

tively large experimental uncertainties, the carbide lies on the line joining Os(1) and Os(3), and in the plane of the other three Os atoms. The cluster may thus be described as a trigonal bipyramid, distorted considerably from equilateral geometry in the equatorial plane (as shown by the angles given in Table 3).

Potential-energy calculations based on the ligand configuration indicate that the hydride bridges the edge Os(12)—Os(15) [Os(22)—Os(25) in the second molecule]; suitable holes in the close-packed carbonyl arrangement can be seen in Figs. 1 and 2. This assignment is consistent with the relatively large P—H coupling (36 Hz) observed in the NMR spectrum; the H and P atoms are both bonded to the same Os. Unusually, the hydride does not bridge the longest Os—Os bond. Except for the orientation of one of the

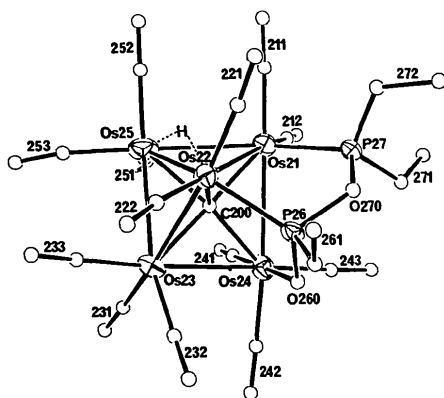


Fig. 2. Molecule 2, showing 50% probability ellipsoids for the anisotropic atoms.

OCH_3 groups, the overall molecular geometry is very similar for the two independent molecules. However, the bond lengths involving Os(25) are in some cases significantly different from those involving Os(15) [e.g. Os(25)—Os(22) 2.902 (3), Os(15)—Os(12) 2.927 (3), Os(25)—Os(23) 2.890 (4), Os(15)—Os(13) 2.866 (4) Å]. Os(25) is relatively anisotropic, and the orientation of C(251)—O(251) is significantly different from that of C(151)—O(151). These observations are all consistent with some disorder of the bridging hydride in the second molecule, *i.e.* with the hydride bridging Os(25)—Os(23) rather than Os(25)—Os(22) in some molecules. This type of disorder might well be fluxional on an NMR time scale in solution, *i.e.* only an average hydride chemical shift and P—H coupling would be seen.

We thank Dr J. Fernandez for providing the compound, the Science Research Council for a contribution towards the cost of the diffractometer, and BP Ltd for financial support to AGO. Calculations were performed with the Cambridge University IBM 370/165 computer and programs written by the authors.

References

- BRUCE, M. I., HOWARD, J. A. K., NOWELL, I. W., SHAW, G. & WOODWARD, P. W. (1972). *Chem. Commun.* pp. 1041–1042.
 EADY, C. R., JOHNSON, B. F. G. & LEWIS, J. (1975). *J. Chem. Soc. Dalton Trans.* pp. 2606–2611.
 FERNANDEZ, J. M., JOHNSON, B. F. G. & LEWIS, J. (1977). Private communication.
 WADE, K. (1975). *Chem. Br.* **11**, 177–183.

Acta Cryst. (1978). **B34**, 1994–1997

Carbidotetradecacarbonylhydrido(dimethyl phosphonato)pentaosmium, $\text{HOs}_5\text{C}(\text{CO})_{14}[\text{OP}(\text{OCH}_3)_2]$

BY JUAN M. FERNANDEZ, BRIAN F. G. JOHNSON, JACK LEWIS,
PAUL R. RAITHY AND GEORGE M. SHELDRIK

University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England

(Received 21 January 1978; accepted 4 February 1978)

Abstract. $\text{C}_{17}\text{H}_7\text{O}_{17}\text{POs}_5$, monoclinic, $P2_1/n$, $a = 9.200$ (4), $b = 31.809$ (13), $c = 9.486$ (4) Å, $\beta = 103.13$ (3)°, $U = 2703.4$ Å³, $Z = 4$, $D_x = 3.60$ g cm⁻³, $\mu(\text{Mo } K\alpha) = 235.4$ cm⁻¹. The structure was refined to an R of 0.039 for 4781 unique diffractometer data. The Os atoms define a distorted trigonal

bipyramid, with a carbide lying in the equatorial plane. The phosphonate ligand coordinates to two Os atoms to form an Os—P—O—Os linkage.

