

Fig. 1. The cation seen projected down the pseudo S_4 axis of the PtS₄ tetrahedron. The transformation matrix from crystal coordinates is

$$\begin{pmatrix} -0.216 & 0.919 & 0.331 \\ -0.041 & -0.347 & 0.937 \\ 0.976 & 0.189 & 0.113 \end{pmatrix}$$

The coordination of the Pt atom is an almost regular tetrahedron. The Pt and Ta atoms are in a linear arrangement, bridged by the methanethiolato ligands. The acute angles, Ta—S—Pt 70° (mean), and the short distances, Pt—Ta 2.798 Å (mean), are consistent with the criteria for metal—metal bonding (Dahl, Gil & Feltham, 1969). The bis(η -cyclopentadienyl)tantalum groups present the usual geometry described for bent

bis(η -cyclopentadienyl) metal complexes (Prout, Cameron, Forder, Critchley, Denton & Rees, 1974). The perpendicular distances from the Ta atoms to the rings are 2.09 Å for Ta(1)—C(1–5), 2.09 Å for Ta(1)—C(6–10), 2.08 Å for Ta(2)—C(11–15) and 2.04 Å for Ta(2)—C(16–20). These perpendiculars to the cyclopentadienyl rings meet the rings close (0.07 Å mean) to their centroids and make angles of 124.7° at Ta(1) and 129.4° at Ta(2). Also the TaS₂ plane is almost perpendicular to that defined by the normals to the cyclopentadienyl rings [87.4 and 86.6° respectively at Ta(1) and Ta(2)]. All the cyclopentadienyl rings are planar within experimental error and the C—C distances are in the range 1.39–1.42 Å.

References

- CARRUTHERS, J. R. (1975). *CRYSTALS User Manual*. Oxford Univ. Computing Laboratory.
- DAHL, L. F., GIL, E. R. & FELTHAM, R. D. (1969). *J. Am. Chem. Soc.* **91**, 1653–1664.
- DOUGLAS, W. E. & GREEN, M. L. H. (1972). *J. Chem. Soc. Dalton Trans.* pp. 1796–1800.
- PROUT, K., CAMERON, T. S., FORDER, R. A., CRITCHLEY, S. R., DENTON, B. & REES, G. V. (1974). *Acta Cryst.* **B30**, 2290–2304.
- PROUT, K., CRITCHLEY, S. R. & REES, G. V. (1974). *Acta Cryst.* **B30**, 2305–2311.
- ROLLETT, J. S. (1965). *Computing Methods in Crystallography*, p. 40. Oxford: Pergamon Press.
- SIGANPORIA, N. (1977). Part II Thesis, Univ. of Oxford.
- WASER, J. (1963). *Acta Cryst.* **16**, 1091–1094.

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Pentadecacarbonyl(methoxyphosphinato)pentaosmium

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Abstract. C₁₆H₃O₁₆Os₅P, Os₅(CH₃OP)(CO)₁₅, monoclinic, *Cc*, *a* = 15.990 (5), *b* = 9.762 (3), *c* = 16.965 (5) Å, β = 107.53 (2)°, *U* = 2525.2 Å³, *Z* = 4, *D_c* = 3.769 Mg m⁻³, μ (Mo *K* α) = 25.20 mm⁻¹. The structure was refined to an *R* of 0.060 for 3310 unique observed diffractometer data. The Os atoms define a distorted square pyramid. The phosphinato ligand lies under the Os₄ basal plane with the P atom bonded to the four Os atoms. Each Os atom is also coordinated to three terminal CO ligands.

Introduction. The title compound was one of a number of pentanuclear clusters containing uncommon P donor

ligands isolated from the pyrolysis of Os₃(CO)₁₁-P(OCH₃)₃ (Fernandez, Johnson & Lewis, 1979). The crystal structure determination of this product was undertaken to establish the molecular geometry since the results of spectroscopic analysis were ambiguous. A preliminary report has appeared (Fernandez, Johnson, Lewis & Raithby, 1978).

Crystals were obtained as red tablets from ethyl acetate/hexane. 3647 intensities were measured in the range 3.0 < 2 θ < 60.0° on a Syntex P2₁ four-circle diffractometer, with graphite-monochromated Mo *K* α radiation and a crystal 0.32 × 0.32 × 0.63 mm. Lp and semi-empirical absorption corrections (based on

468 azimuthal scan data from 47 reflections) were applied. Transmission factors on the full data set ranged from 0.554 to 0.961. Equivalent reflections were averaged to give 3310 unique observed intensities [$F > 5\sigma(F)$]. Cell dimensions were determined from diffractometer settings for 15 strong reflections in the range $20.0 < 2\theta < 30.0^\circ$.

