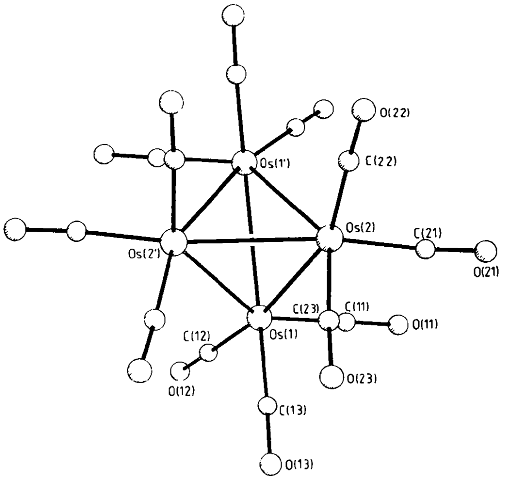

Fig. 1. The $[H_3Os_4(CO)_{12}]^-$ anion.

that one hydride lies on the twofold axis, bridging Os(1)–Os(1′), while the other two bridge Os(1)–Os(2′) and Os(2)–Os(1′), respectively. Thus, this anion is essentially isostructural with the C_2 isomer of $[H_3Ru_4(CO)_{12}]^-$, for which it has been shown that both C_2 and C_{3v} isomers exist in the solid state (Jackson, Johnson, Lewis, McPartlin & Nelson, 1978).

None of the Os—C or C—O lengths deviate from the respective means of 1·863 and 1·178 Å by more than 1·3σ, and the average Os—C—O angle is 177·0°. These lengths are not significantly different from the mean Os—C and C—O distances of 1·89 (4) and 1·16 (4) Å in $Os_5(CO)_{16}$ (Reichert & Sheldrick, 1977).

The tetramethylammonium cation adopts the expected configuration, having a twofold crystallographic axis passing through the N atom.

We thank the SRC for financial support (to PRR) and the Universidad Metropolitana, Venezuela, for a grant (to CZ). We are grateful to Johnson, Matthey and Co. Ltd for the loan of OsO_4. Calculations were performed with the Cambridge University IBM 370/165 computer and programs written by Dr G. M. Sheldrick; the figure was drawn with *PLUTO* written by Dr W. D. S. Motherwell.

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Bis(triphenylphosphine)iminium μ_2-Hydrido-μ_3-sulphido-nonacarbonyltriosmate

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(*Received* 29 *August* 1978; *accepted* 19 *September* 1978)

Abstract. $[(C_{18}H_{15}P)_2N]$ $[Os_3(CO)_9HS]$, monoclinic, $P2_1/c$, $a = 9{\cdot}600\ (3)$, $b = 30{\cdot}074\ (10)$, $c = 15{\cdot}946\ (8)$ Å, $\beta = 101{\cdot}15\ (3)°$, $U = 4516{\cdot}9$ Å^3, $Z = 4$, $D_c = 2{\cdot}05$ Mg m^{-3}, μ(Mo $K\alpha$) = 8·562 mm^{-1}. The structure was refined to an R of 0·057 for 3186 unique diffractometer data. In the anion the three Os atoms define an isosceles triangle, which is capped by the S atom. The carbonyl groups are all terminal, and the ligand distribution suggests that the hydride bridges the long Os—Os edge.

Introduction. The structure determination of the title compound was undertaken as one of a series in our investigations of the changes in cluster geometry in Os_3S systems, due to variations in ionic charge and the number of hydrides present.

The anion was prepared in good yield by treating $H_2Os_3(CO)_9S$ with methanolic KOH, followed by precipitation as the yellow bis(triphenylphosphine)iminium salt (Johnson, Lewis, Pippard & Raithby, 1978). 5705 intensities were measured ($3{\cdot}0 < 2\theta \leq$

50·0°) on a Syntex $P2_1$ four-circle diffractometer, with graphite-monochromated Mo $K\alpha$ radiation, a θ–2θ scan procedure, and a crystal 0·123 × 0·175 × 0·158 mm. Lp and semi-empirical absorption corrections, based on a pseudo-ellipsoid model and 436 azimuthal scan data from ten independent reflections, were applied; transmission factors ranged from 0·204 to 0·248. The data were averaged to give 3186 unique observed intensities [$I > 2{\cdot}5\sigma(I)$]. Cell dimensions were derived from the angular measurements of 15 strong reflections ($20{\cdot}0 < 2\theta < 30{\cdot}0°$).

