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

B1—B2	1.823 (8)	Pd5—P1	2.2986 (15)
B1—B3	1.788 (9)	Pd5—P2	2.2876 (13)
B1—S4	1.905 (6)	S6—B7	1.858 (6)
B1—Pd5	2.261 (6)	B7—B8	1.798 (9)
B1—B9	1.949 (8)	B8—B9	1.789 (9)
B2—B3	1.815 (7)	P1—C11	1.811 (5)
B2—Pd5	2.280 (5)	P1—C17	1.813 (5)
B2—S6	1.921 (6)	P1—C18	1.822 (5)
B2—B7	1.980 (9)	P2—C21	1.818 (5)
B3—B7	1.814 (9)	P2—C27	1.816 (5)
B3—B8	1.736 (9)	P2—C28	1.819 (5)
B3—B9	1.797 (8)	B7—H78	1.47
S4—Pd5	2.3588 (14)	B8—H89	1.13
S4—B9	1.883 (6)	B8—H78	1.15
Pd5—S6	2.6759 (15)	B9—H89	1.47
B1—S4—B9	61.9 (3)	S4—Pd5—P2	146.81 (5)
Pd5—S4—B9	109.7 (2)	S4—Pd5—P1	94.86 (5)
B1—S4—Pd5	63.0 (2)	B1—Pd5—S6	84.9 (2)
B1—Pd5—B2	47.3 (2)	B2—Pd5—S6	44.73 (15)
B1—Pd5—P2	108.3 (2)	S6—Pd5—P2	98.13 (5)
B2—Pd5—P2	88.20 (14)	S6—Pd5—P1	112.87 (5)
B1—Pd5—P1	143.03 (14)	S4—Pd5—S6	102.14 (5)
B2—Pd5—P1	157.25 (15)	B2—S6—B7	63.2 (3)
P1—Pd5—P2	101.15 (5)	Pd5—S6—B7	106.7 (2)
B1—Pd5—S4	48.65 (14)	B2—S6—Pd5	56.7 (2)
B2—Pd5—S4	88.00 (13)		

Fig. 1 was prepared using *ORTEP*II (Johnson, 1976) as implemented in *PLATON* (Spek, 1995*a*). Examination of the structure with *PLATON* showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1992). Cell refinement: *SET*-4 and *CELDIM* (Enraf–Nonius, 1992). Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX*94. Program(s) used to refine structure: *NRCVAX*94 and *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *NRCVAX*94, *PLATON* (Spek, 1995*a*) and *PLUTON* (Spek, 1995*b*). Software used to prepare material for publication: *NRCVAX*94 and *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1335). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bromo[tris(2-cyanoethyl)phosphine]gold(I), [(CEP)AuBr]

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Abstract

In $[AuBr(C_9H_{12}N_3P)]$, two C≡N groups of the tris(2-cyanoethyl)phosphine (CEP) ligand are positioned in the vicinity of square-planar coordination sites so as to shield the central Au^I atom in at least two directions. Although the disposition of two of the C≡N groups is indicative of Au···C≡N interactions, these groups do not participate in the metal coordination. The observed shielding of the metal atom is similar to that reported for one of the polymorphs of the monomeric complex [(CEP)AuCl].

Comment

We have been studying the chemistry and crystal structures of linear monomeric gold(I) complexes with ligands having phosphorus, sulfur and selenium donors as a part of our research program dealing with model compounds for gold drugs (Hussain & Schlemper, 1987; Hussain, 1986; Hussain & Isab, 1984, 1985*a*,*b*; Isab & Hussain, 1986). The triorganophosphine tris(2-

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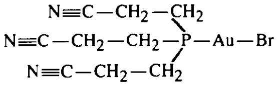
cyanoethyl)phosphine (CEP), synthesized by Rauhut, Hechenbleikner, Currier, Schaefer & Wystrach (1959), with three terminal electron-withdrawing C≡N groups, contains a P atom which is thought to be a much poorer base than that in $P(CH_2CH_3)_3$ (Cotton, Darensbourg & Ilsley, 1981). In spite of its low nucleophilicity, CEP can act as a P-donor ligand, forming strong metal–phosphorus bonds in several metal complexes (Khan, King, Fackler & Winpenny, 1993). The C≡N groups in these complexes do not participate in the coordination.

