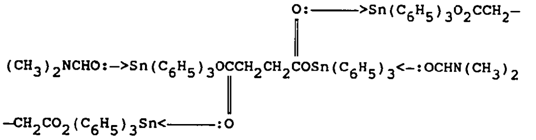
for example, in the quinolyloxyacetate hydrate [Sn←O = 2.338 (7), 2.391 (6) Å (Kumar Das, Chen, Ng & Mak, 1987)] and trichloroacetate.methanol [Sn←O = 2.400 (3) Å (Alcock & Roe, 1989)] complexes. The present tetranuclear complex (I) formally consists of two coordinated DMF and two independent bis(triphenyltin) succinate molecules, *a* and *b*.



$$\begin{array}{l} \qquad\qquad\qquad\qquad\qquad\qquad O{:}\longrightarrow Sn(C_6H_5)_3O_2CCH_2- \\ \qquad\qquad\qquad\qquad\qquad\qquad \| \\ (CH_3)_2NCHO{:}\rightarrow Sn(C_6H_5)_3OCCH_2CH_2COSn(C_6H_5)_3\leftarrow{:}OCHN(CH_3)_2 \\ \qquad\qquad\qquad\qquad\quad \| \\ -CH_2CO_2(C_6H_5)_3Sn\longleftarrow{:}O \end{array}$$

(I)

In the complex, the Sn1*a*←O_{DMF} bond distance is only slightly shorter than the Sn←O carboxylate bridge and compares well with the 2.402 (5) Å length found in the DMF adduct of the *N*-triphenylstannyl derivative of saccharin (Ng, Kuthubutheen, Zainudin, Chen, Kumar Das, Schulze, Molloy, Yip & Mak, 1991). The intermolecular bridging distance of 2.444 (2) Å is longer than that found in a monocarboxylate such as triphenyltin acetate [Sn←O = 2.349 (3) Å (Molloy, Purcell, Quill & Nowell, 1984)], but is similar to those found in the bis(trimethyltin) derivatives of acetylenedicarboxylic [Sn←O = 2.401 (4), 2.408 (4) Å], terephthalic [Sn←O = 2.506 (3) Å] (Glowacki, Huber & Preut, 1988) and malonic [Sn←O = 2.44 (2), 2.46 (2) Å (Schubert, 1978)] acids.

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Bis(tetrabutylammonium) Nonadecaoxohexamolybdenum(VI): a Second Polymorph

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Abstract. $[C_{16}H_{36}N]_2[Mo_6O_{19}]$, M_r = 1364.6, monoclinic, *C*2/*c*, *a* = 16.280 (3), *b* = 17.204 (3), *c* = 17.738 (4) Å, β = 101.42 (2)°, *V* = 4869.3 (17) Å^3, *Z* = 4, D_x = 1.861 g cm^{-3}, λ(Mo *K*α) = 0.71073 Å, μ = 15.66 cm^{-1}, *F*(000) = 2728, *T* = 298 K, *R* = 0.0633 for 2857 observed reflections and 269 least-squares parameters. The six Mo atoms form the standard distorted octahedral cage with O atoms bridging the Mo atoms. There is a crystallographic twofold axis in the anionic cluster ($Mo_6O_{19}^{2-}$) through the central O atom and containing Mo(2) and Mo(4).

Experimental. Orange–red crystals were grown from CH_3CN/Et_2O (0.26 × 0.32 × 0.32 mm). Data were collected on a Siemens *P*4 diffractometer with graphite monochromator, using ω scans. Lattice parameters were determined from least-squares fit of 25 reflections (20 ≤ 2θ ≤ 25°), minimized on *F*. Semi-empirical absorption correction was applied to the data set [*XEMP* (Sheldrick, 1990), T_{max}/T_{min} = 0.178/0.092, μ = 15.66 cm^{-1}]. $2\theta_{max}$ = 50° (*h* = ±20, *k* = +21, *l* = +22). Three standard reflections ($\bar{9}13$, $\bar{1}90$ and 0,0,10) for 4609 reflections collected, showed

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2% decay of 4301 unique reflections ($R_{\text{int}} = 8.18\%$), 2857 were observed with $F_o > 4\sigma(F_o)$ (1752 unobserved reflections). Direct methods (*SOLV*; Sheldrick, 1990) were used for structure solution. Least-squares refinement on 269 parameters, with all non-H atoms anisotropic and all H atoms included as idealized isotropic contributions (C—H = 0.960 Å, $U = 1.2U$ for attached C), converged at $R_F = 6.33\%$, $wR_F = 7.02\%$ [$w^{-1} = \sigma(F_o) + gF_o^2$, $g = 0.0008$], $S = 1.60$; $(\Delta/\sigma)_{\text{max}} = 0.001$; $(\Delta\rho)_{\text{max}} = 1.41$, $(\Delta\rho)_{\text{min}} = -0.93$ e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). *SHELXTL-Plus* (Sheldrick, 1990) computer programs were used in the structure determination.

