This work was supported by a grant for key research projects from the State Science and Technology Commission and National Nature Science Foundation of China.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1025). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(3,3′-dimethylene-2,2′-biquinolinium) Tetrabromoaurate(III) Dibromoaurate (I)

R. F. Baggio*

División Física del Sólido, Departamento de Física,
Comisión Nacional de Energía Atómica,
Av. del Libertador 8250, 1429 Buenos Aires, Argentina

S. A. Moya, R. Schmied, M. Gulppi and J. Guerrero

Facultad de Ciencia, Departamento de Química,
Universidad de Santiago de Chile, Santiago de Chile,
Chile

R. Sartori

Facultad de Ciencias Medicina,
Departamento de Bioquímica, Universidad de Chile,
Santiago de Chile, Chile

M. T. Garland

Facultad de Ciencias Físicas y Matemáticas,
Departamento de Física, Universidad de Chile,
Santiago de Chile, Chile

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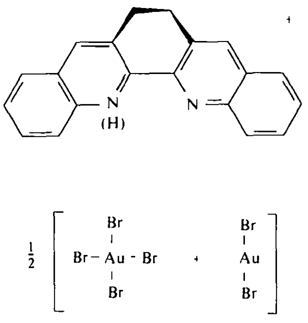
Abstract

The structure of the title compound, $(C_{20}H_{15}N_2)[AuBr_3]$, has been determined. It consists of two anions, $[AuBr_4]^-$ and $[AuBr_2]^-$, attached to two 3,3′-dimethylene-2,2′-biquinolinium (or 6,7-dihydro-benzo[2,1-*b*:3,4-*b*′]diquinolium) cations by hydrogen bonds.

Comment

For some time, gold chemistry has received great attention, mainly because of the specific properties and structure of its compounds (Schmidbaur, 1990). It is known that the reaction of Au^{III} with bidentate nitrogen-containing ligands produced either tetra- or pentacoordinated auriferous complexes of Au^{III} having distorted square-planar, tetrahedral, square-pyramidal or bipyramidal coordination (O'Connor & Sinn, 1978; Robinson & Sinn, 1975; Chalton, Harris, Petit & Stephenson, 1966). However, reactions carried out under special conditions using similar bidentate nitrogen ligands can generate gold compounds having an ionic structure different from the classic complex one. In the title ionic structure, (I), the protonated nitrogen-containing ligand plays the role of the cation while the two associated anions are tetrabromoaurate(III) and dibromoaurate(I). Though the mechanism of the reaction is not yet clear, the origin of the Au^I species should be found in the oxidation of the solvent by the starting Au^{III} compound. In this work we report the structure of $(C_{20}H_{15}N_2)[AuBr_3]$ obtained from the reaction of potassium tetrabromoaurate(III) and 3,3′-dimethylene-2,2′-biquinoline under refluxing methanol conditions.



(I)

The structure of the title compound consists of discrete $[AuBr_4]^-$ and $[AuBr_2]$ units, with the gold ions located at inversion centers, and substituted biquinolinium cations as counterions (Fig. 1). The $[AuBr_4]^-$ unit has regular square-planar geometry with *trans* angles of 90.0° and Au—Br distances of 2.420 (1) and 2.416 (1) Å. The $[AuBr_2]^-$ unit is strictly linear with an Au—Br distance of 2.386 (1) Å. The two 'wings' of the organic cation are effectively planar and are twisted around the C(19)—C(20) bond by equal amounts,which is seen in the two identical torsion angles C(12)—C(11)—C(10)—C(9) = C(7)—C(8)—C(9)—C(10) = −153.8°.

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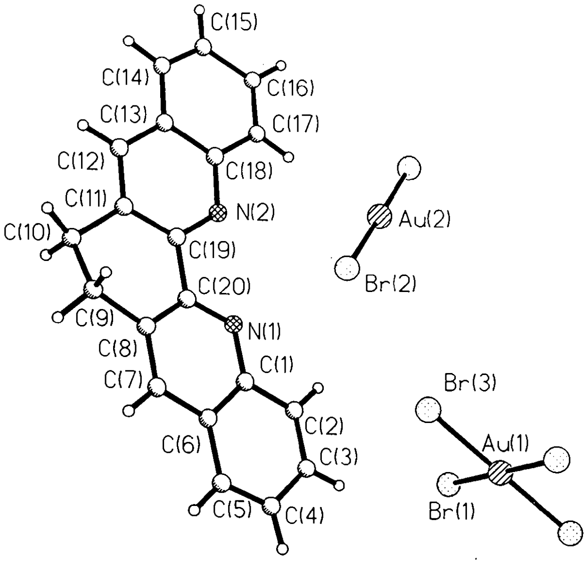

Fig. 1. The structural units and numbering scheme of the title compound.