When gold(I) chloride reacts with CEP, two crystalline polymorphs are reported to be formed. These polymorphs of monomeric chloro[tris(2-cyanoethyl)phosphine]gold(I), [(CEP)AuCl], have recently been synthesized and crystallographically characterized (Fackler, Staples, Khan & Winpenny, 1994). With gold(I) bromide, however, we find that the CEP ligand yields only a single crystalline form of the monomeric [(CEP)AuBr] complex, (I). We now report the X-ray structure analysis of this complex.



$$\begin{array}{l} N{\equiv}C{-}CH_2{-}CH_2 \\ N{\equiv}C{-}CH_2{-}CH_2{-}P{-}Au{-}Br \\ N{\equiv}C{-}CH_2{-}CH_2 \end{array}$$

(I)

The structure determination of the complex revealed two C≡N groups positioned in the vicinity of square-planar coordination sites so as to shield the Au^I center in at least two directions (Fig. 1). In this respect, the structure of [(CEP)AuBr] resembles that of polymorph (2) of [(CEP)AuCl] (Fackler, Staples, Khan & Winpenny, 1994). The non-bonding P···C(5)···N(2) and P···C(8)···N(3) angles are 99.2 (7) and 95.5 (8)°, respectively. The shielding of the metal observed in [(CEP)AuBr] is structurally suggestive of Au···C≡N interactions, but the non-bonded Au···C(6) and Au···C(9) separations [3.34 (4) and 3.42 (3) Å, respectively] and the Au···N(2) and Au···N(3) separations [3.43 (7) and 3.49 (5) Å, respectively] are considerably longer than the sum of the respective van der Waals radii, ruling out the possibility of any kind of coordination with gold. The relevant structural parameters for some related monomeric gold(I) complexes are listed in Table 3. The Au—P distance of 2.230 (4) Å is within the range previously observed for other P-donor monomeric gold(I) complexes. As expected, the Au—Br distance of 2.410 (2) Å is longer than the average Au—Cl distance of 2.305 (3) Å observed for the corresponding chloro complexes. The P—Au—Br angle of 176.7 (1)° is close to the average P—Au—Cl angle of 177.5 (5)°. The torsion angles Au—P—C(5)—N(2) and Au—P—C(8)—N(3) have values of 18 (1) and −1 (1)°, respectively.

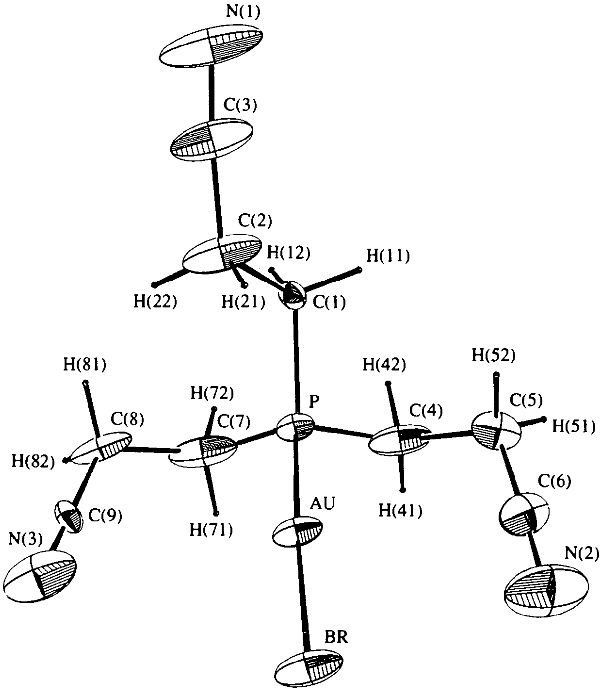


Fig. 1. *ORTEP* (Johnson, 1965) drawing of a molecule of bromo[tris(2-cyanoethyl)phosphine]gold(I), [(CEP)AuBr], showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The N atoms of the C≡N groups are highly anisotropic because of the unrestricted free rotation possible for these groups.

Gold(I) phosphine complexes are widely used as anti-arthritic and antitumor drugs (Stinson, 1989; Parish & Cottril, 1987; Mirabelli *et al.*, 1986; Johnson *et al.*, 1985). Complexes such as $[Et_3PAuCl]$ show some activity when administered orally (Walz, DiMartino & Sutton, 1974). Since CEP is less basic than Et_3P and, therefore, somewhat less prone to oxidation to the phosphine oxide, it is possible that [(CEP)AuBr] might find some utility as a drug.

Experimental

The title complex, [(CEP)AuBr], was synthesized by standard procedures (Kauffman & Cowan, 1960; Uson, Laguna & Laguna, 1989) using the corresponding bromo salt $AuBr.2H_2O$.

