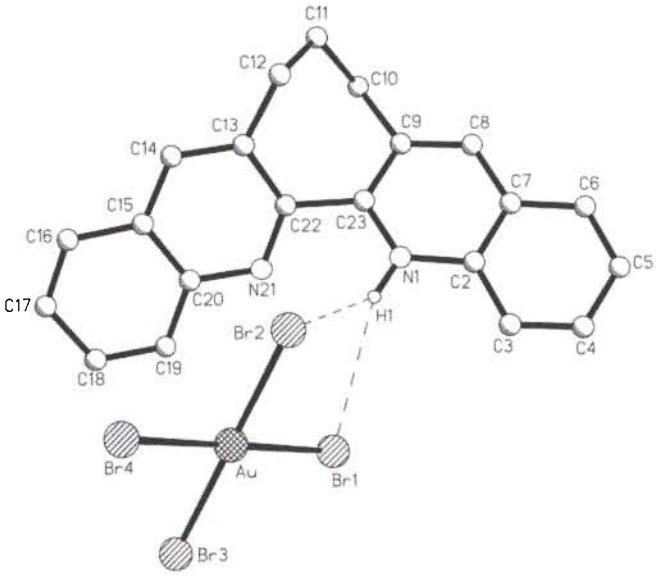

Abstract

$C_{21}H_{17}AuBr_4N_2$, monoclinic, $P12_1/n1$ (No. 14), a = 13.718(1) Å, b = 16.262(2) Å, c = 10.132(1) Å, β = 93.80(1)°, V = 2255.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.055, $wR(F)$ = 0.028, T = 293 K.

Source of material

An ethanolic solution of $KAuBr_4$ was treated with the stoichiometric quantity of 3,3′-trimethylene-2,2′-biquinolinium bromide. The resulting deep brownish red precipitate was recrystallized from methanol resulting dark red crystals [1].

Discussion

The resulting compound is obviously a byproduct of the reaction as above. Bond lengths in the square planar tetrabromoaurate(III)(1–) anion: d(Au—Br1) =242.9 pm, d(Au—Br2) = 242 pm, d(Au—Br3) = 242, 1 pm, d(Au—Br4) = , 241.4(1) pm). The anion is connected with the 3,3′-trimethylene-2,2′-biquinolinium cation $(C_{21}H_{16}NNH)^+$ by one bifurcated (three center) Br···H(N)···Br hydrogen bond (*trans* configuration, d(Br···H) = 290 pm). The plane of the hydrogen bonded chinoline fragment is almost perpendicular to the plane of the anion [2]. The torsion angle around the C22—C23 bond is about 38°.

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Table 1. Data collection and handling.

Crystal:	dark red prism, size 0.20 × 0.35 × 0.18 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	136.20 cm^{-1}
Diffractometer, scan mode:	Stoe-Stadi 4, θ/θ
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5726, 3973
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 2\,\sigma(F_{obs})$, 2643
$N(param)_{refined}$:	256
Program:	SHELXTL-plus [3]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.16552	0.92494	0.53644	0.10(4)
H(3)	4*e*	0.2626(7)	0.9462(7)	0.3490(9)	0.08
H(4)	4*e*	0.3938(7)	1.0120(7)	0.265(1)	0.08
H(5)	4*e*	0.4839(7)	1.1106(7)	0.392(1)	0.08
H(6)	4*e*	0.4549(7)	1.1325(7)	0.611(1)	0.08
H(8)	4*e*	0.3471(6)	1.1064(6)	0.7977(9)	0.08
H(10B)	4*e*	0.1246(7)	1.0632(6)	0.9151(9)	0.08
H(10A)	4*e*	0.2275(7)	1.0988(6)	0.9614(9)	0.08
H(11B)	4*e*	0.2722(7)	0.9853(7)	1.0676(9)	0.08
H(11A)	4*e*	0.1619(7)	0.9933(7)	1.0974(9)	0.08
H(12B)	4*e*	0.1800(6)	0.8612(6)	1.0473(8)	0.08
H(12A)	4*e*	0.2424(6)	0.8817(6)	0.9274(8)	0.08
H(14)	4*e*	0.0166(6)	0.8159(6)	0.9923(8)	0.08
H(16)	4*e*	–0.1472(7)	0.7529(7)	0.915(1)	0.08
H(17)	4*e*	–0.2606(7)	0.7263(8)	0.744(1)	0.08
H(18)	4*e*	–0.2516(6)	0.7920(6)	0.537(1)	0.08
H(19)	4*e*	–0.1208(6)	0.8776(6)	0.499(1)	0.08

