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*Acta Cryst.* (1994). C**50**, 1052–1054

# Octadecacarbonylhexaosmium Chloroform Solvate

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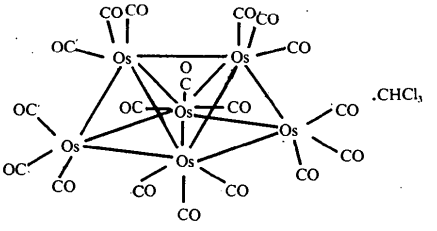
(*Received* 2 *December* 1993; *accepted* 10 *January* 1994)

## Abstract

The hexaosmium clusters in octadecacarbonyl-1$\kappa^3C$,-2$\kappa^3C$,3$\kappa^3C$,4$\kappa^3C$,5$\kappa^3C$,6$\kappa^3C$-hexaosmium-chloroform (1/1), $[Os_6(CO)_{18}].CHCl_3$, have essentially the same internal geometry as in unsolvated $Os_6(CO)_{18}$ but participate in O···H and O···Cl contacts with the chloroform solvate molecules.

## Comment

The structure of the title compound (I) consists of bicapped tetrahedral $Os_4$ clusters within molecules of $Os_6(CO)_{18}$, each of which interacts with molecules of the chloroform solvate through O···H contacts [O(9)···H(1S$^{ii}$) = 2.65 (3), O(16)···H(1S$^{iii}$) = 2.55 (3) Å] and O···Cl contacts [O(9)···Cl(3$^{i}$) = 3.010 (19) Å; symmetry codes: (i) $-1+x, y, z$; (ii) $1-x, -y, -z$; (iii) $1-x, -y, 1-z$]. The Os···Os distances within the $Os_4$ tetrahedron range from 2.7395 (11) to 2.8159 (12) Å, while those involving the $\mu_3$-capping Os atoms average 2.808 (18) Å; these values are similar to those of the unsolvated molecule (Mason, Thomas & Mingos, 1973). The Os—C—O angles range from 169.6 (16) to 179.6 (17)°.




(I)

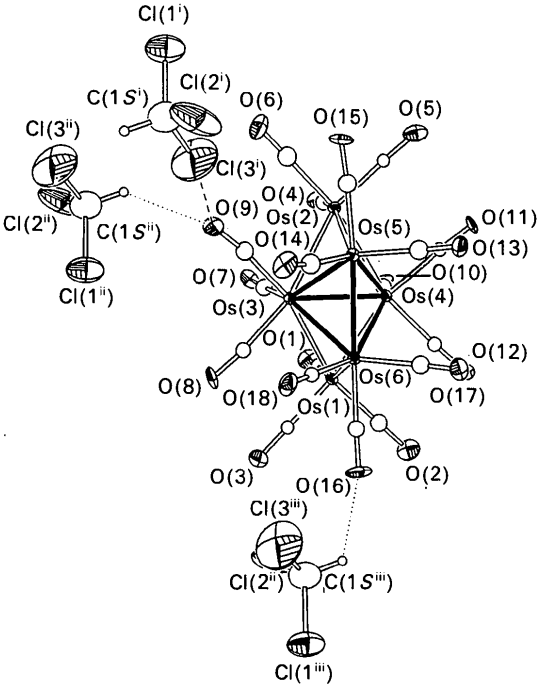



Fig. 1. One $Os_6(CO)_{18}$ molecule and the $CHCl_3$ solvate molecules with which it interacts are shown. The C atoms of the carbonyl groups bear the same numbers as the corresponding O atoms. The central $Os_4$ tetrahedron is indicated by solid bonds, O···H interactions by dotted lines and the Cl···O interaction by a dashed line. Displacement ellipsoids are shown at the 50% level for Os, Cl and O atoms. [Symmetry codes: (i) $-1+x, y, z$; (ii) $1-x, -y, -z$; (iii) $1-x, -y, 1-z$.]

## Experimental

The title compound was prepared by the crystallization of $Os_6(CO)_{18}$ from $CHCl_3$.

*Crystal data*

$[Os_6(CO)_{18}].CHCl_3$
$M_r$ = 1764.75
Monoclinic
$P2_1/n$
$a$ = 10.074 (3) Å
$b$ = 27.293 (9) Å
$c$ = 11.286 (3) Å
$\beta$ = 91.47 (3)°
$V$ = 3102.1 (16) Å$^3$
$Z$ = 4
$D_x$ = 3.779 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\lambda$ = 0.71073 Å
Cell parameters from 32 reflections
$\theta$ = 15–16°
$\mu$ = 24.8 mm$^{-1}$
$T$ = 150.0 (2) K
Block
0.35 × 0.31 × 0.23 mm
Dark red

*Data collection*

Stoe Stadi-4 four-circle diffractometer
$\omega$ scans with on-line profile fitting
Absorption correction: Semi-empirical [*SHELXTL/PC* (Sheldrick, 1990) and *DIFABS* (Walker & Stuart, 1983)]
$T_{min}$ = 0.048, $T_{max}$ = 0.117
4274 measured reflections
3855 independent reflections
3335 observed reflections [$I > 2\sigma(I)$]
$R_{int}$ = 0.040
$\theta_{max}$ = 22.55°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 29$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity variation: <2%