Crystal data

$[AuBr(C_9H_{12}N_3P)]$
$M_r = 470.06$
Monoclinic
$P2_1/c$
$a = 8.725\ (2)$ Å
$b = 7.891\ (3)$ Å
$c = 19.127\ (3)$ Å
$\beta = 100.03\ (3)°$
$V = 1296.7\ (6)\ Å^3$
$Z = 4$
$D_x = 2.408$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 2.37–30.00°$
$\mu = 14.52\ mm^{-1}$
$T = 296\ (2)$ K
Plate
0.95 × 0.85 × 0.45 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (*SDP*; Frenz, 1991)
$T_{\min} = 0.308$, $T_{\max} = 0.908$
3883 measured reflections
3776 independent reflections
2372 observed reflections [$F_o^2 > 2\sigma(F_o^2)$]
$R_{\rm int} = 0.0411$
$\theta_{\max} = 30.0°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 11$
$l = -26 \rightarrow 26$
3 standard reflections monitored every 97 reflections intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0384$
$wR(F^2) = 0.0421$
$S = 1.001$
2372 reflections
136 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.2275P)^2 + 8.12P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 1.12$ e Å^{-3}
$\Delta\rho_{\min} = -0.88$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from Cromer (1974) and *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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	0.4587 (1)	0.2585 (1)	0.0938 (1)	0.029 (1)
Br	0.1871 (2)	0.3313 (3)	0.0623 (1)	0.043 (1)
P	0.7060 (5)	0.1787 (6)	0.1265 (2)	0.025 (1)
N(1)	1.006 (3)	0.752 (2)	0.1637 (15)	0.056 (6)
N(2)	0.360 (3)	0.064 (5)	0.2402 (13)	0.068 (7)
N(3)	0.519 (3)	0.233 (3)	−0.0816 (14)	0.065 (7)
C(1)	0.838 (2)	0.346 (2)	0.1594 (8)	0.028 (3)
C(2)	0.811 (2)	0.518 (3)	0.1195 (12)	0.043 (5)
C(3)	0.924 (3)	0.645 (3)	0.1416 (13)	0.050 (5)
C(4)	0.732 (2)	0.028 (3)	0.2006 (11)	0.042 (5)
C(5)	0.660 (3)	0.086 (3)	0.2637 (11)	0.050 (6)
C(6)	0.500 (3)	0.071 (4)	0.2525 (11)	0.056 (6)
C(7)	0.786 (2)	0.070 (3)	0.0588 (1)	0.042 (5)
C(8)	0.791 (2)	0.149 (3)	−0.0111 (11)	0.038 (4)
C(9)	0.638 (2)	0.199 (3)	−0.0495 (9)	0.035 (4)

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

Au—P	2.230 (4)	Au—Br	2.410 (2)
P—C(1)	1.79 (2)	N(1)—C(3)	1.14 (3)
P—C(4)	1.83 (2)	N(2)—C(6)	1.21 (3)
P—C(7)	1.79 (2)	N(3)—C(9)	1.15 (3)
C(1)—C(2)	1.56 (3)	C(4)—C(5)	1.52 (3)
C(2)—C(3)	1.42 (3)	C(5)—C(6)	1.38 (3)
C(7)—C(8)	1.48 (2)	C(8)—C(9)	1.46 (3)
Au···N(2)	3.43 (7)	Au···C(6)	3.34 (4)
Au···N(3)	3.49 (5)	Au···C(9)	3.42 (3)
P—Au—Br	176.7 (1)	C(5)—C(4)—P	114 (2)
C(7)—P—C(1)	107.2 (8)	C(8)—C(7)—P	122 (2)
C(7)—P—C(4)	103.7 (9)	C(6)—C(5)—C(4)	114 (2)
C(1)—P—C(4)	102.8 (8)	C(3)—C(2)—C(1)	116 (2)
C(7)—P—Au	114.4 (6)	C(9)—C(8)—C(7)	114 (2)
C(1)—P—Au	115.0 (6)	N(2)—C(6)—C(5)	177 (3)
C(4)—P—Au	112.5 (6)	N(3)—C(9)—C(8)	178 (2)
N(1)—C(3)—C(2)	173 (3)	P—C(2)—N(3)	95.5 (8)
C(2)—C(1)—P	116 (1)	P—C(5)—N(2)	99.2 (7)

Table 3. *Comparison of bond lengths (Å) and angles (°) for mononuclear gold(I) triorganophosphine chloride/bromide complexes*

	Au—X	Au—L	L—Au—X
[(CEP)AuCl][a] (1)	2.295 (3)	2.225 (3)	177.8 (6)
[(CEP)AuCl][a] (2)	2.315 (2)	2.243 (5)	177.1 (5)
[(Ph$_3$P)SeAuCl][b]	2.277 (3)	2.371 (4)	178.6 (5)
[(Ph$_3$P)SAuCl][c]	2.255 (4)	2.256 (3)	175.6 (4)
[Cy$_2$PhPAuCl][d]	2.281 (3)	2.234 (4)	178.3 (4)
[(CEP)AuBr][e]	2.410 (2)	2.230 (4)	176.7 (1)

References: (*a*) Fackler, Staples, Khan & Winpenny (1994); (*b*) Isab & Hussain (1986); (*c*) Hussain & Schlemper (1987); (*d*) Muir, Cuadrado & Muir (1991); (*e*) this work.

