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Dibromo[N-(2-pyridyl)acetamide]mercury(II)

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Abstract. $C_7H_8Br_2HgN_2O$, $[HgBr_2(C_7H_8N_2O)]$, monoclinic, $P2_1/c$, $a = 8.070$ (1), $b = 8.802$ (1), $c = 15.942$ (3) Å, $\beta = 94.13$ (1)°, $V = 1129.5$ (5) Å³, $Z = 4$, $M_r = 496.56$, $d_x = 2.92$ Mg m⁻³, $\mu(Cu\,K\alpha) = 17.62$ mm⁻¹. The structure was solved by the heavy-atom method and refined by full-matrix least-squares calculations. The final R was 0.077 for 2057 observed reflexions. The Hg atom is coordinated to two Br atoms, one O and one N. The ligand molecule bridges two Hg ions related by a twofold screw axis.

Introduction. Zinc(II), cadmium(II) and mercury(II) halide complexes with N-(2-pyridyl)acetamide were synthesized (Airoldi & Gonçalves, 1978). The structure determination of colourless crystals of the title compound was undertaken to study the coordination around the Hg^{II} atom. Cell dimensions were determined and refined by a least-squares fit to the setting angles of 25 reflexions on a CAD-4 automatic diffractometer. Intensity measurements were carried out up to $\theta = 75°$, using the $\omega-2\theta$ scan mode, with graphite-monochromated Cu $K\alpha$ radiation and a prismatic crystal with a hexagonal base, approximated to a sphere of radius 0.2 mm. Lorentz, polarization and absorption corrections were applied (International Tables for X-ray Crystallography, 1972). Of the 2311 measured unique reflexions, 2057 were considered observed $[I > 3\sigma(I)]*$ and retained for use in structure determination and refinement.

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* Lists of structure factors, anisotropic thermal parameters, interatomic distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34973 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates for the mercury atom* $(\times 10^5)$ *and for the other non-hydrogen atoms* $(\times 10^4)$ *with their e.s.d.'s in parentheses*

	x	y	z
Hg	10220 (4)	30255 (4)	55333 (2)
Br(1)	3254 (2)	1588 (1)	4960 (1)
Br(2)	−1784 (2)	3996 (1)	5642 (1)
O	311 (9)	1065 (6)	6822 (4)
N(1)	−2725 (8)	−555 (8)	8360 (4)
N(2)	−840 (9)	−1005 (7)	7343 (4)
C(2)	−2309 (8)	−283 (8)	7588 (4)
C(3)	−3298 (11)	587 (9)	7021 (5)
C(4)	−4704 (12)	1222 (11)	7287 (7)
C(5)	−5110 (11)	979 (11)	8105 (6)
C(6)	−4142 (12)	57 (12)	8611 (5)
C(7)	333 (9)	−305 (8)	6930 (5)
C(8)	1668 (14)	−1276 (11)	6622 (6)

The structure was solved by the heavy-atom method. It was refined by full-matrix least-squares calculations by minimization of $\sum w(k|F_o| - |F_c|)^2$, where $w = (0.016\,|F|^2 - 0.516\,|F| + 6.927)^{-1}$ for observed and $w = 0$ for unobserved reflexions, until all the atomic parameter shifts were smaller than each standard deviation. The final unweighted R factor, omitting unobserved reflexions, was 0.077, and including them 0.082. Anisotropic temperature factors were assigned to all non-hydrogen atoms. Complex neutral-atom scattering factors were used (International Tables for X-ray Crystallography, 1974). The H atoms were placed at their calculated positions; their isotropic temperature factor was taken as 6.0 Å². Positional parameters are given in Table 1.

