Crystal structure of tribromo(3,3′-dimethyl-2,2′-biquinoline)gold(III) hemiglycolate, $(C_9H_5CH_3N)_2AuBr_3 \cdot 0.5(CH_2OH)_2$

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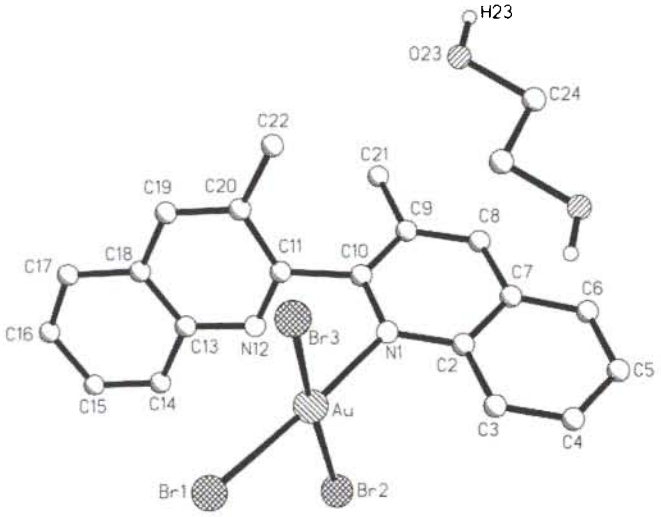

Abstract

$C_{22}H_{22}AuBr_3N_2O_2$, monoclinic, $P12_1/n1$ (No. 14), a = 13.071(2) Å, b = 18.345(4) Å, c = 9.670(2) Å, β = 102.14(1)°, V = 2266.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.044, $wR(F)$ = 0.037, T = 293 K.

Source of material

Equimolar (0.4 mmol) methanolic solutions of 3,3′-dimethyl-2,2′-biquinoline (50 mL) and $KAuBr_4$ (40 mL) were mixed dropwise under stirring. The dark red solution (refluxing for 1h under N_2) was rotoevaporated to dryness, treated with 50 mL dichloromethane, filtered and partially rotoevaporated again. Dark red crystals, formed after cooling, were recrystallized from dichloromethane/glycol [1].

Discussion

Instead of the expected fivefold coordinated gold(III) the reaction yielded square planar tribromo(3,3′-dimethyl-2,2′-biquinolino-κ*N*)gold(III)complex. Bond lengths Au—N1, Au—Br1, Au—Br2, Au—Br3 are 207.3 pm, 239.3 pm, 243.1 pm, 242.2 pm, respectively (significant *trans* effect). The plane of the κ*N*-bonded chinoline fragment is almost perpendicular to that of $AuBr_3N$. The twist angle around C10–C11 is about 66°.The solvens molecule glycol is located in cavities without any interactions to Au.

Table 1. Data collection and handling.

Crystal:	dark red prism, size 0.25 × 0.25 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	117.90 cm^{-1}
Diffractometer, scan mode:	Siemens R3m/V, Wyckoff
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5594, 5208
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\,\sigma(F_{obs})$, 3957
$N(param)_{refined}$:	254
Program:	SHELXTL-plus [2]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	0.8206(7)	0.7611(5)	1.048(1)	0.08
H(4)	4*e*	0.7375(8)	0.7109(5)	1.2139(9)	0.08
H(5)	4*e*	0.6498(8)	0.5987(6)	1.169(1)	0.08
H(6)	4*e*	0.6495(8)	0.5349(5)	0.965(1)	0.08
H(8)	4*e*	0.7037(7)	0.5265(4)	0.732(1)	0.08
H(14)	4*e*	0.752(1)	0.8879(6)	0.411(1)	0.08
H(15)	4*e*	0.802(1)	0.9581(7)	0.237(1)	0.08
H(16)	4*e*	0.941(2)	0.9214(9)	0.139(2)	0.08
H(17)	4*e*	1.035(1)	0.8171(9)	0.213(1)	0.08
H(19)	4*e*	1.0642(8)	0.7109(7)	0.387(1)	0.08
H(21A)	4*e*	0.8182(8)	0.5931(5)	0.454(1)	0.08
H(21B)	4*e*	0.7096(8)	0.5595(5)	0.463(1)	0.08
H(21C)	4*e*	0.8132(8)	0.5177(5)	0.529(1)	0.08
H(22A)	4*e*	1.0004(8)	0.5994(6)	0.668(1)	0.08
H(22B)	4*e*	1.1042(8)	0.6346(6)	0.642(1)	0.08
H(22C)	4*e*	1.0381(8)	0.5835(6)	0.527(1)	0.08
H(23)	4*e*	1.01504	0.42107	0.79564	0.08
H(24A)	4*e*	0.90467	0.46138	0.93824	0.08
H(24B)	4*e*	1.00241	0.42270	1.03101	0.08

