Crystal structure of trichloro(3,3′)-trimethylene-2,2′-biquinoline)gold(III) chloroform hemisolvate, $[(C_9H_5N)(CH_3)][(C_9H_6N)(CH_3)]AuCl_3 \cdot 0.5CHCl_3$ and of tribromo(3,3′)-trimethylene-2,2′-biquinoline)gold(III) chloroform hemisolvate, $[(C_9H_5N)(CH_3)][(C_9H_6N)(CH_3)]AuBr_3 \cdot 0.5CHCl_3$

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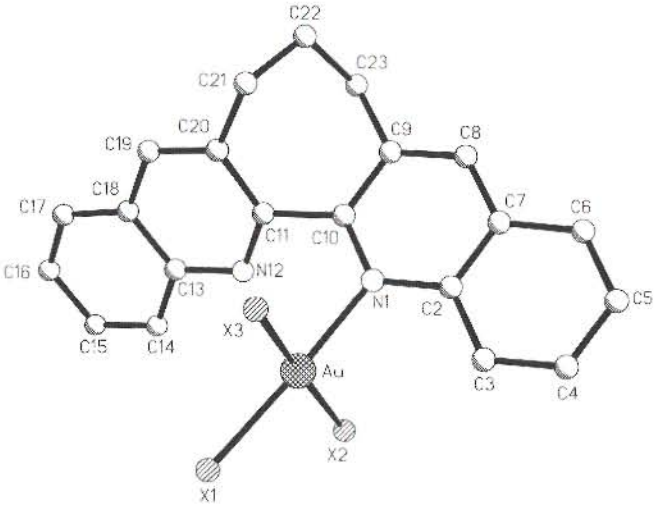

Abstract

$C_{21.5}H_{16.5}AuCl_{4.5}N_2$, monoclinic, $P12_1/n1$ (No. 14), $a = 9.777(2)$ Å, $b = 18.734(4)$ Å, $c = 12.339(2)$ Å, $\beta = 101.87(3)°$, $V = 2211.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.034$, $wR(F) = 0.031$, $T = 293$ K.

$C_{21.5}H_{16.5}AuBr_3Cl_{1.5}N_2$, monoclinic, $P12_1/n1$ (No. 14), $a = 9.775(2)$ Å, $b = 19.063(4)$ Å, $c = 12.621(3)$ Å, $\beta = 103.07(3)°$, $V = 2290.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.056$, $wR(F) = 0.044$, $T = 293$ K.

Source of material

The compounds were synthesized by addition of a suspension of 3,3′-trimethylene-2,2′-biquinoline to a solution of $KAuX_4$, in methanol (X = Cl, Br). Potassium halogenide was separated by filtration, the red solution was reduced to a small volume, and the product was recrystallized from chloroform [1].

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Discussion

Instead of the expected fivefold coordinated gold(III) the reaction yielded square planar trihalogeno(3,3′-trimethylene-2,2′-biquinolino-κ*N*)gold(III)complexes. Bond lengths Au—N1, Au—Cl1, Au—Cl2, Au—Cl3 are 205.9 pm, 227.2 pm, 228.7 pm, 228.2 pm, respectively, and Au—N1, Au—Br1, Au—Br2, Au—Br3 are 207.1 pm, 239.8 pm, 243.1 pm, 242.6 pm, respectively. The *trans* effect is more pronounced in the bromide. The plane of the κ*N*-bonded chinoline fragment is almost perpendicular to that of AuX_3N. The twist angle around C10–C11 is about 35°. The chloroform solvent molecules are placed in cavities without any interactions to Au. The trimethylene bridge between the two aromatic fragments gives rise to remarkable differences in the C—C bond lengths (131 pm to 142 pm). The C—N bonds are not affected by the Au—N interaction. In the crystal the CHcl3 molecule is slightly disordered. Hence the corresponding H atoms could not be localized.

1. Trichloro(3,3′)-trimethylene-2,2′-biquinoline)gold(III) chloroform hemisolvate, $[(C_9H_5N)(CH_3)][(C_9H_6N)(CH_3)]AuCl_3 \cdot 0.5CHCl_3$

Table 1. Data collection and handling.