The Os atoms were located by multisolution $\sum_2$ sign expansion, and the other non-hydrogen atoms from a subsequent difference synthesis. The structure was refined by the blocked-cascade least-squares method (Os and P anisotropic, C, N and O isotropic). Phenyl groups were refined as rigid groups with C—C 1·395, C—H 1·08 Å, C—C—C and C—C—H 120·0°; phenyl H atoms were assigned a common isotropic temperature factor fixed at 0·123 Å^2. The hydridic H atom was not located. An empirical extinction parameter x which refined to a value of 0·00006 (2) was also included; F_c is multiplied by $(1 - 0{\cdot}0001xF_c^2/\sin\theta)$. Complex neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974) and a weighting scheme $w = [\sigma^2(F) + 0{\cdot}001|F_o|^2]^{-1}$, were employed. The refinement converged to $R = 0{\cdot}057$ and $R' = [\sum w^{1/2}\Delta/\sum w^{1/2}|F_o|] = 0{\cdot}057$. The final atomic coordinates for the non-hydrogen atoms are listed in Table 1 and those for the phenyl H atoms in Table 2. Some important bond lengths and angles are presented in Table 3.*

Discussion. The pseudo-tetrahedral Os_3S core of the anion has non-crystallographic C_s (m) symmetry. In terms of skeletal electron counting (Eady, Johnson & Lewis, 1975) the S atom must act as a four-electron donor which results in an 'electron-precise' 48e system.

Table 1. *Atom coordinates* (×10^4)

	x	y	z
Os(1)	7060 (1)	1242 (1)	2602 (1)
Os(2)	4104 (1)	1500 (1)	2117 (1)
Os(3)	4908 (1)	617 (1)	2334 (1)
S(1)	5246 (6)	1159 (3)	3432 (4)
C(11)	8383 (31)	856 (11)	3094 (20)
O(11)	9086 (22)	557 (7)	3509 (14)
C(12)	8093 (31)	1746 (11)	2960 (19)
O(12)	8766 (24)	2067 (8)	3261 (14)
C(13)	7741 (28)	1146 (9)	1569 (20)
O(13)	8051 (23)	1120 (7)	925 (15)
C(21)	3911 (38)	2101 (13)	2355 (22)
O(21)	3737 (30)	2478 (10)	2373 (18)
C(22)	2255 (29)	1336 (10)	2224 (18)
O(22)	1059 (28)	1295 (8)	2188 (16)
C(23)	3708 (27)	1503 (9)	924 (18)
O(23)	3575 (20)	1482 (6)	175 (13)
C(31)	3134 (24)	376 (8)	2372 (15)
O(31)	2004 (23)	213 (7)	2329 (14)
C(32)	5980 (30)	101 (10)	2793 (19)
O(32)	6626 (22)	−185 (7)	3145 (13)
C(33)	4960 (27)	506 (9)	1188 (17)
O(33)	5021 (19)	446 (6)	496 (12)
N(1)	−39 (19)	1420 (6)	−2576 (11)
P(1)	−369 (6)	1346 (2)	−1652 (4)
P(2)	−627 (6)	1364 (2)	−3551 (4)
C(101)	342 (16)	1812 (6)	−1006 (8)
C(102)	1331 (16)	2090 (6)	−1278 (8)
C(103)	1961 (16)	2435 (6)	−751 (8)
C(104)	1602 (16)	2501 (6)	46 (8)
C(105)	612 (16)	2224 (6)	317 (8)
C(106)	−18 (16)	1879 (6)	−209 (8)
C(111)	544 (17)	859 (5)	−1193 (10)
C(112)	662 (17)	778 (5)	−321 (10)
C(113)	1319 (17)	390 (5)	40 (10)
C(114)	1859 (17)	84 (5)	−472 (10)
C(115)	1741 (17)	165 (5)	−1344 (10)
C(116)	1084 (17)	553 (5)	−1705 (10)
C(121)	−2191 (16)	1278 (4)	−1576 (10)
C(122)	−2768 (16)	856 (4)	−1524 (10)
C(123)	−4201 (16)	809 (4)	−1487 (10)
C(124)	−5057 (16)	1185 (4)	−1501 (10)
C(125)	−4480 (16)	1607 (4)	−1553 (10)
C(126)	−3047 (16)	1654 (4)	−1590 (10)
C(201)	−1606 (13)	862 (6)	−3861 (11)
C(202)	−936 (13)	486 (6)	−4110 (11)
C(203)	−1715 (13)	100 (6)	−4349 (11)
C(204)	−3164 (13)	89 (6)	−4340 (11)
C(205)	−3835 (13)	464 (6)	−4092 (11)
C(206)	−3056 (13)	851 (6)	−3852 (11)
C(211)	−1812 (18)	1810 (5)	−3970 (9)
C(212)	−2600 (18)	1777 (5)	−4800 (9)
C(213)	−3482 (18)	2126 (5)	−5418 (9)
C(214)	−3576 (18)	2508 (5)	−4667 (9)
C(215)	−2787 (18)	2541 (5)	−3837 (9)
C(216)	−1905 (18)	2192 (5)	−3489 (9)
C(221)	902 (17)	1368 (6)	−4058 (8)
C(222)	2235 (17)	1435 (6)	−3543 (8)
C(223)	3449 (17)	1428 (6)	−3903 (8)
C(224)	3330 (17)	1355 (6)	−4778 (8)
C(225)	1997 (17)	1289 (6)	−5293 (8)
C(226)	783 (17)	1296 (6)	−4933 (8)