Introduction. The pyrolysis of $\text{Os}_3(\text{CO})_{11}[\text{P}(\text{OCH}_3)_3]$ yields a number of pentanuclear clusters containing

uncommon P donor ligands (Fernandez, Johnson & Lewis, 1977). The characterization of these compounds by spectroscopic techniques has proved difficult, and the crystal structure determination of this product has been undertaken to establish the molecular geometry. The structure of another derivative has been reported (Orpen & Sheldrick, 1978).

Crystals were obtained as yellow, elongated rectangular blocks from ethyl acetate/hexane. 6682 intensities were recorded on a Syntex P2₁ four-circle diffractometer, with graphite-monochromated Mo K α radiation, a θ -2 θ scan procedure, and a crystal 0.28 × 0.11 × 0.09 mm. Lp and semi-empirical absorption corrections were applied (based on azimuthal scan data from 36 strong reflexions). Transmission factors ranged from 0.418 to 0.736. Cell dimensions were

obtained by least squares from the 2 θ angles for 15 strong high-angle (20.0 < 2 θ ≤ 30.0°) reflexions.

The Os atoms were located by multisolution Σ_2 sign expansion, and the light atoms from difference syntheses; no attempt was made to locate the H atoms. The structure was refined by blocked-cascade least squares, with the weighting scheme $w = 1/[\sigma^2(F) + 0.00115|F|^2]$. The Os, and the P, O and C atoms in the dimethyl phosphonate ligand were assigned anisotropic thermal parameters; an empirical extinction parameter x which refined to 0.00011 (1) was also included; F_c is multiplied by (1 - 0.0001 x $F_o^2/\sin \theta$). Complex neutral-atom scattering factors were employed. The refinement converged to $R_w = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0.041$ and $R = 0.039$. Final atomic coordinates and isotropic thermal parameters are listed in Table 1, bond lengths and angles in Tables 2 and 3.*

Table 1. Atom coordinates ($\times 10^4$) and isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>
Os(1)	4185 (1)	966 (1)	1749 (1)	*
Os(2)	3278 (1)	957 (1)	4451 (1)	*
Os(3)	2726 (1)	1773 (1)	1199 (1)	*
Os(4)	5005 (1)	1655 (1)	3799 (1)	*
Os(5)	618 (1)	1333 (1)	2589 (1)	*
P(1)	1978 (4)	660 (1)	617 (3)	*
O(1)	1765 (12)	169 (3)	820 (10)	*
O(2)	1711 (13)	711 (4)	-1103 (9)	*
O(3)	634 (11)	841 (3)	1084 (10)	*
C(1)	2588 (24)	-137 (5)	212 (24)	*
C(2)	261 (24)	579 (7)	-2049 (17)	*
C(10)	2962 (15)	1360 (4)	2806 (13)	28 (3)
C(11)	4904 (18)	992 (4)	8 (15)	36 (3)
O(11)	5310 (14)	1010 (3)	-1020 (12)	49 (3)
C(12)	5189 (15)	466 (4)	2370 (13)	27 (3)
O(12)	5804 (13)	145 (3)	2753 (12)	47 (3)
C(21)	5040 (18)	683 (5)	5470 (16)	39 (3)
O(21)	6109 (15)	510 (4)	6057 (14)	63 (3)
C(22)	2817 (18)	1204 (5)	6127 (16)	41 (3)
O(22)	2571 (15)	1385 (4)	7075 (13)	61 (3)
C(23)	2200 (19)	444 (5)	4387 (16)	43 (4)
O(23)	1485 (15)	149 (4)	4376 (13)	57 (3)
C(31)	1206 (19)	1735 (4)	-519 (16)	39 (3)
O(31)	248 (16)	1729 (4)	-1558 (14)	65 (4)
C(32)	2268 (18)	2337 (5)	1701 (16)	40 (3)
O(32)	2107 (15)	2662 (4)	2114 (13)	64 (3)
C(33)	4093 (19)	1941 (5)	49 (17)	44 (4)
O(33)	4899 (15)	2033 (4)	-643 (13)	59 (3)
C(41)	6208 (20)	2063 (5)	3095 (17)	46 (4)
O(41)	6858 (18)	2331 (4)	2720 (15)	74 (4)
C(42)	6563 (18)	1490 (5)	5338 (16)	40 (3)
O(42)	7486 (16)	1397 (4)	6358 (14)	67 (4)
C(43)	4204 (21)	2050 (5)	4904 (18)	49 (4)
O(43)	3752 (16)	2289 (4)	5602 (13)	62 (3)
C(51)	-1068 (19)	1565 (5)	1251 (17)	42 (3)
O(51)	-2062 (17)	1682 (4)	407 (15)	69 (4)
C(52)	-597 (19)	990 (5)	3500 (16)	42 (3)
O(52)	-1320 (16)	783 (4)	4023 (14)	70 (4)
C(53)	542 (19)	1769 (5)	3894 (17)	44 (4)
O(53)	462 (15)	2028 (4)	4729 (14)	62 (3)