A small colorless crystal was mounted on a glass fiber in a random orientation. The Au atom was found by direct methods, the remaining non-H atom positions being located by Fourier techniques. H atoms were placed in idealized positions (C—H 0.95 Å), with fixed isotropic displacement parameters ($U = 0.05$ Å^2). All computations were carried out on a Compaq PC using a combination of the PCDOS versions of *SHELXS*86 (Sheldrick, 1990) and *SHELXL*93 (Sheldrick, 1993) or the personal *SDP* software (Frenz, 1988, 1991).

One of us (MSH) is thankful to the KFUPM Research Committee and the Chemistry Department for upgrading the CAD-4 computer system in the Crystallographic Laboratory.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1183). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Bis[*N*-(4-bromophenyl)salicylaldiminato]cobalt(II)

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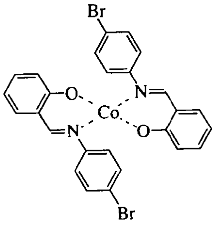
Abstract

In the title compound, *trans*-bis[2-(4-bromophenyliminomethyl)phenolato-*N*,*O*]cobalt(II), $[Co(C_{13}H_9BrNO)_2]$, the Co atom is situated on a twofold axis and the geometry around the metal centre can be described as distorted tetrahedral with a *trans* conformation. The Co—O and Co—N bond lengths are 1.891 (3) and 2.006 (4) Å, respectively.

Comment

Because of their preparative accessibility and structural variability a great number of Schiff base complexes of cobalt have been subject to extensive studies. Tetracoordinate Schiff base metal complexes may form *trans* and *cis* planar or tetrahedral structures. A strictly planar or slightly distorted *trans* configuration is characteristic for transition metal complexes of cobalt(II) with a CoN_2O_2 coordination sphere (Garnovskii, Nivorozhkin & Minkin, 1993). Structural and molecular properties of related Co^{II} complexes have been reported (Hennig, 1971; Panova, Vikulova & Popotov, 1980; Kogan, Kharabov, Osipov & Kochin, 1981).

In the course of a systematic investigation of the coordination spheres of Co^{II} in Schiff base complexes the structure of the title complex, (I), formed by the reaction of *N*-(4-bromophenyl)salicylaldimine with cobalt(II) acetate, was determined.



(I)

The Co atom lies on a twofold axis and is coordinated by two O and two N atoms. The distortion from idealized geometry is due to the O1—Co—N1 angle of 96.75 (4)°, which is smaller than the O1—Co—N1 angle of 111.59 (14)°. The Co—O and Co—N distances found in the structure agree with the values in other tetrahedral complexes.

The phenyl rings show small distortions from ideal geometry, with the C—C distances for the phenyl rings close to the expected value for aromatic rings [1.394 (5) Å; Sutton, 1965]. The bond lengths between the N1 and C1 atoms are typical of C═N double-bond lengths [1.287 (6) Å]. The bond lengths Br1—C11 of 1.892 (5), O1—C3 of 1.319 (5) and N1—C8 of 1.428 (6) Å are within expected ranges.

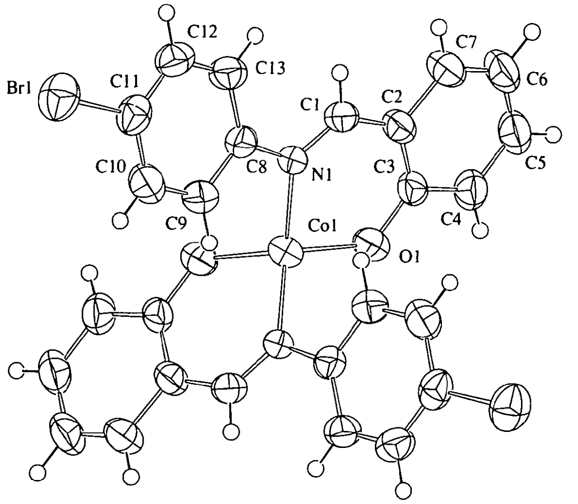


Fig. 1. *ORTEP* view (Johnson, 1965) of the molecular structure of the title compound. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

0.5 mmol of $[Co(AcO)_2].4H_2O$ in methanol was added to 1.0 mmol of *N*-(4-bromophenyl)salicylaldimine in acetonitrile with stirring and heating. The mixture was refluxed for 6 h. The red crystals that formed were washed twice with ethanol.

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