The anionic character of the two gold–bromine units requires the positive charge of a proton attached to the organic moiety to balance it. The environments of the two N atoms in the cation are disimilar, with a strong electron-density concentration near N(2) at a distance of about 0.7 Å. This is interpreted as the localization of an N—H$^+$ bond on this N atom.

Besides the ionic coulombic interaction there are two short contacts [Br(2)···H(17) = 2.991 and Br(1)···H(12)(1–*x*, –*y*, –*z*) = 2.997 Å] which contribute to the cohesion of the title structure (Fig. 2).

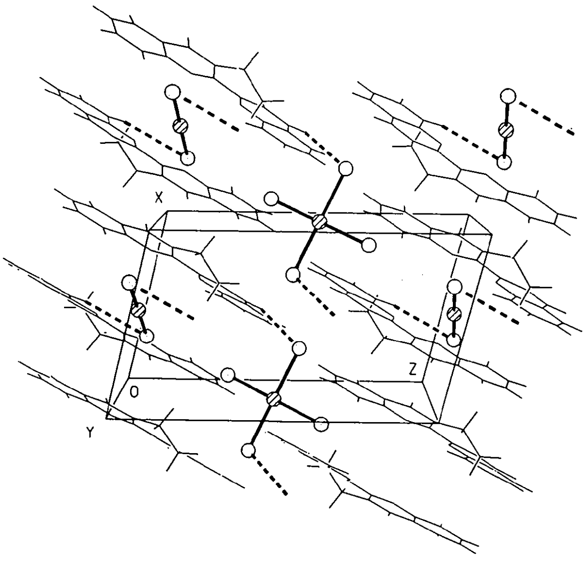

Fig. 2. The crystal packing viewed along *b* showing the hydrogen bonds.

Experimental

Crystal data

$(C_{20}H_{15}N_2)[AuBr_3]$
$M_r = 720.0$
Triclinic
$P\bar{1}$
$a = 7.957$ (1) Å
$b = 9.488$ (1) Å
$c = 13.312$ (3) Å
$\alpha = 89.68$ (1)°
$\beta = 76.86$ (1)°
$\gamma = 81.17$ (1)°
$V = 966.5$ (3) Å^3
$Z = 2$
$D_x = 2.471$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 13.8$ mm^{-1}
$T = 293$ K
Polyhedron
0.50 × 0.21 × 0.16 mm
Reddish

Data collection

Siemens *R*3*m* diffractometer
θ–2θ scans (4.19–29.3° min^{-1})
Absorption correction: empirical
$T_{\min} = 0.082$, $T_{\max} = 0.138$
3204 measured reflections
2547 independent reflections
2168 observed reflections [$F > 4\sigma(F)$]

$R_{\rm int} = 0.0152$
$\theta_{\max} = 22.5°$
$h = -1 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$
2 standard reflections monitored every 48 reflections
intensity variation: none

Refinement

Refinement on *F*
$R = 0.0455$
$wR = 0.0595$
$S = 0.80$
2168 reflections
238 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F) + 0.005F^2]$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 1.73$ e Å^{-3}
$\Delta\rho_{\min} = -0.95$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
Au(1)	0	1/2	1/2	0.063 (1)
Au(2)	1/2	1/2	0	0.070 (1)
Br(1)	0.2817 (1)	0.4314 (1)	0.5412 (1)	0.077 (1)
Br(2)	0.3195 (2)	0.3188 (1)	0.0384 (1)	0.080 (1)
Br(3)	0.1097 (2)	0.3776 (2)	0.3339 (1)	0.095 (1)
C(1)	0.1327 (13)	−0.0722 (10)	0.1139 (8)	0.067 (4)
C(2)	0.1044 (15)	0.0216 (11)	0.2014 (9)	0.074 (4)
C(3)	0.0113 (15)	−0.0111 (12)	0.2928 (8)	0.076 (4)
C(4)	−0.0628 (16)	−0.1392 (13)	0.3072 (10)	0.083 (5)
C(5)	−0.0343 (15)	−0.2337 (13)	0.2283 (9)	0.079 (4)
C(6)	0.0595 (14)	−0.2012 (11)	0.1280 (9)	0.070 (4)
C(7)	0.0866 (14)	−0.2913 (10)	0.0390 (9)	0.073 (4)
C(8)	0.1799 (13)	−0.2561 (10)	−0.0540 (8)	0.065 (4)
C(9)	0.2014 (15)	−0.3438 (12)	−0.1508 (8)	0.074 (4)
C(10)	0.3684 (16)	−0.3295 (11)	−0.2319 (9)	0.082 (5)
C(11)	0.4065 (14)	−0.1792 (10)	−0.2404 (8)	0.067 (4)
C(12)	0.5022 (14)	−0.1293 (10)	−0.3300 (8)	0.069 (4)
C(13)	0.5436 (14)	0.0075 (12)	−0.3355 (8)	0.074 (4)
C(14)	0.6401 (15)	0.0640 (12)	−0.4261 (8)	0.076 (4)
C(15)	0.6790 (16)	0.1924 (14)	−0.4253 (9)	0.087 (5)
C(16)	0.6265 (17)	0.2852 (12)	−0.3348 (9)	0.083 (5)
C(17)	0.5369 (14)	0.2344 (10)	−0.2467 (8)	0.070 (4)