* Anisotropic thermal parameters have been deposited.

Discussion. The geometry of the metal framework (Fig. 1) is derived from that of Os₅(CO)₁₆ (Reichert & Sheldrick, 1977) by the breaking of one Os(equatorial)—Os(equatorial) and one Os(equatorial)—Os(axial) bond; Os(1)⋯Os(5) 3.74 and Os(2)⋯Os(3) 3.98 Å. In terms of a skeletal electron-counting scheme (Eady, Johnson & Lewis, 1975), Os₅(CO)₁₆ is a 72-electron system. In HO₅C(CO)₁₄[OP(OCH₃)₂], the carbide, a

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33387 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. Bond lengths (Å)

Os(1)—Os(2)	2.870 (2)	Os(1)—C(11)	1.915 (16)
Os(1)—Os(3)	2.889 (2)	Os(1)—C(12)	1.868 (12)
Os(1)—Os(4)	2.914 (2)	Os(2)—C(21)	1.901 (15)
Os(2)—Os(4)	2.879 (2)	Os(2)—C(22)	1.905 (16)
Os(2)—Os(5)	2.928 (2)	Os(2)—C(23)	1.904 (16)
Os(3)—Os(4)	2.876 (2)	Os(3)—C(31)	1.895 (14)
Os(3)—Os(5)	2.937 (2)	Os(3)—C(32)	1.927 (15)
Os(1)—P(1)	2.288 (3)	Os(3)—C(33)	1.918 (19)
Os(5)—O(3)	2.122 (10)	Os(4)—C(41)	1.922 (18)
Os(1)—C(10)	2.086 (14)	Os(4)—C(42)	1.874 (14)
Os(2)—C(10)	1.988 (12)	Os(4)—C(43)	1.888 (18)
Os(3)—C(10)	1.988 (12)	Os(5)—C(51)	1.915 (15)
Os(4)—C(10)	2.121 (12)	Os(5)—C(52)	1.904 (17)
Os(5)—C(10)	2.121 (14)	Os(5)—C(53)	1.870 (16)
P(1)—O(1)	1.590 (9)	C(31)—O(31)	1.164 (19)
P(1)—O(2)	1.603 (9)	C(32)—O(32)	1.126 (20)
P(1)—O(3)	1.519 (11)	C(33)—O(33)	1.135 (24)
O(1)—C(1)	1.434 (24)	C(41)—O(41)	1.142 (23)
O(2)—C(2)	1.489 (22)	C(42)—O(42)	1.171 (19)
C(11)—O(11)	1.122 (20)	C(43)—O(43)	1.147 (23)
C(12)—O(12)	1.182 (16)	C(51)—O(51)	1.132 (20)
C(21)—O(21)	1.155 (20)	C(52)—O(52)	1.127 (23)
C(22)—O(22)	1.134 (21)	C(53)—O(53)	1.157 (20)
C(23)—O(23)	1.144 (20)		