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}
Au	4*e*	0.00890(3)	0.73924(2)	0.38003(4)	0.0371(2)	0.0364(2)	0.0401(2)	0.0055(2)	–0.0007(1)	0.0010(2)
Br(1)	4*e*	0.04342(7)	0.87501(6)	0.2947(1)	0.0613(6)	0.0464(7)	0.0523(6)	–0.0000(5)	–0.0001(5)	0.0140(5)
Br(2)	4*e*	0.14962(6)	0.75241(7)	0.5358(1)	0.0452(5)	0.0450(7)	0.0578(6)	0.0071(5)	–0.0118(4)	0.0010(6)
Br(3)	4*e*	–0.13349(7)	0.72718(8)	0.2268(1)	0.0547(6)	0.0743(9)	0.0582(7)	–0.0016(6)	–0.0186(5)	0.0019(6)
Br(4)	4*e*	–0.02021(8)	0.60257(7)	0.4616(1)	0.0697(8)	0.0444(7)	0.0937(9)	–0.0057(6)	–0.0080(6)	0.0185(7)
N(1)	4*e*	0.2034(5)	0.9609(5)	0.5888(7)	0.046(5)	0.037(5)	0.042(4)	–0.011(4)	0.004(4)	–0.005(4)
C(2)	4*e*	0.2807(6)	0.9984(6)	0.5333(9)	0.038(5)	0.046(6)	0.045(6)	–0.007(5)	–0.005(5)	0.007(5)
C(3)	4*e*	0.3016(7)	0.9839(7)	0.4030(9)	0.059(7)	0.075(8)	0.043(6)	–0.022(6)	0.007(5)	0.001(6)
C(4)	4*e*	0.3773(7)	1.0233(7)	0.353(1)	0.069(7)	0.090(9)	0.042(6)	0.000(7)	0.008(6)	0.006(6)
C(5)	4*e*	0.4325(7)	1.0809(7)	0.431(1)	0.045(6)	0.085(9)	0.067(8)	–0.014(6)	0.011(6)	0.011(7)
C(6)	4*e*	0.4147(7)	1.0951(7)	0.558(1)	0.057(7)	0.054(7)	0.068(7)	–0.014(6)	0.002(6)	0.007(6)
C(7)	4*e*	0.3359(6)	1.0541(6)	0.6122(9)	0.038(5)	0.046(6)	0.043(6)	0.000(5)	0.002(4)	0.002(5)
C(8)	4*e*	0.3090(6)	1.0689(6)	0.7422(9)	0.045(6)	0.037(6)	0.059(7)	–0.001(5)	–0.001(5)	–0.007(5)
C(9)	4*e*	0.2305(7)	1.0312(6)	0.7913(9)	0.052(6)	0.033(6)	0.042(6)	0.000(5)	–0.001(5)	–0.011(5)
C(10)	4*e*	0.1931(7)	1.0511(6)	0.9273(9)	0.056(6)	0.041(6)	0.056(7)	–0.014(5)	0.003(5)	–0.011(5)
C(11)	4*e*	0.2062(7)	0.9834(7)	1.0297(9)	0.070(7)	0.073(9)	0.043(7)	–0.012(7)	–0.012(6)	–0.009(6)
C(12)	4*e*	0.1867(6)	0.8977(6)	0.9740(8)	0.057(6)	0.051(7)	0.036(5)	–0.000(5)	0.001(4)	0.009(5)
C(13)	4*e*	0.0976(6)	0.8881(5)	0.8810(8)	0.043(5)	0.035(6)	0.035(5)	0.005(5)	0.002(4)	0.000(5)
C(14)	4*e*	0.0192(6)	0.8407(6)	0.9066(8)	0.051(6)	0.044(6)	0.035(5)	–0.003(5)	0.005(5)	–0.006(5)
C(15)	4*e*	–0.0578(6)	0.8276(6)	0.8112(9)	0.042(5)	0.036(6)	0.052(6)	–0.008(5)	0.012(5)	–0.005(5)
C(16)	4*e*	–0.1398(7)	0.7772(7)	0.830(1)	0.067(6)	0.072(8)	0.055(6)	–0.025(6)	0.022(5)	–0.009(6)
C(17)	4*e*	–0.2076(7)	0.7632(8)	0.731(1)	0.054(6)	0.11(1)	0.074(7)	–0.044(7)	0.016(6)	–0.026(8)
C(18)	4*e*	–0.2014(6)	0.8014(6)	0.606(1)	0.043(6)	0.062(7)	0.067(7)	0.005(6)	–0.004(5)	–0.014(6)
C(19)	4*e*	–0.1250(6)	0.8516(6)	0.584(1)	0.033(5)	0.056(7)	0.062(6)	–0.001(5)	0.013(5)	–0.001(6)
C(20)	4*e*	–0.0531(6)	0.8651(5)	0.6839(8)	0.040(5)	0.031(5)	0.040(5)	–0.000(4)	0.007(4)	–0.006(5)
N(21)	4*e*	0.0251(5)	0.9139(4)	0.6575(6)	0.038(4)	0.031(4)	0.034(4)	0.005(4)	0.003(3)	–0.002(3)
C(22)	4*e*	0.0952(6)	0.9229(5)	0.7528(8)	0.044(5)	0.033(5)	0.029(5)	0.008(4)	0.001(4)	–0.005(4)
C(23)	4*e*	0.1786(6)	0.9728(6)	0.7116(8)	0.032(5)	0.043(6)	0.036(5)	0.000(4)	0.001(4)	0.006(5)

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