C(18)	0.4905 (14)	0.0982 (11)	−0.2454 (7)	0.066 (4)
C(19)	0.3537 (13)	−0.0862 (10)	−0.1548 (7)	0.064 (4)
C(20)	0.2483 (14)	−0.1288 (10)	−0.0563 (7)	0.065 (4)
N(1)	0.2276 (11)	−0.0389 (8)	0.0221 (6)	0.064 (3)
N(2)	0.3995 (11)	0.0437 (9)	−0.1578 (6)	0.065 (3)

Table 2. *Selected geometric parameters* (Å, °)

Au(1)—Br(1)	2.420 (1)	Au(1)—Br(3)	2.416 (1)
Au(1)—Br(1*A*)	2.420 (1)	Au(1)—Br(3*A*)	2.416 (1)
Au(2)—Br(2)	2.386 (1)	Au(2)—Br(2*A*)	2.386 (1)
C(1)—C(2)	1.424 (15)	C(1)—C(6)	1.428 (15)
C(1)—N(1)	1.346 (12)	C(2)—C(3)	1.335 (15)
C(3)—C(4)	1.424 (18)	C(4)—C(5)	1.341 (18)
C(5)—C(6)	1.431 (15)	C(6)—C(7)	1.421 (16)
C(7)—C(8)	1.359 (14)	C(8)—C(9)	1.501 (15)
C(8)—C(20)	1.396 (15)	C(9)—C(10)	1.533 (15)
C(10)—C(11)	1.501 (15)	C(11)—C(12)	1.385 (14)
C(11)—C(19)	1.390 (14)	C(12)—C(13)	1.385 (16)
C(13)—C(14)	1.425 (15)	C(13)—C(18)	1.423 (14)
C(14)—C(15)	1.303 (18)	C(15)—C(16)	1.439 (16)
C(16)—C(17)	1.354 (15)	C(17)—C(18)	1.397 (15)
C(18)—N(2)	1.369 (12)	C(19)—C(20)	1.478 (13)
C(19)—N(2)	1.336 (13)	C(20)—N(1)	1.316 (13)
Br(1)—Au(1)—Br(3)	90.0 (1)	Br(1)—Au(1)—Br(1*A*)	180.0 (1)
Br(3)—Au(1)—Br(1*A*)	90.0 (1)	Br(1)—Au(1)—Br(3*A*)	90.0 (1)
Br(3)—Au(1)—Br(3*A*)	180.0 (1)	Br(1*A*)—Au(1)—Br(3*A*)	90.0 (1)
Br(2)—Au(2)—Br(2*A*)	180.0 (1)	C(2)—C(1)—C(6)	117.8 (9)
C(2)—C(1)—N(1)	120.1 (9)	C(6)—C(1)—N(1)	122.2 (9)
C(1)—C(2)—C(3)	120.4 (10)	C(2)—C(3)—C(4)	122.0 (11)
C(3)—C(4)—C(5)	119.9 (11)	C(4)—C(5)—C(6)	120.0 (12)
C(1)—C(6)—C(5)	119.7 (10)	C(1)—C(6)—C(7)	116.6 (9)
C(5)—C(6)—C(7)	123.7 (10)	C(6)—C(7)—C(8)	121.0 (10)
C(7)—C(8)—C(9)	122.5 (10)	C(7)—C(8)—C(20)	116.4 (9)
C(9)—C(8)—C(20)	121.0 (9)	C(8)—C(9)—C(10)	113.3 (10)
C(9)—C(10)—C(11)	113.0 (8)	C(10)—C(11)—C(12)	122.5 (9)
C(10)—C(11)—C(19)	120.2 (9)	C(12)—C(11)—C(19)	117.2 (9)
C(11)—C(12)—C(13)	122.0 (9)	C(12)—C(13)—C(14)	124.0 (10)
C(12)—C(13)—C(18)	119.0 (9)	C(14)—C(13)—C(18)	117.0 (10)
C(13)—C(14)—C(15)	121.0 (10)	C(14)—C(15)—C(16)	122.7 (11)
C(15)—C(16)—C(17)	118.1 (11)	C(16)—C(17)—C(18)	120.6 (10)
C(13)—C(18)—C(17)	120.6 (9)	C(13)—C(18)—N(2)	117.1 (9)
C(17)—C(18)—N(2)	122.2 (9)	C(11)—C(19)—C(20)	121.2 (9)
C(11)—C(19)—N(2)	121.3 (9)	C(20)—C(19)—N(2)	117.5 (8)
C(8)—C(20)—C(19)	118.9 (9)	C(8)—C(20)—N(1)	126.3 (9)
C(19)—C(20)—N(1)	114.8 (9)	C(1)—N(1)—C(20)	117.5 (9)
C(18)—N(2)—C(19)	123.2 (8)		