The two shorter Os—Os bonds (mean 2·768 Å) are in close agreement with the 2·764 (1) Å for the non-hydride bridged bond in $H_2Os_3(CO)_9S$ (Johnson, Lewis, Pippard, Raithby, Sheldrick & Rouse, 1978). These distances, in both compounds, are significantly shorter than the average Os—Os distance of 2·877 (1) Å in $Os_3(CO)_{12}$ (Churchill & DeBoer, 1977), which illustrates the bond-shortening influence of the μ_3-S ligand. The long Os—Os length is similar to the mean of 2·915 Å in the dihydride (Johnson, Lewis, Pippard, Raithby, Sheldrick & Rouse, 1978) for the H-bridged bonds. The carbonyl arrangement (Fig. 1) also suggests that the hydride lies in the hole in the ligand polyhedron close to this long Os—Os bond. The *cis* Os—Os—C bond angles involving Os(1), Os(2), C(12), C(13), C(21) and C(23) average 107·5°, the remainder 96·0°. A similar widening of metal—metal—carbonyl

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33912 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Hydrogen-atom coordinates* ($\times 10^4$)

	x	*y*	*z*
H(102)	1610 (16)	2039 (6)	−1895 (8)
H(103)	2728 (16)	2650 (6)	−962 (8)
H(104)	2090 (16)	2768 (6)	453 (8)
H(105)	334 (16)	2275 (6)	935 (8)
H(106)	−784 (16)	1664 (6)	1 (8)
H(112)	244 (17)	1015 (5)	76 (10)
H(113)	1410 (17)	327 (5)	715 (10)
H(114)	2368 (17)	−216 (5)	−193 (10)
H(115)	2159 (17)	−72 (5)	−1741 (10)
H(116)	992 (17)	616 (5)	−2380 (10)
H(122)	−2106 (16)	565 (4)	−1513 (10)
H(123)	−4647 (16)	482 (4)	−1447 (10)
H(124)	−6166 (16)	1149 (4)	−1472 (10)
H(125)	−5142 (16)	1898 (4)	−1564 (10)
H(126)	−2601 (16)	1981 (4)	−1630 (10)
H(202)	187 (13)	495 (6)	−4117 (11)
H(203)	−1196 (13)	−191 (6)	−4542 (11)
H(204)	−3768 (13)	−211 (6)	−4526 (11)
H(205)	−4957 (13)	456 (6)	−4084 (11)
H(206)	−3575 (13)	1141 (6)	−3659 (11)
H(212)	−2528 (18)	1480 (5)	−5172 (9)
H(213)	−4093 (18)	2100 (5)	−5790 (9)
H(214)	−4259 (18)	2778 (5)	−4937 (9)
H(215)	−2859 (18)	2837 (5)	−3465 (9)
H(216)	−1295 (18)	2218 (5)	−2846 (9)
H(222)	2327 (17)	1491 (6)	−2866 (8)
H(223)	4481 (17)	1479 (6)	−3504 (8)
H(224)	4270 (17)	1350 (6)	−5056 (8)
H(225)	1905 (17)	1233 (6)	−5970 (8)
H(226)	−249 (17)	1244 (6)	−5332 (8)

