Crystal structure of pyridinogold(III) bromide, $[AuBr_2(C_5H_5N)_2]^+[AuBr_4]^-[AuBr_3(C_5H_5N)]_2$, *a frozen-in autoionization system*

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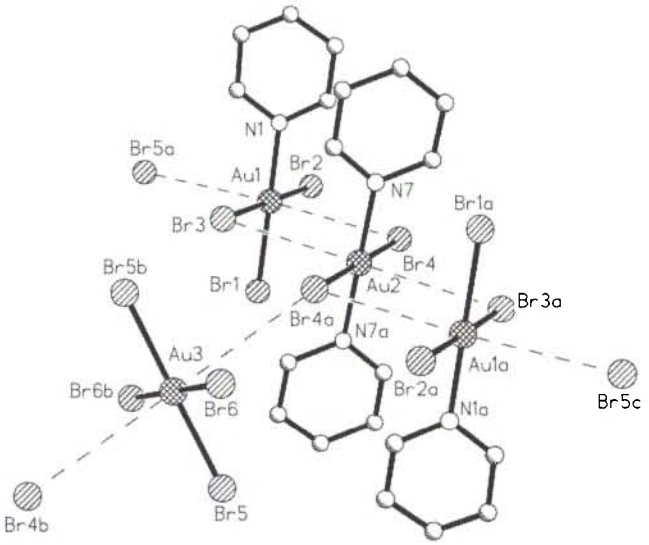

These square planar complexes in the ratio 1:1:2 organize an arrangement which complements each square by two bromine atoms from neighbored complexes; (d(Au—Br) = 354 pm – 386 pm) [2]. Including these links a 3D framework is formed: $^3_\infty\{[AuBr^t_2Br^t_{2/2}Br^t_{2/3}]^-AuBr^t_{2/2}Br^t_{2/3}N^t_2]^+ [AuBr^c_{2/2}(Br_{1/3}N)^c]_2\}$ (t = *trans*, c = *cis*).

Table 1. Data collection and handling.

Crystal:	scarlet plate, size 0.09 × 0.18 × 0.08 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	276.80 cm^{-1}
Diffractometer, scan mode:	Siemens R3m/V, Wyckoff
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4801, 4420
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 3274
$N(param)_{refined}$:	185
Program:	SHELXTL-plus [3]

Abstract

$C_{10}H_{10}Au_2Br_6N_2$, monoclinic, $P12_1/n1$ (No. 14), a = 11.395(2) Å, b = 15.437(2) Å, c = 11.091(2) Å, β = 99.44(1)°, V = 1924.5 $Å^3$, Z = 4, $R_{gt}(F)$ = 0.063, $wR(F)$ = 0.050, T = 293 K.

Source of material

Equimolar methanolic solutions of $KAuBr_4$ and pyridine were added slowly under constant stirring. After refluxing (30 min) the solvent was removed in vacuum to give a fine brownish red solid, and scarlet crystals grown from chloroform [1].

Dicussion

In contrast to the corresponding chloride $AuCl_3py$ [2] the present crystal structure shows a unique arrangement of three different square planar Au(III) species, which represents a frozen-in autoionization system. Preserving the stoichiometric ratio $AuBr_3py$, the reaction obviously results in the simultaneous formation of cationic, anionic and neutral complexes, namely $[AuBr_2(py)_2]^+$ (d(Au—Br) = 243.3 pm; d(Au—N) = 201 pm), $[AuBr_4]^-$ (d(Au—Br) = 241.2 pm; 242.0 pm) and $AuBr_3py$ (d(Au—Br) = 241.2 pm, 238.6 pm and 242.1 pm; d(Au—N) = 204 pm).

