Johnson, C. K. (1976). *ORTEP* II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
Jones, P. E., Ansell, G. B. & Katz, L. (1969). *Acta Cryst.* B**25**, 1939–1943.
Kastalsky, V. & McConnell, J. F. (1969). *Acta Cryst.* B**25**, 909–915.
McConnell, J. F. & Kastalsky, V. (1967). *Acta Cryst.* **22**, 853–859.
Poll, W., Wunderlich, H. & Wussow, H.-G. (1979). *Z. Kristallogr.* **149**, 103–104.
Porta, P., Sgamellotti, A. & Vinciguerra, N. (1968). *Inorg. Chem.* **7**, 2625–2629.
Shetty, P. S. & Fernando, Q. (1969). *Acta Cryst.* B**25**, 1294–1298.
Wasson, J. R., Woltermann, G. M. & Stoklosa, H. J. (1973). *Fortschr. Chem. Forsch.* **35**, 65–129.

Acta Cryst. (1980). B**36**, 1194–1196

The Structure of Bis(benzamido)mercury(II)

By Joan Halfpenny* and R. W. H. Small

*Chemistry Department, The University, Lancaster LA*1 4*YA, England*

(*Received* 13 *November* 1979; *accepted* 22 *January* 1980)

Abstract. $[Hg(C_7H_6NO)_2]$, $C_{14}H_{12}HgN_2O_2$, monoclinic, $C2/c$, $a = 21.33$ (2), $b = 15.69$ (2), $c = 8.42$ (2) Å, $\beta = 110.5$ (1)°, $D_m = 2.19$, $D_x = 2.216$ Mg m^{-3} with $Z = 8$. The structure has been determined from 931 diffractometer-measured intensities ($R = 0.064$). Hg is almost linearly bonded to two N atoms [Hg–N = 2·06 (3), 2·04 (2) Å, N–Hg–N = 172 (1)°] and also equatorially to two O atoms of adjacent molecules [Hg–O = 2·67 (2) and 2·83 (3) Å]. The O–Hg–O angle is 76 (1)°. There is also a weak intermolecular hydrogen bond [N···O = 3·03 (5) Å].

Introduction. Interest in the structures of the Hg derivatives of acid amides lies in the nature of the Hg–O interaction, whether the amide group is to any extent bidentate and, in the crystal, the extent of intermolecular coordination. The title compound, kindly supplied by Dr F. G. Thorpe, was recrystallized by slow evaporation at 277 K from an ethanol solution to give needles elongated about c showing the forms {110} but without well-defined end faces. The crystals decomposed slowly upon irradiation. Cell dimensions and systematic absences (hkl when $h + k = 2n + 1$, $h0l$ when $l = 2n + 1$) were obtained from Weissenberg photographs with Cu $K\alpha$ radiation ($\lambda = 1.542$ Å). The crystal density was measured by flotation. With a crystal of dimensions 0·06 × 0·05 × 0·29 mm set about c, intensity data for 1387 reflexions in the range $4° < 2\theta < 40°$ were measured on a Stoe STADI-2 automatic two-circle diffractometer; graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å) was used. Absorption corrections ($\mu = 11.234$ mm^{-1}) were made using the *SHELX* 76 suite of programs (Sheldrick, 1976). Only 931 reflexions, for which $I > 3\sigma(I)$, were used in subsequent calculations employing the XRAY 72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Patterson maps which gave strong peaks at $y = 0$ indicated the space group $C2/c$ rather than Cc. Hg coordinates obtained from these maps were used to phase ($F_o - F_c$) maps to give the remainder of the atoms other than H. Full-matrix least-squares refinement (using unit weights) of all non-H atom positions, anisotropic U_{ij} values for Hg and isotropic U's for the remaining non-H atoms was alternated with refinement of the scale factors for each layer ($l = 0$ to 7) of the diffractometer data, U_{33} being fixed to avoid excessive correlation. At an intermediate stage an ($F_o - F_c$) map showed positive regions in the neighbourhoods of most expected H positions. Inclusion of H atoms for the phenyl groups at calculated positions (fixed 1·08 Å from the parent atom with U fixed at 0·05 Å^2) was found to improve the agreement. When refinement was complete (shift/error < 0·1 for all parameters), R was 0·064 and a final ($F_o - F_c$) map showed no significant features. Atomic scattering factors for Hg, including anomalous scattering, were taken from Cromer & Mann (1968) and Cromer & Liberman (1970); those for all other atoms were from *International Tables for X-ray Crystallography* (1962). Final coordinates and temperature factors for atoms other than H are given in Table 1.† A diagram of the molecule is shown in Fig. 1.