Atomic coordinates and equivalent isotropic displacement coefficients are given in Table 1. Bond lengths and angles are given in Table 2.* Fig. 1 shows the structure of the $Mo_6O_{19}^{2-}$ anion and Fig. 2 the unit-cell packing.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* (Å$^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Mo(1)	5619 (1)	2898 (1)	6431 (1)	56 (1)
Mo(2)	5000	1545 (1)	7500	51 (1)
Mo(3)	6311 (1)	2892 (1)	8295 (1)	57 (1)
Mo(4)	5000	4244 (1)	7500	65 (1)
O(1)	5000	2895 (5)	7500	43 (3)
O(2)	6083 (5)	2919 (4)	5663 (4)	72 (3)
O(3)	5509 (4)	1797 (4)	6655 (4)	55 (2)
O(4)	6563 (3)	2896 (4)	7298 (4)	55 (2)
O(5)	5503 (4)	3995 (4)	6638 (4)	64 (3)
O(6)	5000	571 (6)	7500	79 (4)
O(7)	6048 (4)	1806 (4)	8148 (4)	57 (2)
O(8)	7250 (4)	2872 (5)	8882 (4)	80 (3)
O(9)	6059 (4)	3979 (4)	8150 (4)	66 (3)
O(10)	5000	5210 (6)	7500	93 (5)
O(11)	5560 (4)	2897 (4)	9014 (4)	61 (2)
N(1)	2474 (4)	−135 (4)	509 (4)	52 (3)
C(1)	2541 (6)	624 (6)	99 (6)	63 (4)
C(2)	2214 (7)	628 (7)	−763 (6)	75 (5)
C(3)	2371 (8)	1400 (8)	−1090 (8)	93 (6)
C(4)	2106 (9)	1436 (9)	−1935 (8)	116 (7)
C(5)	2934 (6)	−59 (6)	1356 (5)	61 (4)
C(6)	2540 (6)	488 (7)	1846 (6)	70 (4)
C(7)	3173 (7)	688 (8)	2570 (6)	77 (5)
C(8)	3796 (9)	1260 (9)	2447 (8)	119 (7)
C(9)	1546 (6)	−316 (6)	468 (6)	65 (4)
C(10)	1360 (7)	−1026 (7)	887 (7)	74 (5)
C(11)	421 (7)	−1112 (8)	829 (7)	84 (5)
C(12)	176 (8)	−1789 (8)	1238 (8)	102 (6)
C(13)	2873 (5)	−804 (5)	145 (6)	52 (3)
C(14)	3777 (6)	−672 (6)	80 (6)	60 (4)
C(15)	4137 (5)	−1357 (6)	−267 (6)	57 (4)
C(16)	5042 (6)	−1220 (7)	−333 (7)	77 (5)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Mo(1)—O(1)	2.317 (1)	Mo(1)—O(2)	1.683 (8)
Mo(1)—O(3)	1.951 (6)	Mo(1)—O(4)	1.947 (6)
Mo(1)—O(5)	1.939 (6)	Mo(1)—O(11*a*)	1.926 (6)
Mo(2)—O(1)	2.323 (8)	Mo(2)—O(3)	1.902 (7)
Mo(2)—O(6)	1.676 (10)	Mo(2)—O(7)	1.913 (6)
Mo(3)—O(1)	2.313 (1)	Mo(3)—O(4)	1.893 (7)
Mo(3)—O(7)	1.924 (6)	Mo(3)—O(8)	1.671 (6)
Mo(3)—O(9)	1.920 (6)	Mo(3)—O(11)	1.932 (7)
Mo(4)—O(1)	2.321 (8)	Mo(4)—O(5)	1.921 (8)
Mo(4)—O(9)	1.929 (6)	Mo(4)—O(10)	1.661 (10)