Crystal:	yellow plate, size 0.45 × 0.45 × 0.05 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	72.10 cm^{-1}
Diffractometer, scan mode:	Siemens R3m/V, Wyckoff
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5129, 4762
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 4020
$N(param)_{refined}$:	261
Program:	SHELXTL-plus [2]

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(3)	4*e*		–0.0237(6)	0.2175(4)	0.2245(5)	0.08
H(4)	4*e*		–0.1957(7)	0.2696(4)	0.3046(5)	0.08
H(5)	4*e*		–0.1634(7)	0.3838(4)	0.3831(5)	0.08
H(6)	4*e*		0.0309(7)	0.4508(4)	0.3704(5)	0.08

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(8)	4e		0.2624(7)	0.4591(3)	0.3122(5)	0.08
H(14)	4e		0.5531(7)	0.0819(4)	0.2322(6)	0.08
H(15)	4e		0.7374(7)	0.0201(5)	0.1800(7)	0.08
H(16)	4e		0.8761(7)	0.0755(4)	0.0694(7)	0.08
H(17)	4e		0.8259(6)	0.1893(4)	0.0046(6)	0.08
H(19)	4e		0.6695(6)	0.2997(4)	–0.0073(5)	0.08
H(21A)	4e		0.5059(7)	0.3961(4)	–0.0273(5)	0.08
H(21B)	4e		0.3626(7)	0.3836(4)	0.0080(5)	0.08

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(22A)	4e		0.4373(7)	0.4821(4)	0.0975(6)	0.08
H(22B)	4e		0.5945(7)	0.4586(4)	0.1207(6)	0.08
H(23A)	4e		0.5647(6)	0.3869(4)	0.2635(5)	0.08
H(23B)	4e		0.4972(6)	0.4606(4)	0.2821(5)	0.08
Cl(4)	4e	0.5	–0.0476(5)	0.5922(3)	0.0040(4)	0.096(1)
Cl(5)	4e	0.5	–0.1207(7)	0.4552(4)	0.0630(6)	0.125(2)
Cl(6)	4e	0.5	–0.1234(9)	0.5252(5)	0.0352(8)	0.172(3)
C	4e	0.5	0.033(2)	0.499(1)	0.042(2)	0.109(7)

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	4e	0.18452(2)	0.17563(1)	0.12656(2)	0.0367(1)	0.0271(1)	0.0359(1)	–0.0018(1)	0.01515(7)	–0.0023(1)
Cl(1)	4e	0.1570(2)	0.06898(8)	0.0370(1)	0.070(1)	0.0323(8)	0.0556(9)	–0.0073(7)	0.0239(8)	–0.0108(7)
Cl(2)	4e	0.2200(2)	0.11803(9)	0.2934(1)	0.081(1)	0.045(1)	0.0470(8)	0.0038(9)	0.0224(8)	0.0109(7)
Cl(3)	4e	0.1259(2)	0.23422(9)	–0.0386(1)	0.067(1)	0.0459(9)	0.0395(7)	0.0030(8)	0.0142(7)	0.0037(7)
N(1)	4e	0.2161(4)	0.2741(2)	0.2017(4)	0.035(2)	0.028(2)	0.036(2)	–0.002(2)	0.011(2)	–0.000(2)
C(2)	4e	0.1116(5)	0.3042(3)	0.2481(4)	0.036(3)	0.031(3)	0.033(3)	0.006(2)	0.009(2)	–0.002(2)
C(3)	4e	–0.0106(6)	0.2650(4)	0.2538(5)	0.042(3)	0.044(4)	0.045(3)	–0.004(3)	0.016(3)	–0.008(3)
C(4)	4e	–0.1111(7)	0.2951(4)	0.3024(5)	0.045(3)	0.068(5)	0.048(4)	0.007(3)	0.019(3)	–0.010(3)
C(5)	4e	–0.0939(7)	0.3645(4)	0.3466(5)	0.048(4)	0.073(5)	0.048(4)	0.017(4)	0.019(3)	–0.006(4)
C(6)	4e	0.0211(7)	0.4033(4)	0.3408(5)	0.061(4)	0.046(4)	0.049(3)	0.023(3)	0.015(3)	–0.008(3)
C(7)	4e	0.1280(6)	0.3744(3)	0.2903(4)	0.044(3)	0.045(4)	0.035(3)	0.012(3)	0.009(2)	–0.005(3)
C(8)	4e	0.2501(7)	0.4111(3)	0.2847(5)	0.065(4)	0.029(3)	0.044(3)	0.003(3)	0.005(3)	–0.002(3)
C(9)	4e	0.3520(6)	0.3811(3)	0.2380(4)	0.042(3)	0.029(3)	0.039(3)	–0.002(2)	0.011(2)	–0.002(2)
C(10)	4e	0.3322(6)	0.3103(3)	0.1967(5)	0.041(3)	0.030(3)	0.038(3)	–0.001(2)	0.009(2)	–0.000(2)
C(11)	4e	0.4436(5)	0.2741(3)	0.1489(4)	0.034(3)	0.032(3)	0.038(3)	–0.004(2)	0.010(2)	–0.002(2)
N(12)	4e	0.4707(4)	0.2085(3)	0.1827(4)	0.033(2)	0.033(3)	0.052(3)	–0.000(2)	0.015(2)	–0.001(2)
C(13)	4e	0.5784(6)	0.1738(3)	0.1481(5)	0.033(3)	0.043(4)	0.050(3)	–0.001(3)	0.013(2)	–0.011(3)
C(14)	4e	0.6080(7)	0.1043(4)	0.1856(6)	0.053(4)	0.040(4)	0.084(5)	0.005(3)	0.020(4)	0.004(4)
C(15)	4e	0.7176(7)	0.0683(5)	0.1553(7)	0.056(4)	0.052(5)	0.097(6)	0.014(3)	0.014(4)	–0.014(4)
C(16)	4e	0.7989(7)	0.1010(4)	0.0881(7)	0.039(3)	0.058(5)	0.091(5)	0.005(3)	0.016(4)	–0.020(4)
C(17)	4e	0.7706(6)	0.1676(4)	0.0515(6)	0.039(3)	0.063(5)	0.060(4)	–0.010(3)	0.020(3)	–0.023(4)
C(18)	4e	0.6579(5)	0.2070(4)	0.0797(5)	0.032(3)	0.045(4)	0.042(3)	–0.004(3)	0.007(2)	–0.012(3)
C(19)	4e	0.6193(6)	0.2767(4)	0.0421(5)	0.039(3)	0.052(4)	0.042(3)	–0.013(3)	0.018(3)	–0.007(3)
C(20)	4e	0.5123(6)	0.3114(3)	0.0750(5)	0.044(3)	0.038(4)	0.036(3)	–0.003(2)	0.007(2)	–0.003(2)
C(21)	4e	0.4622(7)	0.3846(4)	0.0335(5)	0.066(4)	0.049(4)	0.049(3)	0.001(3)	0.018(3)	0.011(3)
C(22)	4e	0.5005(7)	0.4430(4)	0.1190(6)	0.067(4)	0.040(4)	0.065(4)	–0.009(3)	0.021(4)	0.008(3)
C(23)	4e	0.4892(6)	0.4190(4)	0.2357(5)	0.049(3)	0.036(3)	0.062(4)	–0.011(3)	0.011(3)	–0.005(3)