---

* Present address: School of Chemistry, Brunel University, Uxbridge, England.

† Lists of structure factors and bond lengths and angles around non-Hg atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35053 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0567-7408/80/051194-03$01.00

Discussion. In all reported primary-amide structures, the carboxamide group has been found to be planar (Leiserowitz & Schmidt, 1969) with C—N longer than C—O, both being intermediate between double and single bonds. On account of the predominant scattering power of Hg in the present compound, N and O atoms could not be reliably differentiated; however, the method of preparation of this compound and its IR absorption spectrum indicate that Hg is attached to N. The two Hg—N distances, Table 2, are typical of covalent bonding with Hg joined to each amide group so that HgN(1)C(1)O(1)C(2) and HgN(2)C(8)O(2)-C(9) are planar to within 0·05 and 0·07 Å respectively; the dihedral angle between these planes is 56°. The C—N and C—O bonds around C(1) and C(8) are all intermediate between double and single, and although the C—N bonds [1·24 (4) and 1·21 (5) Å] in each amide group appear shorter than the C—O bonds [1·33 (5) and 1·40 (5) Å], the differences are only marginally significant. Typical average distances in primary amides are C—N = 1·33 and C—O = 1·25 Å. In view of the partial double-bond character of the C—N bonds, the Hg atom may be described as being *cisoid* relative to O in both groups.

Table 1. *Fractional atomic coordinates* (× 10^3, *for* Hg × 10^4) *and thermal parameters* (× 10^3)

	x	y	z	U (Å^2)
N(1)	374 (1)	666 (2)	569 (4)	35 (8)
C(1)	338 (2)	693 (2)	428 (5)	30 (9)
O(1)	326 (1)	645 (2)	290 (4)	50 (8)
C(2)	312 (1)	781 (2)	401 (4)	16 (8)
C(3)	328 (2)	835 (2)	528 (5)	29 (9)
C(4)	304 (2)	918 (3)	502 (6)	50 (11)
C(5)	264 (2)	951 (3)	351 (6)	60 (12)
C(6)	243 (2)	893 (2)	216 (5)	37 (10)
C(7)	268 (2)	807 (2)	243 (5)	44 (11)
N(2)	419 (1)	411 (2)	610 (4)	16 (7)
C(8)	444 (2)	370 (2)	523 (5)	31 (9)
O(2)	469 (1)	410 (1)	411 (3)	35 (7)
C(9)	445 (2)	275 (2)	527 (5)	22 (9)
C(10)	486 (3)	229 (3)	467 (7)	78 (16)
C(11)	483 (3)	135 (3)	464 (7)	82 (16)
C(12)	437 (2)	97 (3)	498 (4)	64 (14)
C(13)	397 (2)	144 (3)	559 (7)	72 (15)
C(14)	403 (2)	233 (3)	576 (6)	53 (12)
Hg	4039 (1)	5396 (1)	5929 (2)	

Anisotropic thermal parameters for Hg (× 10^4)

U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
475 (10)	326 (9)	240 (*)	−12 (9)	187 (7)	−13 (10)

* Restrained.