angles has been observed in $[H_6Re_4(CO)_{12}]^{2-}$ (Ciani, Sironi & Albano, 1977) where all the Re–Re bonds are considered to be μ_2-H bridged.

The S atom lies 1·75 Å below the Os_3 triangle, on the opposite side of the metal plane to the proposed hydride position. Os(3)–S(1) is slightly shorter than the other two Os–S bonds which reflects the difference in coordination geometry and electronic environment of Os(3) and the other two Os atoms. The anionic charge seems to be delocalized over the HOs_3S unit.

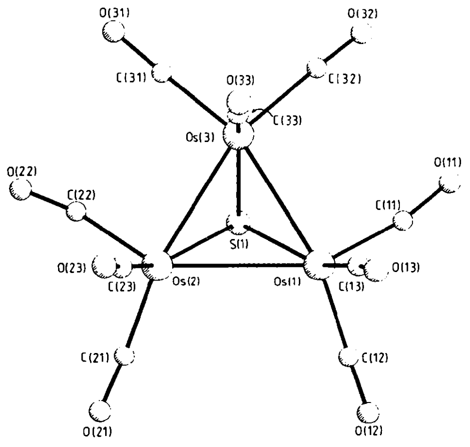

Fig. 1. The $[HOs_3(CO)_9S]^-$ anion.

Table 3. *Selected bond lengths* (Å) *and angles* (°)