2. Tribromo(3,3′)-trimethylene-2,2′-biquinoline)gold(III) chloroform hemisolvate, $[(C_9H_5N)(CH_3)][(C_9H_6N)(CH_3)]AuBr_3 \cdot 0.5CHCl_3$

Table 4. Data collection and handling.

Crystal:	red-brownish prism, size 0.2 × 0.2 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	118.40 cm^{-1}
Diffractometer, scan mode:	Siemens R3m/V, Wyckoff
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5723, 5226
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 3631
$N(param_{refined}$:	261
Program:	SHELXTL-plus [2]

Table 5. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(3)	4e		–0.024(2)	0.2246(8)	0.216(1)	0.08
H(4)	4e		–0.189(2)	0.2778(8)	0.297(1)	0.08
H(5)	4e		–0.155(2)	0.3888(9)	0.373(1)	0.08
H(6)	4e		0.047(2)	0.4519(8)	0.366(1)	0.08
H(8)	4e		0.270(2)	0.4586(7)	0.308(1)	0.08
H(14)	4e		0.552(2)	0.0868(8)	0.236(2)	0.08
H(15)	4e		0.727(2)	0.022(1)	0.186(2)	0.08
H(16)	4e		0.870(2)	0.074(1)	0.080(2)	0.08
H(17)	4e		0.818(2)	0.185(1)	0.005(1)	0.08
H(19)	4e		0.662(2)	0.2953(8)	–0.010(1)	0.08
H(21A)	4e		0.357(2)	0.3806(8)	0.006(1)	0.08
H(21B)	4e		0.496(2)	0.3918(8)	–0.034(1)	0.08
H(22A)	4e		0.595(2)	0.4558(8)	0.112(1)	0.08
H(22B)	4e		0.438(2)	0.4805(8)	0.090(1)	0.08
H(23A)	4e		0.501(2)	0.4600(8)	0.274(1)	0.08
H(23B)	4e		0.569(2)	0.3875(8)	0.258(1)	0.08
Cl(1)	4e	0.5	–0.058(1)	0.5833(7)	0.006(1)	0.106(4)
Cl(2)	4e	0.5	–0.108(3)	0.507(2)	0.053(2)	0.16(1)
Cl(3)	4e	0.5	–0.087(2)	0.433(1)	0.060(2)	0.308(7)
C	4e	0.5	0.041(5)	0.497(3)	0.040(3)	0.11(1)