Table 3. Bond angles (°)

Os(2)—Os(1)—Os(3)	87.3 (1)	Os(2)—Os(1)—C(11)	176.2 (4)
Os(2)—Os(1)—Os(4)	59.7 (1)	Os(3)—Os(1)—C(11)	92.5 (4)
Os(3)—Os(1)—Os(4)	59.4 (1)	Os(4)—Os(1)—C(11)	117.1 (4)
Os(1)—Os(2)—Os(4)	60.9 (1)	Os(2)—Os(1)—C(12)	86.2 (4)
Os(1)—Os(2)—Os(5)	80.2 (1)	Os(3)—Os(1)—C(12)	172.2 (4)
Os(4)—Os(2)—Os(5)	88.9 (1)	Os(4)—Os(1)—C(12)	113.4 (4)
Os(1)—Os(3)—Os(4)	60.7 (1)	Os(1)—Os(2)—C(21)	94.1 (5)
Os(1)—Os(3)—Os(5)	79.8 (1)	Os(4)—Os(2)—C(21)	90.9 (5)
Os(4)—Os(3)—Os(5)	88.8 (1)	Os(5)—Os(2)—C(21)	173.6 (5)
Os(1)—Os(4)—Os(2)	59.4 (1)	Os(1)—Os(2)—C(22)	155.0 (5)
Os(1)—Os(4)—Os(3)	59.9 (1)	Os(4)—Os(2)—C(22)	95.7 (5)
Os(2)—Os(4)—Os(3)	87.4 (1)	Os(5)—Os(2)—C(22)	91.2 (4)
Os(2)—Os(5)—Os(3)	85.3 (1)	Os(1)—Os(2)—C(23)	103.6 (5)
Os(2)—Os(1)—P(1)	90.4 (1)	Os(4)—Os(2)—C(23)	164.5 (5)
Os(3)—Os(1)—P(1)	88.2 (1)	Os(5)—Os(2)—C(23)	88.5 (4)
Os(4)—Os(1)—P(1)	134.5 (1)	Os(1)—Os(3)—C(31)	108.7 (4)
Os(2)—Os(1)—C(10)	43.8 (3)	Os(4)—Os(3)—C(31)	168.8 (4)
Os(3)—Os(1)—C(10)	43.5 (3)	Os(5)—Os(3)—C(31)	85.5 (5)
Os(4)—Os(1)—C(10)	46.7 (3)	Os(1)—Os(3)—C(32)	154.3 (4)
Os(1)—Os(2)—C(10)	46.6 (4)	Os(4)—Os(3)—C(32)	93.8 (4)
Os(4)—Os(2)—C(10)	47.4 (4)	Os(5)—Os(3)—C(32)	97.4 (5)
Os(5)—Os(2)—C(10)	46.4 (4)	Os(1)—Os(3)—C(33)	90.7 (5)
Os(1)—Os(3)—C(10)	46.2 (4)	Os(4)—Os(3)—C(33)	94.6 (4)
Os(4)—Os(3)—C(10)	47.5 (4)	Os(5)—Os(3)—C(33)	166.7 (5)
Os(5)—Os(3)—C(10)	46.2 (4)	Os(1)—Os(4)—C(41)	111.0 (5)
Os(1)—Os(4)—C(10)	45.7 (4)	Os(2)—Os(4)—C(41)	170.2 (5)
Os(2)—Os(4)—C(10)	43.7 (3)	Os(3)—Os(4)—C(41)	88.8 (5)
Os(3)—Os(4)—C(10)	43.7 (3)	Os(1)—Os(4)—C(42)	110.2 (5)
Os(2)—Os(5)—C(10)	42.8 (3)	Os(2)—Os(4)—C(42)	88.8 (5)