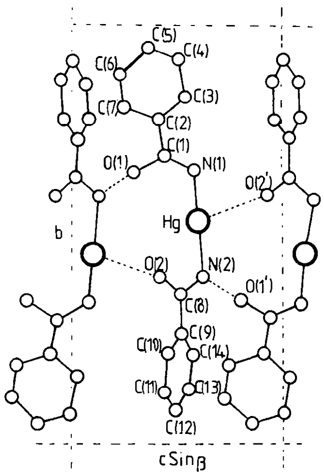

Fig. 1. Projection of part of the unit cell along *a*.

Table 2. *Bond distances* (Å) *and angles* (°) *around* Hg

Hg—N(1)	2·06 (3)	Hg—O(2')	2·67 (2)
Hg—N(2)	2·04 (3)	Hg—O(2'')	2·83 (3)
N(1)—Hg—N(2)	172 (1)	N(1)—Hg—O(2')	81 (1)
N(1)—Hg—O(2'')	90 (1)	O(2)^{1}—Hg—O(2'')	76 (1)
N(2)—Hg—O(2')	102 (1)	N(2)—Hg—O(2'')	98 (1)

Symmetry code: (') $x, 1-y, \frac{1}{2}+z$; ('') $1-x, 1-y, 1-z$.

The angles of twist between the planes of the amide groups and those of the phenyl rings for the two halves of the molecule are 7° for that at C(1)C(2) and 22° for that at C(8)C(9). These may be compared with the following, found in other benzamide derivatives: benzamide, 24·6° (Blake & Small, 1972); 2:1 complex of benzamide and succinic acid, 24° (Huang, Leiserowitz & Schmidt, 1973); *m*-hydroxybenzamide, 24·4° (Katsube, Sasada & Kakudo, 1966); *N,N'*-(*p*-phenylene)-dibenzamide, 29·1° (Harkema & Gaymans, 1977); terephthalamide, 23° (Cobbledick & Small, 1972). In all these cases the H atoms have been located and the twist is attributable to repulsion between the *ortho* H in the phenyl group and the H *trans* to O in the amide group. Calculation shows that in a completely planar molecule these two H atoms would be impossibly close (1·80 Å); twisting the amide group through 7°, as in the C(1)C(2) group of the present compound, would only increase this distance to 1·81 Å, well below the expected van der Waals value of 2·0 Å. Indirect but strong evidence is thus provided that there can be no H on N(1), *i.e.* this fragment is in the enol form with the H attached to O. A similar conclusion concerning the enol character of bis(acetamido)mercury(II) was reached by Kamenar & Grdenić (1969) on the basis of the relative lengths of the C—N and C—O bonds, and although similar values (with lower standard deviations) are found in the present compound, it is considered that the strongest evidence for the enol form is from the small angle of twist; only accurate H atom location would provide conclusive proof. No similar conclusion is possible for the amide group at C(8)C(9) where the angle of twist approaches that found in primary amides; it is improbable that two similar groups attached to the same Hg would be in different tautomeric forms. In this case the twist of the amide group at C(8)C(9) may be due to intermolecular interactions.

In addition to the covalent Hg—N bonds, there are intermolecular contacts from Hg to O(2), one of 2·67 (2) Å to O(2′) of a *c*-glide-related molecule, and the other of 2·83 (3) Å to O(2″) of a centrosymmetrically related molecule. The effective coordination of Hg is thus of an irregular fourfold type similar to that found in (chloromercurio)acetaldehyde (Halfpenny & Small, 1979). The angles around Hg are given in Table 2. There are no short contacts from O(1) to neighbouring Hg atoms; instead, there is an intermolecular distance of 3·03 (5) Å to N(2) of a *c*-glide-related molecule; an H atom bonded with normal distances and angles to either O(1) or N(2) would be close to the O(1′)···N(2) direction. This contact must therefore be regarded as a weak hydrogen bond, either N—H···O or N···H—O depending upon whether the amide group at C(8) is in the keto or enol form. The presence, in this structure, of a balance between intermolecular Hg···O bonding and hydrogen bonds suggest that there is a similarity in the energies of the two types of bond. The molecules are arranged with their longest directions parallel to *b*; *c*-glide-related molecules are bound by Hg···O bonds and hydrogen bonds into strips one molecule wide. Pairs of adjacent centrosymmetrically related strips are bonded by Hg···O bonds.