The Os atoms were located by multiresolution tangent refinement, and the remaining non-hydrogen atoms from subsequent difference syntheses. The structure was refined by full-matrix least squares with anisotropic thermal parameters for Os and P, and the C and O atoms isotropic. In view of the relatively large uncertainties in the light-atom positions, a weak constraint was applied in the form of extra observational equations to make the C—O lengths equal. The mean C—O distance refined to 1.168 (13) Å; the constraint did not significantly increase R . Also included was an empirical extinction parameter x which refined to 0.00008 (1); F_c is multiplied by $(1 - 0.0001xF_c^2/\sin \theta)$.

Complex neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974) and the weighting scheme $w = 2.2772/[\sigma^2(F) + 0.0011|F_o^2|]$ were employed. The refinement converged to $R = 0.060$ and $R' = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0.060$. The final atomic coordinates are given in Table 1, bond lengths and angles in Table 2.*

Discussion. The Os atoms adopt a square-based pyramidal configuration and the cluster geometry as a whole resembles that of $\text{Fe}_5\text{C}(\text{CO})_{15}$ (Braye, Dahl, Hübel & Wampler, 1962) with which it is formally isoelectronic. The P atom donates four electrons to the cluster so that each Os atom obeys the 18-electron rule and this results in a 74-electron system. The observed metal geometry is in agreement with Wade's (1975) prediction for the structure of a 74-electron species. Fig. 1 shows the molecule.

The four Os atoms in the basal face are coplanar [maximum deviation 0.029 (4) Å] and the apical Os atom lies 1.981 (2) Å above this plane. The Os(basal)—Os(apical) bonds (mean 2.851 Å) are shorter than the Os(basal)—Os(basal) bonds (mean 2.898 Å). A similar, although less marked, trend has been observed in $\text{Os}_6\text{C}(\text{CO})_{16}(\text{H}_3\text{CC}:\text{CCH}_3)$ (Eady, Fernandez, Johnson, Lewis, Raithby & Sheldrick, 1978) which also contains a square-based pyramidal Os_5 fragment.

The P atom of the phosphinato ligand lies 1.185 (8) Å below the Os_4 square plane and bonds fairly symmetrically to the four metal atoms; the mean Os—P distance is 2.367 Å. The P—O vector makes an angle of $84.1(1)^\circ$ with the metal plane. μ_4 -Capping P donor ligands have been found in $\text{Co}_4(\text{CO})_{10}(\text{PC}_6\text{H}_5)_2$ (Simon

Table 1. Atom coordinates ($\times 10^4$)

	<i>x</i>	<i>y</i>	<i>z</i>
Os(1)	1585 (1)	1585 (1)	1210 (1)
Os(2)	2194 (1)	4202 (1)	813 (1)
Os(3)	1328 (1)	2321 (1)	-482 (1)
Os(4)	2557 (1)	197 (1)	296 (1)
Os(5)	3395 (1)	2073 (1)	1654 (1)
P(1)	2864 (6)	2545 (8)	211 (5)
O(1)	3485 (18)	3168 (32)	-289 (19)
C(1)	3919 (36)	2400 (63)	-758 (34)
C(11)	402 (17)	1066 (38)	731 (21)
O(11)	-315 (17)	670 (37)	449 (21)
C(12)	1850 (21)	-37 (30)	1887 (20)
O(12)	2046 (20)	-1022 (30)	2291 (19)
C(13)	1313 (28)	2605 (46)	2066 (25)
O(13)	1123 (27)	3192 (44)	2587 (26)
C(21)	2824 (23)	5353 (39)	1705 (23)
O(21)	3298 (26)	6104 (42)	2164 (26)
C(22)	2332 (25)	5510 (41)	72 (22)
O(22)	2404 (27)	6216 (45)	-460 (24)
C(23)	1153 (19)	5033 (40)	936 (23)
O(23)	493 (20)	5407 (44)	1023 (24)
C(31)	719 (27)	837 (41)	-1099 (28)
O(31)	304 (23)	-75 (38)	-1460 (23)
C(32)	1484 (21)	3202 (36)	-1413 (19)
O(32)	1610 (21)	3738 (37)	-1982 (19)
C(33)	300 (23)	3326 (48)	-507 (31)
O(33)	-367 (20)	3853 (39)	-545 (22)
C(41)	2726 (22)	-381 (40)	-687 (18)
O(41)	2764 (21)	-721 (38)	-1335 (18)
C(42)	1657 (22)	-1134 (37)	227 (25)
O(42)	1139 (22)	-1981 (35)	217 (24)
C(43)	3476 (22)	-1012 (36)	821 (23)
O(43)	4034 (22)	-1784 (38)	1137 (24)
C(51)	4521 (17)	2739 (35)	1721 (20)
O(51)	5190 (18)	3215 (35)	1717 (22)
C(52)	3334 (33)	3024 (55)	2585 (25)
O(52)	3345 (25)	3444 (40)	3232 (22)
C(53)	3827 (26)	519 (38)	2333 (25)
O(53)	4164 (21)	-439 (35)	2709 (21)