Anion			
Os(1)–Os(2)	2·899 (2)	Os(1)–C(11)	1·786 (30)
Os(1)–Os(3)	2·766 (2)	Os(1)–C(12)	1·841 (31)
Os(2)–Os(3)	2·769 (3)	Os(1)–C(13)	1·908 (32)
Os(1)–S(1)	2·396 (7)	Os(2)–C(21)	1·865 (39)
Os(2)–S(1)	2·401 (6)	Os(2)–C(22)	1·881 (29)
Os(3)–S(1)	2·369 (7)	Os(2)–C(23)	1·867 (27)
C(11)–O(11)	1·237 (37)	Os(3)–C(31)	1·863 (24)
C(12)–O(12)	1·208 (38)	Os(3)–C(32)	1·926 (30)
C(13)–O(13)	1·126 (41)	Os(3)–C(33)	1·867 (28)
C(21)–O(21)	1·147 (50)	C(31)–O(31)	1·180 (32)
C(22)–O(22)	1·145 (39)	C(32)–O(32)	1·142 (36)
C(23)–O(23)	1·177 (35)	C(33)–O(33)	1·132 (34)
Cation			
N(1)–P(1)	1·580 (20)	N(1)–P(2)	1·557 (17)
P(1)–C(101)	1·796 (16)	P(2)–C(201)	1·798 (17)
P(1)–C(111)	1·789 (16)	P(2)–C(211)	1·801 (16)
P(1)–C(121)	1·789 (17)	P(2)–C(221)	1·809 (18)
Anion			
Os(2)–Os(1)–Os(3)	58·5 (1)	Os(2)–Os(1)–C(11)	150·1 (10)
Os(1)–Os(2)–Os(3)	58·4 (1)	Os(3)–Os(1)–C(11)	94·1 (10)
Os(1)–Os(3)–Os(2)	63·2 (1)	Os(2)–Os(1)–C(12)	107·7 (10)
Os(2)–Os(1)–S(1)	52·9 (1)	Os(3)–Os(1)–C(12)	161·8 (10)
Os(3)–Os(1)–S(1)	54·1 (2)	Os(2)–Os(1)–C(13)	106·9 (8)
Os(1)–Os(2)–S(1)	52·7 (2)	Os(3)–Os(1)–C(13)	97·7 (8)
Os(3)–Os(2)–S(1)	54·0 (2)	Os(1)–Os(2)–C(21)	109·6 (11)
Os(1)–Os(3)–S(1)	55·0 (2)	Os(3)–Os(2)–C(21)	160·2 (10)
Os(2)–Os(3)–S(1)	55·0 (2)	Os(1)–Os(2)–C(22)	143·0 (8)
S(1)–Os(1)–C(11)	102·7 (11)	Os(3)–Os(2)–C(22)	89·0 (9)
S(1)–Os(1)–C(12)	108·7 (11)	Os(1)–Os(2)–C(23)	105·6 (8)
S(1)–Os(1)–C(13)	150·2 (8)	Os(3)–Os(2)–C(23)	97·4 (8)
S(1)–Os(2)–C(21)	106·4 (10)	Os(1)–Os(3)–C(31)	156·9 (8)
S(1)–Os(2)–C(22)	95·1 (8)	Os(2)–Os(3)–C(31)	98·2 (8)
S(1)–Os(2)–C(23)	149·5 (8)	Os(1)–Os(3)–C(32)	99·2 (9)
S(1)–Os(3)–C(31)	103·8 (8)	Os(2)–Os(3)–C(32)	159·3 (8)
S(1)–Os(3)–C(32)	106·6 (9)	Os(1)–Os(3)–C(33)	96·4 (8)
S(1)–Os(3)–C(33)	145·1 (9)	Os(2)–Os(3)–C(33)	96·2 (8)
Os(1)–S(1)–Os(2)	74·4 (2)	C(11)–Os(1)–C(12)	96·0 (14)
Os(1)–S(1)–Os(3)	71·0 (2)	C(11)–Os(1)–C(13)	87·0 (13)
Os(2)–S(1)–Os(3)	71·0 (2)	C(12)–Os(1)–C(13)	98·0 (13)
Os(1)–C(11)–O(11)	167·4 (28)	C(21)–Os(2)–C(22)	95·9 (14)
Os(1)–C(12)–O(12)	174·8 (26)	C(21)–Os(2)–C(23)	101·2 (13)
Os(1)–C(13)–O(13)	173·3 (24)	C(22)–Os(2)–C(23)	94·8 (12)
Os(2)–C(21)–O(21)	169·8 (31)	C(31)–Os(3)–C(32)	95·7 (12)
Os(2)–C(22)–O(22)	168·0 (25)	C(31)–Os(3)–C(33)	99·2 (11)
Os(2)–C(23)–O(23)	173·6 (22)	C(32)–Os(3)–C(33)	96·6 (12)
Os(3)–C(31)–O(31)	174·6 (22)	Os(3)–C(33)–O(33)	178·2 (27)
Os(3)–C(32)–O(32)	173·1 (27)		
Cation			
P(1)–N(1)–P(2)	144·7 (13)		
N(1)–P(1)–C(101)	107·6 (9)	N(1)–P(2)–C(201)	115·7 (9)
N(1)–P(1)–C(111)	109·0 (9)	N(1)–P(2)–C(211)	111·8 (9)
C(101)–P(1)–C(111)	107·6 (7)	C(201)–P(2)–C(211)	105·3 (7)
N(1)–P(1)–C(121)	117·0 (9)	N(1)–P(2)–C(221)	106·1 (9)
C(101)–P(1)–C(121)	108·5 (8)	C(201)–P(2)–C(221)	108·0 (8)
C(111)–P(1)–C(121)	106·7 (8)	C(211)–P(2)–C(221)	109·7 (8)
P(1)–C(101)–C(102)	119·7 (5)	P(2)–C(201)–C(202)	120·9 (5)
P(1)–C(101)–C(106)	120·1 (5)	P(2)–C(201)–C(206)	119·1 (5)
P(1)–C(111)–C(112)	119·4 (6)	P(2)–C(211)–C(212)	119·1 (5)
P(1)–C(111)–C(116)	120·5 (6)	P(2)–C(211)–C(216)	120·9 (5)
P(1)–C(121)–C(122)	120·8 (5)	P(2)–C(221)–C(222)	118·0 (4)
P(1)–C(121)–C(126)	119·2 (5)	P(2)–C(221)–C(226)	122·0 (4)