Table 6. 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*	0.18155(6)	0.18006(3)	0.12223(4)	0.0376(3)	0.0295(2)	0.0339(3)	–0.0016(3)	0.0132(2)	–0.0020(3)
Br(1)	4*e*	0.1532(2)	0.06914(7)	0.0302(1)	0.075(1)	0.0349(8)	0.052(1)	–0.0073(8)	0.0225(9)	–0.0092(7)
Br(2)	4*e*	0.2217(2)	0.12014(8)	0.2966(1)	0.082(1)	0.0516(9)	0.0431(9)	0.0072(9)	0.0212(9)	0.0108(8)
Br(3)	4*e*	0.1119(2)	0.24048(8)	–0.0508(1)	0.064(1)	0.0492(9)	0.0368(9)	0.0006(8)	0.0114(8)	0.0043(7)
N(1)	4*e*	0.214(1)	0.2780(5)	0.1946(8)	0.029(6)	0.026(5)	0.033(6)	0.004(5)	0.012(5)	–0.003(5)
C(2)	4*e*	0.113(1)	0.3072(6)	0.242(1)	0.035(8)	0.045(8)	0.029(7)	0.006(6)	0.010(6)	–0.002(6)
C(3)	4*e*	–0.008(2)	0.2707(8)	0.247(1)	0.042(9)	0.046(9)	0.06(1)	–0.007(8)	0.012(8)	–0.007(8)
C(4)	4*e*	–0.105(2)	0.3030(8)	0.294(1)	0.041(9)	0.07(1)	0.05(1)	–0.003(8)	0.020(8)	–0.012(9)
C(5)	4*e*	–0.086(2)	0.3689(9)	0.339(1)	0.06(1)	0.07(1)	0.05(1)	0.03(1)	0.024(9)	–0.010(9)
C(6)	4*e*	0.033(2)	0.4060(8)	0.334(1)	0.07(1)	0.056(9)	0.038(9)	0.023(9)	0.007(8)	–0.006(8)
C(7)	4*e*	0.134(2)	0.3766(7)	0.285(1)	0.046(9)	0.036(8)	0.038(8)	0.015(7)	0.008(7)	–0.000(7)
C(8)	4*e*	0.255(2)	0.4119(7)	0.279(1)	0.06(1)	0.029(7)	0.038(8)	0.001(7)	–0.006(7)	–0.003(6)
C(9)	4*e*	0.357(1)	0.3824(7)	0.232(1)	0.041(9)	0.036(8)	0.029(7)	–0.006(7)	0.000(6)	0.000(6)
C(10)	4*e*	0.332(1)	0.3120(6)	0.190(1)	0.037(8)	0.033(7)	0.032(7)	–0.002(6)	0.004(6)	0.004(6)
C(11)	4*e*	0.442(1)	0.2754(7)	0.144(1)	0.032(8)	0.035(7)	0.038(8)	–0.003(6)	0.006(6)	–0.013(6)
N(12)	4*e*	0.469(1)	0.2120(5)	0.1811(9)	0.036(7)	0.034(6)	0.046(7)	–0.001(5)	0.010(6)	–0.001(5)
C(13)	4*e*	0.575(2)	0.1748(8)	0.150(1)	0.041(9)	0.053(9)	0.06(1)	–0.004(8)	0.020(8)	–0.020(9)
C(14)	4*e*	0.606(2)	0.1074(8)	0.190(2)	0.06(1)	0.040(9)	0.10(1)	0.003(9)	0.03(1)	0.02(1)
C(15)	4*e*	0.709(2)	0.070(1)	0.160(2)	0.07(2)	0.05(1)	0.11(2)	0.01(1)	0.02(1)	–0.01(1)
C(16)	4*e*	0.793(2)	0.101(1)	0.095(2)	0.04(1)	0.07(1)	0.09(1)	0.01(1)	0.02(1)	–0.04(1)
C(17)	4*e*	0.763(2)	0.166(1)	0.052(1)	0.03(1)	0.09(1)	0.07(1)	–0.01(1)	0.014(9)	–0.04(1)
C(18)	4*e*	0.654(2)	0.2077(8)	0.080(1)	0.045(9)	0.043(8)	0.047(8)	–0.007(7)	0.022(7)	–0.012(7)
C(19)	4*e*	0.614(2)	0.2743(8)	0.040(1)	0.044(9)	0.06(1)	0.038(9)	–0.020(8)	0.015(7)	–0.005(8)
C(20)	4*e*	0.508(2)	0.3107(7)	0.071(1)	0.043(9)	0.035(8)	0.045(8)	0.002(7)	0.012(7)	0.002(7)
C(21)	4*e*	0.458(2)	0.3819(8)	0.028(1)	0.06(1)	0.05(1)	0.052(9)	–0.004(9)	0.010(8)	0.008(8)
C(22)	4*e*	0.501(2)	0.4417(8)	0.112(1)	0.06(1)	0.06(1)	0.07(1)	–0.015(9)	0.01(1)	0.017(9)
C(23)	4*e*	0.493(2)	0.4186(8)	0.229(1)	0.06(1)	0.047(9)	0.043(9)	–0.017(8)	0.003(8)	–0.009(8)

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