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34413 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

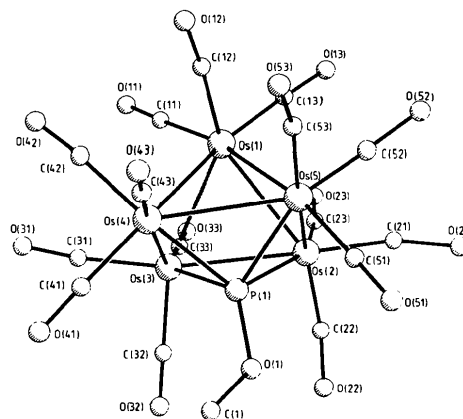


Fig. 1. $\text{Os}_5(\text{CO})_{15}\text{POCH}_3$, showing the atom numbering.

Table 2. Bond lengths (Å) and angles (°)

Os(2)—Os(1)	2.886 (3)	C(11)—Os(1)	1.889 (24)	P(1)—Os(4)—Os(3)	53.1 (2)	C(32)—Os(3)—Os(2)	101.2 (11)
Os(3)—Os(1)	2.866 (3)	C(12)—Os(1)	1.926 (26)	P(1)—Os(4)—Os(5)	52.1 (2)	C(32)—Os(3)—Os(4)	117.9 (11)
Os(4)—Os(1)	2.848 (3)	C(13)—Os(1)	1.915 (38)	P(1)—Os(5)—Os(1)	74.8 (2)	C(33)—Os(3)—Os(1)	90.8 (15)
Os(5)—Os(1)	2.803 (3)	P(1)—Os(2)	2.338 (9)	P(1)—Os(5)—Os(2)	51.5 (2)	C(33)—Os(3)—Os(2)	84.4 (15)
Os(3)—Os(2)	2.878 (3)	C(21)—Os(2)	1.910 (31)	P(1)—Os(5)—Os(4)	51.4 (2)	C(33)—Os(3)—Os(4)	147.6 (16)
Os(5)—Os(2)	2.896 (3)	C(22)—Os(2)	1.851 (35)	C(21)—Os(2)—P(1)	123.5 (12)	C(41)—Os(4)—Os(1)	152.3 (11)
Os(4)—Os(3)	2.887 (3)	C(23)—Os(2)	1.919 (29)	C(22)—Os(2)—P(1)	91.0 (13)	C(41)—Os(4)—Os(3)	94.1 (11)
Os(5)—Os(4)	2.932 (3)	P(1)—Os(3)	2.393 (8)	C(23)—Os(2)—P(1)	150.0 (11)	C(41)—Os(4)—Os(5)	137.8 (11)
C(31)—Os(3)	1.878 (33)	P(1)—Os(5)	2.381 (8)	C(31)—Os(3)—P(1)	128.8 (14)	C(42)—Os(4)—Os(1)	80.4 (13)
C(32)—Os(3)	1.880 (29)	C(51)—Os(5)	1.885 (25)	C(32)—Os(3)—P(1)	89.7 (10)	C(42)—Os(4)—Os(3)	93.7 (11)
C(33)—Os(3)	1.904 (32)	C(52)—Os(5)	1.859 (37)	C(33)—Os(3)—P(1)	135.7 (15)	C(42)—Os(4)—Os(5)	128.9 (12)
P(1)—Os(4)	2.357 (8)	C(53)—Os(5)	1.905 (32)	C(41)—Os(4)—P(1)	98.7 (12)	C(43)—Os(4)—Os(1)	121.0 (12)
C(41)—Os(4)	1.855 (29)	O(1)—P(1)	1.607 (32)	C(42)—Os(4)—P(1)	145.3 (12)	C(43)—Os(4)—Os(3)	172.4 (12)
C(42)—Os(4)	1.916 (30)	C(1)—O(1)	1.418 (64)	C(43)—Os(4)—P(1)	119.3 (12)	C(43)—Os(4)—Os(5)	85.0 (12)
C(43)—Os(4)	1.887 (29)			C(51)—Os(5)—P(1)	91.9 (10)	C(51)—Os(5)—Os(1)	164.8 (10)
				C(52)—Os(5)—P(1)	133.1 (16)	C(51)—Os(5)—Os(2)	105.2 (10)
				C(53)—Os(5)—P(1)	136.7 (14)	C(51)—Os(5)—Os(4)	117.7 (11)
Os(3)—Os(1)—Os(2)	60.0 (1)	C(11)—Os(1)—Os(2)	120.1 (11)	O(5)—P(1)—Os(2)	74.9 (3)	C(52)—Os(5)—Os(1)	89.9 (16)
Os(4)—Os(1)—Os(2)	90.9 (1)	C(11)—Os(1)—Os(3)	79.3 (11)	Os(4)—P(1)—Os(2)	121.0 (4)	C(52)—Os(5)—Os(2)	82.2 (16)
Os(4)—Os(1)—Os(3)	60.7 (1)	C(11)—Os(1)—Os(4)	106.2 (11)	Os(4)—P(1)—Os(3)	74.9 (2)	C(52)—Os(5)—Os(4)	148.3 (16)