We thank the Science Research Council for the award of a research studentship to one of us (JH).

References

Blake, C. C. F. & Small, R. W. H. (1972). *Acta Cryst.* **B28**, 2201–2206.

Cobbledick, R. E. & Small, R. W. H. (1972). *Acta Cryst.* **B28**, 2893–2896.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Halfpenny, J. & Small, R. W. H. (1979). *Acta Cryst.* B**35**, 1239–1241.

Harkema, S. & Gaymans, R. J. (1977). *Acta Cryst.* B**33**, 3609–3611.

Huang, C.-M., Leiserowitz, L. & Schmidt, G. M. J. (1973). *J. Chem. Soc. Perkin Trans.* 2, pp. 503–508.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

Kamenar, B. & Grdenić, D. (1969). *Inorg. Chim. Acta*, **3**, 25–28.

Katsube, Y., Sasada, Y. & Kakudo, M. (1966). *Bull. Chem. Soc. Jpn*, **39**, 2576–2583.

Leiserowitz, L. & Schmidt, G. M. J. (1969). *J. Chem. Soc. A*, pp. 2576–2583.

Sheldrick, G. M. (1976). *SHELX* 76. Program for crystal structure determination. Univ. of Cambridge, England.

Stewart, J. M., Kruger, G. J., Ammon, H. L., Dickinson, C. & Hall, S. R. (1972). The XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1980). B**36**, 1196–1198

(2-Benzoylphenyl)tricarbonyl(triphenylphosphine)rhenium

By Hans Preut and Hans-Jürgen Haupt

Lehrstuhl für Anorganische Chemie II *der Universität, D*-4600 *Dortmund* 50, *Federal Republic of Germany*

(*Received* 27 *November* 1979; *accepted* 28 *January* 1980)

Abstract. $[\mathrm{Re}(C_{13}H_9O)(C_{18}H_{15}P)(CO)_3]$, $C_{34}H_{24}O_4PRe$, $M_r = 713{\cdot}7$, triclinic, $a = 11{\cdot}708\,(2)$, $b = 12{\cdot}371\,(2)$, $c = 9{\cdot}922\,(2)$ Å, $\alpha = 88{\cdot}60\,(7)$, $\beta = 77{\cdot}45\,(7)$, $\gamma = 89{\cdot}58\,(7)°$, $U = 1402$ Å^3; $Z = 2$, $D_c = 1{\cdot}691$ Mg m^{-3}; $F(000) = 350$; Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, μ(Mo $K\alpha$) $= 2{\cdot}11$ mm^{-1}. Space group $P\bar{1}$. Final $R = 0{\cdot}08$ for 5780 unique X-ray diffractometer data. The molecular structure has been elucidated.

Introduction. The title compound is a metallation product of benzophenone and $[\mathrm{Re}(C_6H_5)(CO)_5]$ reacted with phosphine (Schwab, 1975). The following is a structural characterization of this Re metallated benzophenone. The structure was solved by Patterson and Fourier methods and refined by blocked full-matrix least squares with isotropic temperature factors for C(1), O(1), C(2), O(2), C(3), O(3), C(4) and O(4), a common isotropic temperature factor for H [0·05 (1) Å^2] and anisotropic temperature factors for the remaining atoms. The phenyl rings were treated as rigid groups (C—C—C = 120, C—C—H = 120°, C—C = 1·395, C—H = 1·08 Å). Refinement converged with unit weights to $R = 0{\cdot}08$.*

---

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35065 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0567-7408/80/051196-03$01.00