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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}
Au	4*e*	0.90110(3)	0.81020(2)	0.82860(4)	0.0401(2)	0.0327(2)	0.0472(2)	–0.0037(2)	0.0146(1)	–0.0021(2)
Br(1)	4*e*	0.97894(7)	0.92878(5)	0.8442(1)	0.0573(6)	0.0388(5)	0.0935(8)	–0.0115(4)	0.0259(6)	–0.0052(5)
Br(2)	4*e*	0.72801(7)	0.86521(5)	0.7758(1)	0.0477(5)	0.0505(5)	0.0903(8)	0.0060(4)	0.0201(5)	0.0041(5)
Br(3)	4*e*	1.07194(7)	0.75552(5)	0.9086(1)	0.0458(5)	0.0572(6)	0.0743(7)	0.0058(4)	0.0095(5)	–0.0030(5)
N(1)	4*e*	0.8345(5)	0.7072(3)	0.8020(7)	0.035(3)	0.029(3)	0.056(4)	–0.006(3)	0.007(3)	–0.005(3)
C(2)	4*e*	0.7887(6)	0.6779(4)	0.9047(8)	0.038(4)	0.035(5)	0.044(5)	–0.003(3)	0.005(4)	0.007(3)
C(3)	4*e*	0.7866(7)	0.7146(5)	1.030(1)	0.061(6)	0.043(5)	0.060(6)	–0.010(4)	0.020(5)	–0.004(4)
C(4)	4*e*	0.7373(8)	0.6855(5)	1.1271(9)	0.068(6)	0.075(7)	0.042(5)	–0.013(6)	0.017(5)	–0.004(5)
C(5)	4*e*	0.6858(8)	0.6183(6)	1.101(1)	0.066(7)	0.093(8)	0.064(7)	–0.016(6)	0.014(6)	0.034(6)
C(6)	4*e*	0.6858(8)	0.5806(5)	0.981(1)	0.076(7)	0.057(6)	0.059(6)	–0.020(5)	0.008(5)	0.018(5)
C(7)	4*e*	0.7392(7)	0.6085(5)	0.8803(9)	0.051(5)	0.043(5)	0.050(5)	–0.010(4)	0.001(4)	0.004(4)
C(8)	4*e*	0.7383(7)	0.5728(4)	0.751(1)	0.064(6)	0.033(5)	0.059(6)	–0.004(4)	0.001(5)	–0.002(4)
C(9)	4*e*	0.7861(7)	0.6033(5)	0.6507(9)	0.051(5)	0.043(5)	0.039(5)	–0.001(4)	–0.000(4)	–0.002(4)
C(10)	4*e*	0.8362(6)	0.6711(4)	0.6812(9)	0.047(5)	0.038(4)	0.042(5)	0.005(4)	0.011(4)	–0.001(3)
C(11)	4*e*	0.8834(6)	0.7119(5)	0.5734(9)	0.045(5)	0.055(5)	0.038(5)	–0.009(4)	0.012(4)	–0.009(4)
N(12)	4*e*	0.8324(5)	0.7702(4)	0.5249(7)	0.048(4)	0.047(4)	0.042(4)	–0.008(3)	0.012(3)	–0.003(3)
C(13)	4*e*	0.8647(8)	0.8093(5)	0.4236(9)	0.073(6)	0.059(6)	0.042(5)	–0.019(5)	0.009(5)	0.005(5)
C(14)	4*e*	0.810(1)	0.8729(6)	0.373(1)	0.13(1)	0.072(8)	0.053(6)	–0.030(7)	0.013(7)	0.014(6)
C(15)	4*e*	0.839(1)	0.9142(7)	0.269(1)	0.21(2)	0.069(8)	0.059(8)	–0.06(1)	0.01(1)	0.008(7)
C(16)	4*e*	0.922(2)	0.8924(9)	0.213(2)	0.25(2)	0.12(1)	0.055(9)	–0.12(2)	0.05(1)	–0.018(9)
C(17)	4*e*	0.978(1)	0.8314(9)	0.256(1)	0.14(1)	0.16(1)	0.054(7)	–0.11(1)	0.047(8)	–0.044(9)
C(18)	4*e*	0.9492(9)	0.7881(7)	0.368(1)	0.075(7)	0.087(8)	0.051(6)	–0.033(6)	0.017(6)	–0.012(6)
C(19)	4*e*	1.0043(8)	0.7258(7)	0.423(1)	0.051(6)	0.12(1)	0.066(7)	–0.026(6)	0.031(5)	–0.028(7)
C(20)	4*e*	0.9742(7)	0.6842(6)	0.530(1)	0.051(5)	0.074(7)	0.061(6)	0.004(5)	0.008(5)	–0.028(5)
C(21)	4*e*	0.7814(8)	0.5649(5)	0.512(1)	0.085(7)	0.051(5)	0.063(6)	0.003(5)	0.006(6)	–0.016(5)
C(22)	4*e*	1.0346(8)	0.6196(6)	0.598(1)	0.062(7)	0.11(1)	0.100(9)	0.039(7)	0.012(7)	–0.032(8)
O(23)	4*e*	1.0324(8)	0.4644(7)	0.848(2)	0.069(7)	0.17(1)	0.24(2)	–0.020(7)	–0.023(8)	0.10(1)
C(24)	4*e*	0.981(1)	0.4636(8)	0.970(2)	0.09(1)	0.08(1)	0.20(2)	–0.022(8)	–0.06(1)	–0.00(1)

References

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