Os(5)—Os(1)—Os(2)	61.2 (1)	C(11)—Os(1)—Os(5)	168.6 (11)	Os(5)—P(1)—Os(2)	75.7 (3)	C(53)—Os(5)—Os(1)	100.6 (12)
Os(5)—Os(1)—Os(3)	92.9 (1)	C(12)—Os(1)—Os(2)	146.9 (9)	Os(5)—P(1)—Os(3)	118.9 (4)	C(53)—Os(5)—Os(2)	159.3 (13)
Os(5)—Os(1)—Os(4)	62.5 (1)	C(12)—Os(1)—Os(3)	137.9 (11)	Os(5)—P(1)—Os(4)	76.5 (2)	C(53)—Os(5)—Os(4)	88.6 (13)
Os(3)—Os(2)—Os(1)	59.6 (1)	C(12)—Os(1)—Os(4)	82.8 (11)	O(1)—P(1)—Os(4)	113.9 (12)	C(12)—Os(1)—C(11)	92.7 (15)
Os(5)—Os(2)—Os(1)	58.0 (1)	C(12)—Os(1)—Os(5)	87.5 (10)	O(1)—P(1)—Os(3)	118.8 (11)	C(13)—Os(1)—C(12)	92.0 (18)
Os(5)—Os(2)—Os(3)	90.8 (1)	C(13)—Os(1)—Os(2)	83.5 (14)	O(1)—P(1)—Os(4)	125.0 (12)	C(22)—Os(2)—C(21)	89.5 (17)
Os(2)—Os(3)—Os(1)	60.3 (1)	C(13)—Os(1)—Os(3)	129.2 (14)	O(1)—P(1)—Os(5)	122.0 (11)	C(23)—Os(2)—C(21)	86.2 (16)
Os(4)—Os(3)—Os(1)	59.3 (1)	C(13)—Os(1)—Os(4)	161.1 (13)	C(1)—O(1)—P(1)	125.5 (33)	C(23)—Os(2)—C(22)	93.8 (17)
Os(4)—Os(3)—Os(2)	90.3 (1)	C(13)—Os(1)—Os(5)	99.2 (13)	O(11)—C(11)—Os(1)	176.2 (35)	C(23)—Os(2)—C(31)	93.6 (19)
Os(3)—Os(4)—Os(1)	60.0 (1)	C(21)—Os(2)—Os(1)	118.0 (12)	O(2)—C(12)—Os(1)	177.0 (32)	C(33)—Os(3)—C(31)	94.9 (20)
Os(5)—Os(4)—Os(1)	58.0 (1)	C(21)—Os(2)—Os(3)	176.1 (12)	O(2)—C(22)—Os(2)	172.3 (40)	C(33)—Os(3)—C(32)	94.5 (19)
Os(5)—Os(4)—Os(3)	89.9 (1)	C(21)—Os(2)—Os(5)	85.3 (12)	O(23)—C(23)—Os(2)	173.2 (39)	C(42)—Os(4)—C(41)	92.8 (17)
Os(2)—Os(5)—Os(1)	60.8 (1)	C(22)—Os(2)—Os(1)	152.5 (12)	O(31)—C(31)—Os(3)	176.1 (44)	C(43)—Os(4)—C(42)	93.9 (17)
Os(4)—Os(5)—Os(1)	59.5 (1)	C(22)—Os(2)—Os(3)	92.9 (12)	O(32)—C(32)—Os(3)	177.7 (32)	C(52)—Os(5)—C(51)	94.0 (19)
Os(4)—Os(5)—Os(2)	89.0 (1)	C(22)—Os(2)—Os(5)	129.3 (12)	O(33)—C(33)—Os(3)	174.9 (43)	C(53)—Os(5)—C(51)	94.1 (16)
P(1)—Os(2)—Os(1)	73.8 (2)	C(23)—Os(2)—Os(1)	89.1 (12)	O(41)—C(41)—Os(4)	174.7 (32)	C(53)—Os(5)—C(52)	89.2 (22)
P(1)—Os(2)—Os(3)	53.4 (2)	C(23)—Os(2)—Os(3)	96.8 (11)	O(42)—C(42)—Os(4)	176.6 (35)	O(52)—C(52)—Os(5)	169.9 (48)
P(1)—Os(2)—Os(5)	52.8 (2)	C(23)—Os(2)—Os(5)	135.9 (12)	O(43)—C(43)—Os(4)	178.6 (36)	O(53)—C(53)—Os(5)	172.7 (40)
P(1)—Os(3)—Os(1)	73.4 (2)	C(31)—Os(3)—Os(1)	105.0 (15)	O(51)—C(51)—Os(5)	175.2 (30)		
P(1)—Os(3)—Os(2)	51.7 (2)	C(31)—Os(3)—Os(2)	165.2 (15)				
P(1)—Os(3)—Os(4)	52.0 (2)	C(31)—Os(3)—Os(4)	82.4 (14)				
P(1)—Os(4)—Os(1)	74.3 (2)	C(32)—Os(3)—Os(1)	160.2 (10)				