Os(3)—Os(5)—C(10)	42.6 (3)	Os(3)—Os(4)—C(42)	169.9 (5)
O(3)—Os(5)—C(10)	86.1 (4)	Os(1)—Os(4)—C(43)	142.7 (5)
P(1)—Os(1)—C(10)	87.9 (3)	Os(2)—Os(4)—C(43)	95.2 (6)
Os(2)—Os(5)—O(3)	88.4 (2)	Os(3)—Os(4)—C(43)	95.1 (5)
Os(3)—Os(5)—O(3)	86.7 (3)	Os(2)—Os(5)—C(51)	175.8 (5)
Os(1)—P(1)—O(1)	118.8 (4)	Os(3)—Os(5)—C(51)	92.1 (5)
Os(1)—P(1)—O(2)	110.4 (5)	Os(2)—Os(5)—C(52)	89.3 (5)
O(1)—P(1)—O(2)	103.3 (5)	Os(3)—Os(5)—C(52)	173.1 (5)
Os(1)—P(1)—O(3)	113.3 (4)	Os(2)—Os(5)—C(53)	93.2 (5)
O(1)—P(1)—O(3)	101.9 (6)	Os(3)—Os(5)—C(53)	94.1 (5)
O(2)—P(1)—O(3)	108.1 (6)	P(1)—Os(1)—C(11)	93.3 (4)
P(1)—O(1)—C(11)	121.9 (11)	C(10)—Os(1)—C(11)	135.9 (5)
P(1)—O(2)—C(11)	119.6 (11)	P(1)—Os(1)—C(12)	96.1 (4)
Os(5)—O(3)—P(1)	127.4 (6)	C(10)—Os(1)—C(12)	130.0 (5)
Os(1)—C(10)—Os(2)	89.6 (5)	C(11)—Os(1)—C(12)	93.8 (6)
Os(1)—C(10)—Os(3)	90.3 (5)	C(10)—Os(2)—C(21)	130.6 (7)
Os(2)—C(10)—Os(3)	177.6 (8)	C(10)—Os(2)—C(22)	111.7 (6)
Os(1)—C(10)—Os(4)	87.7 (5)	C(10)—Os(2)—C(23)	123.0 (6)
Os(2)—C(10)—Os(4)	88.9 (4)	C(21)—Os(2)—C(22)	95.2 (6)
Os(3)—C(10)—Os(4)	88.8 (5)	C(21)—Os(2)—C(23)	90.0 (6)
Os(1)—C(10)—Os(5)	125.3 (6)	C(22)—Os(2)—C(23)	99.6 (7)
Os(2)—C(10)—Os(5)	90.8 (6)	C(10)—Os(3)—C(31)	123.2 (6)
Os(3)—C(10)—Os(5)	91.2 (5)	C(10)—Os(3)—C(32)	114.7 (6)
Os(4)—C(10)—Os(5)	147.1 (7)	C(10)—Os(3)—C(33)	130.4 (6)
Os(1)—C(11)—O(11)	179.1 (11)	C(31)—Os(3)—C(32)	96.4 (6)
Os(1)—C(12)—O(12)	178.9 (9)	C(31)—Os(3)—C(33)	88.8 (7)
Os(2)—C(21)—O(21)	178.2 (12)	C(32)—Os(3)—C(33)	95.2 (7)
Os(2)—C(22)—O(22)	173.7 (14)	C(10)—Os(4)—C(41)	131.9 (6)

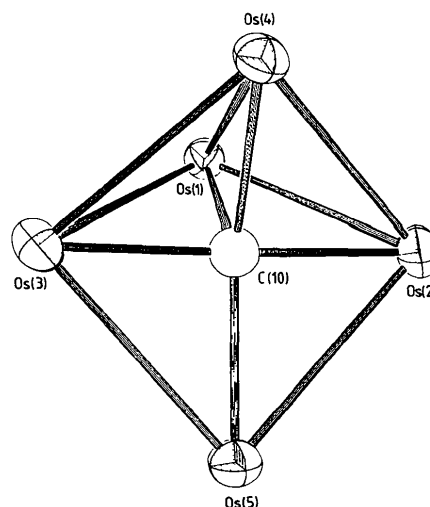
Table 3 (cont.)