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.229(2)	0.189(1)	0.407(2)	0.08
H(3)	4*e*	0.170(2)	0.291(1)	0.545(2)	0.08
H(4)	4*e*	0.249(2)	0.426(1)	0.552(2)	0.08
H(5)	4*e*	0.372(2)	0.468(1)	0.421(2)	0.08
H(6)	4*e*	0.439(2)	0.360(1)	0.295(2)	0.08
H(8)	4*e*	0.311(2)	0.002(1)	0.663(2)	0.08
H(9)	4*e*	0.241(2)	0.095(1)	0.801(2)	0.08
H(10)	4*e*	0.338(2)	0.226(1)	0.846(2)	0.08
H(11)	4*e*	0.504(2)	0.266(1)	0.751(2)	0.08
H(12)	4*e*	0.559(2)	0.1728(9)	0.606(2)	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(1)	4*e*	0.39520(6)	0.17763(4)	0.22359(6)	0.0513(4)	0.0328(3)	0.0342(3)	0.0036(3)	0.0134(3)	0.0002(3)
Au(2)	2*c*	1/2	0	1/2	0.0353(5)	0.0302(4)	0.0367(5)	–0.0017(4)	0.0081(4)	–0.0013(4)
Au(3)	2*b*	1/2	1/2	0	0.0441(6)	0.0320(4)	0.0437(5)	0.0030(4)	0.0122(4)	–0.0023(4)
Br(1)	4*e*	0.4639(2)	0.0744(1)	0.0909(2)	0.069(1)	0.055(1)	0.054(1)	0.0076(9)	0.022(1)	–0.0160(9)
Br(2)	4*e*	0.1998(2)	0.1710(1)	0.1015(2)	0.058(1)	0.062(1)	0.061(1)	0.015(1)	–0.004(1)	–0.003(1)
Br(3)	4*e*	0.5925(2)	0.1838(1)	0.3439(2)	0.056(1)	0.055(1)	0.059(1)	–0.0074(9)	0.003(1)	–0.001(1)
Br(4)	4*e*	0.3007(2)	–0.0010(1)	0.3832(2)	0.041(1)	0.0529(9)	0.050(1)	–0.0027(9)	0.0006(8)	0.0004(9)
Br(5)	4*e*	0.4878(3)	0.3479(1)	0.0461(2)	0.134(2)	0.0342(9)	0.064(1)	–0.001(1)	0.035(1)	0.0004(9)
Br(6)	4*e*	0.5382(2)	0.5361(1)	0.2153(2)	0.089(2)	0.051(1)	0.046(1)	0.002(1)	0.011(1)	–0.0071(9)
N(1)	4*e*	0.339(1)	0.2662(8)	0.338(1)	0.051(9)	0.048(8)	0.024(7)	0.001(7)	0.018(6)	–0.005(6)
C(2)	4*e*	0.261(2)	0.247(1)	0.411(2)	0.06(1)	0.05(1)	0.05(1)	0.011(9)	0.02(1)	–0.002(9)
C(3)	4*e*	0.224(2)	0.306(1)	0.491(2)	0.05(1)	0.09(1)	0.06(1)	0.02(1)	0.04(1)	–0.02(1)
C(4)	4*e*	0.271(2)	0.385(1)	0.494(2)	0.08(2)	0.06(1)	0.05(1)	0.02(1)	0.00(1)	–0.02(1)
C(5)	4*e*	0.344(2)	0.409(1)	0.421(2)	0.11(2)	0.023(8)	0.08(1)	0.01(1)	0.02(1)	–0.004(9)
C(6)	4*e*	0.380(2)	0.346(1)	0.346(2)	0.09(2)	0.05(1)	0.05(1)	–0.00(1)	0.04(1)	–0.006(8)
N(7)	4*e*	0.445(1)	0.0795(7)	0.623(1)	0.053(9)	0.028(6)	0.035(8)	–0.003(6)	0.009(7)	–0.004(5)
C(8)	4*e*	0.349(2)	0.056(1)	0.683(2)	0.05(1)	0.034(8)	0.06(1)	0.005(8)	0.012(9)	0.007(8)
C(9)	4*e*	0.308(2)	0.111(1)	0.763(2)	0.07(1)	0.06(1)	0.05(1)	–0.00(1)	0.04(1)	–0.003(9)
C(10)	4*e*	0.364(2)	0.188(1)	0.788(2)	0.07(1)	0.06(1)	0.06(1)	0.01(1)	0.04(1)	–0.023(9)
C(11)	4*e*	0.463(2)	0.212(1)	0.732(2)	0.06(1)	0.05(1)	0.07(1)	–0.009(9)	0.00(1)	–0.022(9)
C(12)	4*e*	0.495(2)	0.1564(9)	0.648(2)	0.04(1)	0.035(8)	0.06(1)	–0.014(7)	0.013(9)	–0.013(8)

References

1. Schmidt, R.; Binder, H.: Z. Anorg. Allg. Chem., in preparation.
2. Adam, H.-N.; Strähle, J.: Darstellung und Struktur von [Hpy][$AuCl_4$], $AuCl_3$py, [$AuCl_2(py)_2$]Cl · H_2O und [$AuCl_2(py)_2$][$AuCl_2$]. Z. Anorg. Allg. Chem. **485** (1982) 65-80.
3. Sheldrick, G. M.: Program Package SHELXTL-Plus. Release 4.1, Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.