& Dahl, 1973) and $\text{Ni}_8(\text{CO})_8(\text{PC}_6\text{H}_5)_6$ (Lower & Dahl, 1976). P donor ligands prefer to cap square cluster faces whereas C donor ligands, such as CCH_3 , may cap triangular or square faces as in $\text{Os}_6(\text{CO})_{16}(\mu_3\text{-CCH}_3)(\mu_4\text{-CCH}_3)$ (Eady, Fernandez, Johnson, Lewis, Raithby & Sheldrick, 1978).

Each Os atom is also bonded to three terminal CO groups so that all the metals are seven coordinate. The mean Os—C—O angle is $175(3)^\circ$ and the Os—C lengths do not deviate significantly from their mean of $1.89(2)$ Å. The CO groups are arranged so that they are staggered with respect to the Os—Os bonds.

The pyrolysis of $\text{Os}_3(\text{CO})_{11}\text{P}(\text{OCH}_3)_3$ causes a stepwise breakdown of the phosphite ligand as has been shown by the structural characterizations of $\text{HOs}_5(\text{CO})_{14}\text{OP}(\text{OCH}_3)_2$ (Fernandez, Johnson, Lewis, Raithby & Sheldrick, 1978), $\text{HOs}_5\text{C}(\text{CO})_{13}\text{OP}(\text{OCH}_3)\text{O}(\text{OCH}_3)_2$ (Orpen & Sheldrick, 1978), and now $\text{Os}_5(\text{CO})_{15}\text{POCH}_3$. Presumably, under more forcing conditions, the P atom would lose all its organic substituents to become a naked atom as in $[\text{Co}_6\text{P}(\text{CO})_{16}]^-$ (Chini, Ciani, Martinengo, Sironi, Longhetti & Heaton, 1979).