The carbonyl groups are all approximately linear with an average Os–C–O angle of 172·5°. The mean Os–C and C–O distances of 1·87 and 1·17 Å, respectively, are similar to the carbonyl dimensions in $[HOs_3(CO)_{10}O_2COs_6(CO)_{17}]^-$ (Guy & Sheldrick, 1978*a*).

The cation dimensions are similar to those in $[(Ph_3P)_2N][HOs_5(CO)_{15}]$ (Guy & Sheldrick, 1978*b*), with staggered PPh_3 groups.

We thank the Science Research Council for financial support. The calculations were performed on the Cambridge University IBM 370/165 computer with programs written by Dr G. M. Sheldrick; the figure was drawn with *PLUTO* written by Dr W. D. S. Motherwell.

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Acta Cryst. (1978). B**34**, 3770–3772

Tetrakis(acetylacetonato)thorium(IV) Hemibenzene

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(*Received* 18 *August* 1978; *accepted* 25 *September* 1978)

Abstract. $Th(C_5H_7O_2)_4.\frac{1}{2}C_6H_6$, triclinic, $P\bar{1}$, a = 11·591 (4), b = 14·153 (2), c = 9·229 (3) Å, α = 94·20 (2), β = 106·50 (2), γ = 113·51 (2)°, FW = 667·6, $Z = 2$, D_x = 1·670 Mg m^{-3}, R = 0·037, 4181 observed reflections. The inner coordination polyhedron around the Th atom is a square antiprism (D_{4d}-$\bar{8}2m$). Of the four acetylacetonate ligands, two have a fold angle of about 16° over the line joining the two O atoms within the ligand, while the corresponding angle for the remaining two ligands is close to 3°.

Introduction. During investigations of the coordination of Th in chelate complexes with β-diketones, the title compound has been synthesized and its structure investigated. A preliminary structure analysis, with the results of IR measurements, has been published (Allard, 1976). This is the only reported organic solvate of a tetravalent metal acetylacetonate. It is of interest to find out if the benzene molecules influence the coordination around Th.

The complex, $Th(AA)_4$, was precipitated by mixing aqueous solutions of $Th(NO_3)_4$, ammonia and acetylacetone. The precipitate was recrystallized from benzene at room temperature.

The diffraction data were collected on a Syntex $P2_1$ computer-controlled four-circle diffractometer. The parameters of the cell were determined by a least-squares procedure from the coordinates of 15 reflections. The intensities of 4501 independent reflections were measured up to 2θ = 50° with graphite-monochromatized Mo $K\alpha$ radiation (λ = 0·71069 Å). A standard reflection, measured after every 25 reflections, gave no indication of crystal decay. The intensities were corrected for Lorentz and polarization effects but not for absorption. 4181 reflections had intensities $>3\sigma$. The Th atom was located from a Patterson synthesis and the remaining non-hydrogen atoms by a subsequent Fourier synthesis. The structure was refined by a block-diagonal least-squares method (Lindgren, 1977) with anisotropic thermal parameters and an overall scale factor. The final R was 0·037. Scattering factors were those of Doyle & Turner (1968) for C and O, and for Th those of Cromer & Waber (1965). Correction for the real part of the anomalous dispersion was made (*International Tables for X-ray Crystallography*, 1962). Atomic positions are given in Table 1.* The structure factors were weighted according to $w = (a + F_o + cF^2_o)^{-1}$ (Cruickshank, 1965) with a = 80·0 and c = 0·003.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publications No. SUP 33922 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.