Discussion. The complex is found to have an infinite extended chain-like structure with the organic ligand
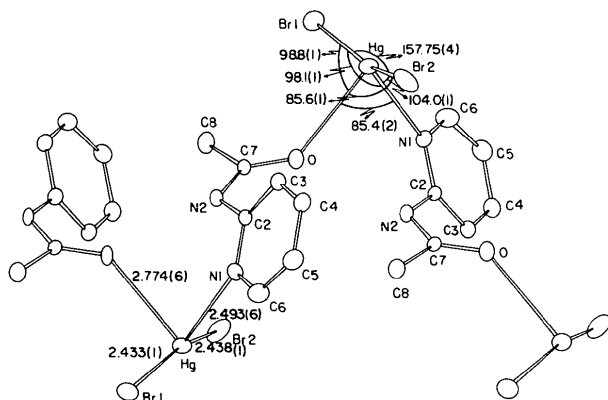
Fig. 1. Diagram of the structure showing the chain along the twofold screw axis.

forming a bridge between two Hg^{II} atoms related by the twofold screw axis. The ligand molecule is bidentate and is coordinated to Hg through N(1) from the pyridyl group and O from the acetyl group (Fig. 1). These two groups are planar within one standard deviation and the angle between them is 51·7°. The inclination of the acetyl group with respect to the pyridyl group is defined by the torsion angles N(1)—C(2)—N(2)—C(7) of 135·3° and C(2)—N(2)—C(7)—O of −8·8°. The average distances and angles agree well with the values for

similar compounds. There is a close intermolecular approach of 2·913 (8) Å between O and N(2) ($-x$, $\frac{1}{2}+y$, $\frac{3}{2}-z$).

The angular geometry about the Hg atom is highly irregular, the X—Hg—Y angles ranging from 85·6 (1) to 157·75 (4)°. The coordination distances around the metal are less than 2·5 Å, except for the Hg—O [2·774 (6) Å]. One of the Br atoms is also close to another Hg atom of a neighbouring chain with Hg—Br(2) ($-x$, $1-y$, $1-z$) of 3·306 (1) Å. Fig. 1 gives the relevant bond distances and angles in the structure.

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Bis(tricyclohexylphosphine)gold(I) Thiocyanate

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Abstract. [Au{(cyclo-C$_6$H$_{11}$)$_3$P}$_2$]SCN, C$_{36}$H$_{66}$Au-P$_2^+$.SCN$^-$, orthorhombic, M_r = 815·9, a = 25·151 (19), b = 15·737 (14), c = 9·630 (9) Å, Z = 4, μ(Mo $K\alpha$) = 3·83 mm^{-1}, $F(000)$ = 1676, V = 3812 Å^3, T = 298 ± 2 K. The complex forms an almost linear, two-coordinate gold compound, with both the bis(tricyclohexylphosphine)gold(I) cation and the thiocyanate anion located on the mirror plane in space group *Pnma*.

Introduction. The title compound was obtained during the attempted preparation of a gold cluster complex analogous to [Au$_{11}$(Ph$_3$P)$_7$](SCN)$_3$ (Cariati & Naldini, 1971). Similarly, in a recent report of an improved synthesis of [Au$_{11}$(Ph$_3$P)$_7$](SCN)$_3$, the isolation of

[Au(Ph$_3$P)$_2$]SCN was described (Vollenbroek, Bouten, Trooster, Van den Berg & Bour, 1978). [Au{(C$_6$-H$_{11}$)$_3$P}$_2$]SCN could be either linear with ionic thiocyanate or trigonal with the thiocyanate coordinated as in the case of [Au(Ph$_3$P)$_2$Cl] (Baenziger, Dittmore & Doyle, 1974). In order to include the compound in a series of complexes of Au^I to be studied by Mössbauer spectroscopy it was of interest to determine its coordination geometry. For this reason, and also since no structures of complexes of Au with (cyclo-C$_6$H$_{11}$)$_3$P were known, the structure determination of [Au{(cyclo-C$_6$H$_{11}$)$_3$P}$_2$]SCN was undertaken.

The title compound was prepared during the reaction between (tricyclohexylphosphine)gold thiocyanate and sodium borohydride in ethanol solution. The reaction