Os(2)—C(23)—O(23)	175.8 (16)	C(10)—Os(4)—C(42)	131.7 (6)
Os(3)—C(31)—O(31)	177.0 (13)	C(10)—Os(4)—C(43)	97.1 (6)
Os(3)—C(32)—O(32)	173.2 (13)	C(41)—Os(4)—C(42)	93.5 (7)
Os(3)—C(33)—O(33)	178.8 (10)	C(41)—Os(4)—C(43)	94.1 (8)
Os(4)—C(41)—O(41)	174.4 (16)	C(42)—Os(4)—C(43)	94.6 (7)
Os(4)—C(42)—O(42)	175.8 (16)	O(3)—Os(5)—C(51)	88.1 (5)
Os(4)—C(43)—O(43)	178.2 (16)	O(3)—Os(5)—C(52)	88.7 (5)
Os(5)—C(51)—O(51)	175.8 (14)	O(3)—Os(5)—C(53)	178.3 (6)
Os(5)—C(52)—O(52)	179.1 (11)	C(10)—Os(5)—C(51)	134.5 (6)
Os(5)—C(53)—O(53)	177.2 (14)	C(10)—Os(5)—C(52)	131.9 (6)
C(51)—Os(5)—C(52)	93.0 (7)	C(10)—Os(5)—C(53)	95.5 (6)
C(52)—Os(5)—C(53)	90.7 (7)	C(51)—Os(5)—C(53)	90.4 (7)

occurs in HO₅C(CO)₁₃[OP(OCH₃)OP(OCH₃)₂] (Orpen & Sheldrick, 1978).

The two longest Os—Os bonds are those involving Os(5), which may help to redress any electron imbalance resulting from the coordination of three CO ligands and the phosphonate O to this atom. The long Os(1)—Os(4) distance may reveal the position of an edge-bridging hydride; the carbonyl ligands bend away from this bond (Fig. 2), with an average *cis* Os—Os—C(carbonyl) bond angle at these two Os atoms of 113°, compared with 93° for the remainder. A similar ligand geometry is observed for the hydride-bridged Os—Os bond in H₂Os₃(CO)₁₁ (Churchill & DeBoer, 1977). The proposed position of the hydride, bonded to the same Os as the P atom, is in agreement with the ¹H NMR data which show a large splitting of the hydride signal (*J* = 35.1 Hz) due to the ³¹P nucleus.

The carbide sits in the Os(1)Os(2)Os(3) triangle (deviation from plane 0.03 Å), lying close to the Os(2)···Os(3) non-bonded edge, though it coordinates to all five Os atoms. It also lies on the pseudo mirror plane passing through Os(4) and the planar Os(1)—P(1)—O(3)—Os(5) system (maximum deviation 0.005

Fig. 1. The Os₅C geometry.

four-electron donor, has the effect of replacing the two CO groups, but, with the OP(OCH₃)₂ ligand acting as a three-electron donor, and a further electron from the hydride, a 76-electron system results. The inclusion of two additional electron pairs requires the observed rupture of two metal—metal bonds. A similar situation

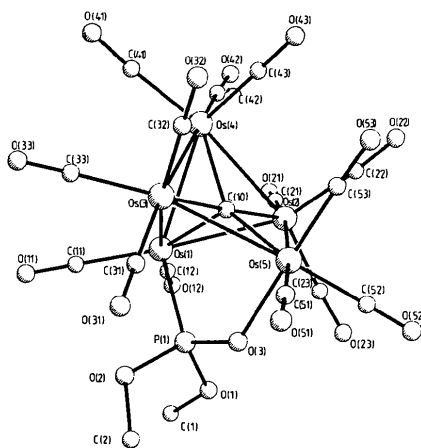


Fig. 2. HOs₅C(CO)₁₄[OP(OCH₃)₂], showing the atom numbering.