O(1)—Mo(1)—O(2)	178.6 (3)	O(1)—Mo(1)—O(3)	76.0 (3)
O(2)—Mo(1)—O(3)	105.0 (3)	O(1)—Mo(1)—O(4)	75.9 (2)
O(2)—Mo(1)—O(4)	103.2 (3)	O(3)—Mo(1)—O(4)	85.8 (3)
O(1)—Mo(1)—O(5)	76.9 (3)	O(2)—Mo(1)—O(5)	102.0 (3)
O(3)—Mo(1)—O(5)	152.9 (3)	O(4)—Mo(1)—O(5)	86.8 (3)
O(1)—Mo(1)—O(11*a*)	77.0 (2)	O(2)—Mo(1)—O(11*a*)	103.9 (3)
O(3)—Mo(1)—O(11*a*)	87.6 (3)	O(4)—Mo(1)—O(11*a*)	152.9 (3)
O(5)—Mo(1)—O(11*a*)	87.2 (3)	O(1)—Mo(2)—O(3)	76.8 (2)
O(1)—Mo(2)—O(6)	180.0	O(3)—Mo(2)—O(6)	103.2 (2)
O(1)—Mo(2)—O(7)	76.4 (2)	O(3)—Mo(2)—O(7)	86.8 (3)
O(6)—Mo(2)—O(7)	103.6 (2)	O(1)—Mo(2)—O(3*a*)	76.8 (2)
O(3)—Mo(2)—O(3*a*)	153.6 (4)	O(7)—Mo(2)—O(3*a*)	87.0 (3)
O(1)—Mo(2)—O(7*a*)	76.4 (2)	O(3)—Mo(2)—O(7*a*)	87.1 (3)
O(7)—Mo(2)—O(7*a*)	152.9 (4)	O(1)—Mo(3)—O(4)	77.0 (2)
O(1)—Mo(3)—O(7)	76.5 (3)	O(4)—Mo(3)—O(7)	87.7 (3)
O(1)—Mo(3)—O(8)	178.6 (3)	O(4)—Mo(3)—O(8)	103.9 (3)
O(7)—Mo(3)—O(8)	102.5 (3)	O(1)—Mo(3)—O(9)	76.7 (3)
O(4)—Mo(3)—O(9)	87.2 (3)	O(7)—Mo(3)—O(9)	153.1 (3)
O(8)—Mo(3)—O(9)	104.4 (3)	O(1)—Mo(3)—O(11)	77.0 (2)
O(4)—Mo(3)—O(11)	154.0 (2)	O(7)—Mo(3)—O(11)	86.6 (3)
O(8)—Mo(3)—O(11)	102.1 (3)	O(9)—Mo(3)—O(11)	86.4 (3)
O(1)—Mo(4)—O(5)	77.1 (2)	O(1)—Mo(4)—O(9)	76.3 (2)
O(5)—Mo(4)—O(9)	87.2 (3)	O(1)—Mo(4)—O(10)	180.0
O(5)—Mo(4)—O(10)	102.9 (2)	O(9)—Mo(4)—O(10)	103.7 (2)
O(5)—Mo(4)—O(5*a*)	154.2 (4)	O(9)—Mo(4)—O(5*a*)	86.7 (3)
O(5)—Mo(4)—O(9*a*)	86.7 (3)	O(9)—Mo(4)—O(9*a*)	152.6 (4)
O(5*a*)—Mo(4)—O(9*a*)	87.2 (3)	Mo(1)—O(1)—Mo(2)	90.1 (2)
Mo(1)—O(1)—Mo(3)	90.0 (1)	Mo(2)—O(1)—Mo(3)	89.9 (2)
Mo(1)—O(1)—Mo(4)	89.9 (2)	Mo(2)—O(1)—Mo(4)	180.0
Mo(3)—O(1)—Mo(4)	90.1 (2)	Mo(1)—O(1)—Mo(1*a*)	179.7 (4)
Mo(1)—O(1)—Mo(3*a*)	90.0 (1)	Mo(2)—O(1)—Mo(3*a*)	89.9 (2)
Mo(3)—O(1)—Mo(3*a*)	179.8 (2)	Mo(1)—O(3)—Mo(2)	117.0 (3)
Mo(1)—O(4)—Mo(3)	117.1 (3)	Mo(1)—O(5)—Mo(4)	116.1 (4)
Mo(2)—O(7)—Mo(3)	117.2 (3)	Mo(3)—O(9)—Mo(4)	116.9 (3)
Mo(3)—O(11)—Mo(1*a*)	116.1 (3)		

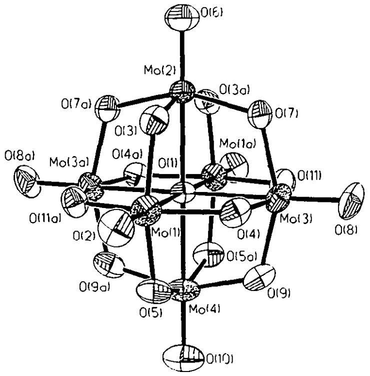

Fig. 1. The structure of the $Mo_6O_{19}^{2-}$ anion.