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References

- BRAYE, E. H., DAHL, L. F., HÜBEL, W. & WAMPLER, D. L. (1962). *J. Am. Chem. Soc.* **84**, 4633–4639.
- CHINI, P., CIANI, G., MARTINENGO, S., SIRONI, A., LONGHETTI, L. & HEATON, B. T. (1979). *Chem. Commun.* pp. 188–189.
- EADY, C. R., FERNANDEZ, J. M., JOHNSON, B. F. G., LEWIS, J., RAITHYBY, P. R. & SHELDRIK, G. M. (1978). *Chem. Commun.* pp. 421–423.
- FERNANDEZ, J. M., JOHNSON, B. F. G. & LEWIS, J. (1979). In preparation.
- FERNANDEZ, J. M., JOHNSON, B. F. G., LEWIS, J. & RAITHYBY, P. R. (1978). *Chem. Commun.* pp. 1015–1016.

FERNANDEZ, J. M., JOHNSON, B. F. G., LEWIS, J., RAITHY, P. R. & SHELDRIK, G. M. (1978). *Acta Cryst.* B34, 1994–1997.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

LOWER, L. D. & DAHL, L. F. (1976). *J. Am. Chem. Soc.* 98, 5046–5047.

ORPEN, A. G. & SHELDRIK, G. M. (1978). *Acta Cryst.* B34, 1992–1994.

SIMON, G. L. & DAHL, L. F. (1973). *J. Am. Chem. Soc.* 95, 2175–2183.

WADE, K. (1975). *Chem. Br.* 11, 177–183.

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1-(4-Bromobenzoyl)-2,5-dimethyl-4-morpholino-5-phenyl-4,5-dihydroimidazole

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Abstract. $C_{22}H_{24}BrN_3O_2$, $M_r = 442.4$, orthorhombic, $Pbca$, $a = 10.356$ (1), $b = 14.691$ (1), $c = 26.724$ (1) Å, $Z = 8$, $\mu(Cu K\alpha) = 3.22$ mm⁻¹, $D_x = 1.445$, $D_m = 1.438$ Mg m⁻³. The structure of the monoadduct obtained by reaction of 1-morpholino-2-phenylpropene with *N*-benzoyl-*N'*-chloroacetamide has been established to be that of the title compound. Bond-length extensions [up to 1.578 (3) Å for C(4)–C(5)] and bond-angle deformations are caused by strong steric hindrance between the substituent groups.

Introduction. The reaction (1) of 1-morpholino-2-phenylpropene with *N*-aroyl-*N'*-chloroacetamide gives 1-aroyl-4,5-dihydroimidazole derivatives whose structure could not be determined unequivocally by chemical and spectroscopic methods (Stradi, 1978). In fact, two possible regioisomers can be obtained, 1-aroyl-2,5-dimethyl-4-morpholino-5-phenyl-4,5-dihydroimidazole (I), or 1-aroyl-2,4-dimethyl-5-morpholino-4-phenyl-4,5-dihydroimidazole (II). To establish the structure of the adduct, its X-ray analysis has been undertaken.

Cell dimensions (see *Abstract*) were obtained by a least-squares fit to the $\sin^2 \theta$ values of 60 hkl reflexions

measured on a diffractometer. The space group $Pbca$ was indicated by the absences $0kl$ with k odd, $h0l$ with l odd, and $hk0$ with h odd. The density was measured by flotation in a dilute K_2HgI_4 solution. For the data collection a crystal $0.34 \times 0.25 \times 0.22$ mm was mounted on a computer-controlled four-circle diffractometer; graphite-monochromated Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å) and a variable θ - 2θ scan technique were used. Background measurements were taken at both ends of the scan range, each for a time equal to one half of the scan time. The periodic measurement of two standard reflexions showed no appreciable trend. Out of 3857 measured reflexions ($2\theta \leq 140^\circ$), 802 having $I < \sigma(I)_{c.s.}$ were assigned zero weight; all other reflexions were assigned variances $\sigma^2(I)$ based on counting statistics plus the additional term $(0.03 \text{ scan count})^2$. Diffraction data were corrected for absorption effects (Wehe, Busing & Levy, 1962), and then for Lorentz and polarization factors.

The structure was solved by interpretation of the Patterson map to derive the Br atom position. Subsequent application of direct methods led to an E map from which all non-hydrogen atoms could be clearly