The structure was solved by direct methods and refined by full-matrix least squares. *SHELXTL/PC* (Sheldrick, 1991) was used for all calculations and drawings.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, selected short intermolecular contacts selected bond lengths and angles, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: HU1070). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Symmetrical Tetrasubstituted Tin Compounds: Tetrakis(2-methoxyphenyl)tin and Tetrakis(2-methoxybenzyl)tin

JENNIFER-NICOLA ROSS AND JAMES L. WARDELL

Department of Chemistry, University of Aberdeen,
Meston Walk, Aberdeen AB9 2UE, Scotland

GEORGE FERGUSON

Department of Chemistry and Biochemistry,
University of Guelph, Guelph, Ontario,
Canada N1G 2W1

JOHN N. LOW

Department of Applied Physics and Electronic &
Mechanical Engineering, University of Dundee,
Dundee DD1 4HN, Scotland

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Abstract

The title compound tetrakis(2-methoxyphenyl)tin, $[Sn(C_7H_7O)_4]$, crystallizes in the triclinic space group $P\bar{1}$ with two molecules in the asymmetric unit. The coordination at the Sn atoms is distorted tetrahedral with C—Sn—C angles between 105.79 (16) and 112.47 (15)°. The Sn—C distances are in the range 2.135 (4)–2.150 (4) Å and the Sn$\cdots$O intramolecular distances are between 3.042 and 3.128 (3) Å. The related molecule tetrakis(2-methoxybenzyl)tin, $[Sn(C_8H_9O)_4]$, crystallizes in space group $P\bar{4}2_1c$ and has crystallographic $\bar{4}$ symmetry, with Sn—CH_2 2.165 (6) Å and an intramolecular Sn$\cdots$O separation of 3.241 (5) Å.

Comment

Only a limited number of crystal structures have been reported for tetraaryltin ($SnAr_4$) derivatives. These include tetrakis(*o*-tolyl)tin (Belsky, Simonenko, Reikhsfeld & Saratov, 1983), tetrakis(*p*-thioanisyl)tin (Wharf & Simard, 1987), tetrakis(*p*-tolyl)tin (Karipides & Wolfe, 1975), tetrakis(pentafluorophenyl)tin (Karipides, Forman, Thomas & Reed, 1974), tetrakis(3-methylphenyl)tin (Karipides & Oertel, 1977), tetraphenyltin (Chieh & Trotter, 1970; Akhmed & Aleksandrov, 1970; Belsky, Simonenko, Reikhsfeld & Saratov, 1983; Engelhardt, Leung, Raston & White, 1982), tetrakis(*p*-anisyl)tin (Wharf & Simard, 1987), tetrakis(*p*-methylsulfonylphenyl)tin (Wharf, Simard & Lamparski, 1990) and tetrakis(*p*-ethoxyphenyl)tin (Wharf & Simard, 1991). All have crystallographic $\bar{4}$ symmetry except

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