Å). Os(1)—P(1) and P(1)—O(3) are shorter than would be expected for pure σ bonds, while Os(5)—O(3) is long. Similar trends have been observed in the Ru—P—O—Ru system of HRu₂(CO)₃[P(OC₆H₄)(OPh)₂][OP(OPh)₂] (Bruce, Howard, Nowell, Shaw & Woodward, 1972), and the short bonds have been explained in terms of π interactions.

Acta Cryst. (1978). B34, 1997–1999

Potassium Dicyano[(dithionitrito-S)amido]nickelate(II)

BY JOHANNES WEISS

Anorganisch-Chemisches Institut der Universität Heidelberg, Im Neuenheimer Feld 270, D 6900 Heidelberg 1, Federal Republic of Germany

(Received 5 December 1977; accepted 20 January 1978)

Abstract. K[Ni(CN)₂HN₂S₂], monoclinic, $P2_1/c$, $a = 4.203$ (2), $b = 18.286$ (2), $c = 10.440$ (2) Å, $\beta = 95.64$ (3)°, $V = 798.5$ Å³, $Z = 4$, $D_x = 2.04$ g cm⁻³. Refined to $R = 0.065$ for 1452 observed reflexions. The Ni is coordinated in a square-planar manner by the C atoms of the CN groups and one S and one N atom of the S₂N₂H chelate ligand.

Introduction. There are several well-characterized complexes containing two chelating S₂N₂H ligands (Weiss, 1966; Weiss & Thewalt, 1968; Mayer & Weiss, 1978), but the title compound is the only stable anionic complex with one chelating S₂N₂H group. Only one complex containing one S₂N₂ (or S₂N₂H?) ligand

The CO groups are all linear, and the mean Os—C and C—O lengths of 1.900 and 1.15 Å, respectively, are in good agreement with the 1.89 and 1.16 Å for Os₅(CO)₁₆ (Reichert & Sheldrick, 1977).

We thank the Science Research Council for financial support. Calculations were performed with the Cambridge University IBM 370/165 computer and programs written by GMS.

References

- BRUCE, M. I., HOWARD, J. A. K., NOWELL, I. W., SHAW, G. & WOODWARD, P. W. (1972). *Chem. Commun.* pp. 1041–1042.
- CHURCHILL, M. R. & DEBOER, B. G. (1977). *Inorg. Chem.* **16**, 878–884.
- EADY, C. R., JOHNSON, B. F. G. & LEWIS, J. (1975). *J. Chem. Soc. Dalton Trans.* pp. 2606–2611.
- FERNANDEZ, J. M., JOHNSON, B. F. G. & LEWIS, J. (1977). Private communication.
- ORPEN, A. G. & SHELDRICK, G. M. (1978). *Acta Cryst.* B34, 1992–1994.
- REICHERT, B. E. & SHELDRICK, G. M. (1977). *Acta Cryst.* B33, 173–175.

has been described: Cu(S₂N₂)X₂, X = Cl, Br (Fluck & Goehring, 1956), but crystals could not be obtained.

The title compound was prepared as described by Weiss (1957). Suitable crystals were obtained by slow evaporation of an ethanolic solution. Lattice constants were calculated from the θ values of 37 reflexions, determined on an automatic single-crystal diffractometer (Siemens), by least squares (Berdiesinski & Nuber, 1966). Intensity measurements on the Siemens diffractometer with the five-value method and θ - 2θ scans with Mo $K\alpha$ radiation up to $2\theta = 64^\circ$ yielded 1452 observed reflexions. Reflexions with $I < 2.58\sigma(I)$ were classified as unobserved. The intensities were corrected for Lorentz and polarization factors only.