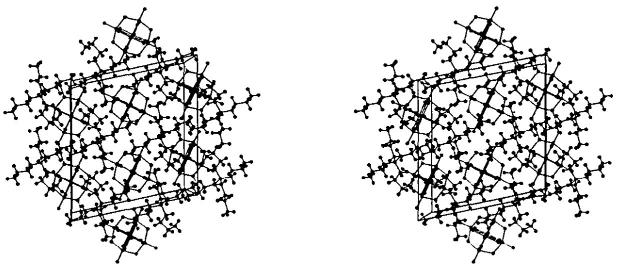

Fig. 2. Unit-cell packing diagram for the title compound as viewed down the *a* axis.

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* Lists of structure factors, bond distances and angles, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55589 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1016]

Related literature. The title compound was obtained as unreacted starting material in an attempt to form $[Mo_5O_{18}(Mo{\equiv}N{-}SiMe_3)]^{2-}$ by reaction with $Me_3SiN{=}PPh_3$. Dahlstrom, Zubieta, Neaves & Dilworth (1982) have reported a different monoclinic polymorph ($P2_1/n$) of the title compound. Several other salts have the same anion: Ph_4As^+ (Clegg, Sheldrick, Garner & Walton, 1982); $C_{22}H_{22}O_2P^+$ and $C_{22}H_{22}P^+$ (Arzoumanian, Baldy, Lai, Odreman, Metzger & Pierrot, 1985); $C_{12}H_{24}O_6.H_3O^+$ (Shoemaker, McAfee, Shoemaker & DeKock, 1986); $C_{20}H_{40}MoN_4S_8{}^+$ (Garner, Howlader, Mabbs, McPhail, Miller & Onan, 1978); $C_{39}H_{33}Mo_2O_4P_2^+$ (Riera, Ruiz, Villafane, Jeannin & Bois, 1988); $C_{12}H_{24}O_6.K^+.H_2O$ (Nagano & Sasaki, 1979); $C_{12}H_{37}N_9P_3^+$ (Allcock, Bissell & Shawl, 1973); and $C_{14}H_{21}ClN_2Rh^+$ (Zhang, Ozawa, Hayashi & Isobe, 1989).

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Structure of Tribenzyl(thiocyanato-*N*)(triphenylarsine oxide-*O*)tin

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Abstract. $[Sn(NCS)(C_{18}H_{15}AsO)(C_7H_7)_3]$, M_r = 772.42, triclinic, $P\bar{1}$, a = 10.099 (2), b = 11.4014 (8), c = 15.682 (3) Å, α = 95.145 (9), β = 92.72 (2), γ = 98.29 (1)°, V = 1776.3 (5) Å^3, Z = 2, D_x = 1.444 g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 17.30 cm^{-1}, $F(000)$ = 780, T = 300 K, R = 0.030 for 4221 reflections. The Sn atom is five-coordinate in a *trans*-C_3SnNO trigonal bipyramidal environment, being displaced out the equatorial plane by 0.030 (1) Å in the direction of the N occupant.

Experimental. An equimolar amount of triphenylarsine oxide was added to an ethanol solution containing tribenzyltin isothiocyanate, which had been prepared *in situ* from the metathetical reaction between tribenzyltin chloride and potassium thiocyanate. Slow evaporation of the filtered solution furnished crystals of the molecular complex, m.p. 419–420 K. ^{119}Sn NMR in $CDCl_3$ (saturated solution): $\delta = -187.5$ p.p.m. relative to $(CH_4)_4Sn$. ^{13}C NMR in $CDCl_3$: $^1J(^{119}Sn-^{13}C)$ = 476.1 Hz. ^{1}H NMR in $CDCl_3$: $^2J(^{119}Sn-^1H)$ = 79.9 Hz.

A 0.22 × 0.22 × 0.29 mm crystal was used for the study on an Enraf–Nonius CAD-4 diffractometer. Unit-cell constants were calculated from the 25 strongest reflections in the θ = 13–15° range. Intensity measurements were obtained by using the ω–2θ scan method to $2\theta_{max}$ = 50° (collection range: h = 0 to 12, $k = -13$ to 13, $l = -18$ to 18). 4221 of the 5836 unique reflections (R_{int} = 0.012) obeyed the $I \geq 3\sigma(I)$ criterion. The data were corrected for decay (minimum/maximum correction = 1.0000/1.0281, average correction = 1.0127), which was monitored by three reflections hourly, and for absorption effects by using the ψ-scan data (minimum/maximum correction = 0.9495/0.9996, average correction = 0.9755). The solution by the heavy-atom method presented some difficulties as the Sn–Sn vector in the Patterson map was found to be the third largest, after the Sn–As vectors. Non-H atoms were refined

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