*Refinement*

Refinement on $F^2$
$R(F)$ = 0.036
$wR(F^2)$ = 0.087
$S$ = 1.038
3851 reflections
326 parameters
H-atom position calculated and fixed
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0439P)^2 + 157.2638P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = −0.171
$\Delta\rho_{max}$ = 1.72 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.41 e Å$^{-3}$
Extinction correction: *SHELXL*93 (Sheldrick, 1994)
Extinction coefficient: 0.00010 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å$^2$)

$U_{iso}$ for C atoms; $U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for Os, Cl and O.

| | $x$ | $y$ | $z$ | $U_{iso}/U_{eq}$ |
|---|---|---|---|---|
| Os1 | 0.44406 (6) | 0.10779 (2) | 0.66126 (6) | 0.0124 (2) |
| Os2 | 0.29571 (6) | 0.18799 (2) | 0.30706 (6) | 0.0128 (2) |
| Os3 | 0.31600 (6) | 0.10048 (2) | 0.43993 (6) | 0.0106 (2) |
| Os4 | 0.31369 (6) | 0.18944 (2) | 0.55623 (6) | 0.0102 (2) |
| Os5 | 0.08291 (6) | 0.15554 (2) | 0.43850 (6) | 0.0116 (2) |
| Os6 | 0.16597 (6) | 0.11138 (2) | 0.64539 (6) | 0.0117 (2) |
| O1 | 0.7282 (13) | 0.1234 (5) | 0.5864 (12) | 0.032 (3) |
| O2 | 0.5033 (13) | 0.1452 (5) | 0.9123 (12) | 0.030 (3) |
| O3 | 0.5157 (14) | 0.0042 (5) | 0.7349 (12) | 0.030 (3) |
| O4 | 0.5818 (12) | 0.2099 (5) | 0.2496 (12) | 0.032 (3) |
| O5 | 0.2013 (12) | 0.2911 (5) | 0.2567 (11) | 0.028 (3) |
| O6 | 0.2427 (14) | 0.1570 (6) | 0.0513 (12) | 0.037 (4) |
| O7 | 0.5957 (11) | 0.1010 (4) | 0.3353 (11) | 0.022 (3) |
| O8 | 0.3255 (12) | −0.0082 (4) | 0.5092 (12) | 0.023 (3) |
| O9 | 0.1871 (14) | 0.0596 (5) | 0.2101 (12) | 0.034 (4) |
| O10 | 0.5966 (12) | 0.2269 (5) | 0.5141 (11) | 0.025 (3) |
| O11 | 0.2042 (14) | 0.2925 (5) | 0.5173 (12) | 0.030 (3) |
| O12 | 0.3111 (14) | 0.2203 (5) | 0.8170 (11) | 0.027 (3) |
| O13 | −0.0962 (13) | 0.2300 (5) | 0.5530 (12) | 0.030 (3) |
| O14 | −0.1162 (13) | 0.0720 (5) | 0.3897 (12) | 0.033 (3) |
| O15 | −0.0278 (13) | 0.1919 (5) | 0.2003 (11) | 0.028 (3) |
| O16 | 0.2405 (14) | 0.0637 (5) | 0.8820 (11) | 0.033 (3) |
| O17 | −0.0100 (15) | 0.1768 (5) | 0.7912 (13) | 0.036 (4) |
| O18 | −0.0209 (15) | 0.0248 (5) | 0.6189 (12) | 0.034 (3) |
| C1 | 0.6196 (17) | 0.1189 (6) | 0.6141 (15) | 0.014 (4) |
| C2 | 0.4822 (19) | 0.1310 (7) | 0.8168 (18) | 0.027 (5) |
| C3 | 0.4861 (16) | 0.0447 (6) | 0.7077 (15) | 0.014 (4) |
| C4 | 0.4726 (19) | 0.2017 (7) | 0.2718 (16) | 0.020 (4) |
| C5 | 0.2440 (16) | 0.2524 (6) | 0.2720 (15) | 0.011 (4) |
| C6 | 0.2631 (19) | 0.1695 (7) | 0.1446 (18) | 0.025 (4) |
| C7 | 0.4939 (20) | 0.1004 (7) | 0.3768 (17) | 0.026 (5) |
| C8 | 0.3219 (17) | 0.0330 (7) | 0.4914 (15) | 0.017 (4) |
| C9 | 0.2329 (20) | 0.0778 (7) | 0.2941 (18) | 0.027 (5) |
| C10 | 0.4876 (18) | 0.2125 (7) | 0.5298 (16) | 0.020 (4) |
| C11 | 0.2408 (16) | 0.2544 (7) | 0.5280 (15) | 0.013 (4) |
| C12 | 0.3102 (17) | 0.2064 (7) | 0.7186 (16) | 0.018 (4) |
| C13 | −0.0299 (17) | 0.2014 (7) | 0.5110 (16) | 0.016 (4) |
| C14 | −0.0397 (21) | 0.1010 (8) | 0.4118 (18) | 0.030 (5) |
| C15 | 0.0230 (18) | 0.1802 (7) | 0.2907 (17) | 0.021 (4) |
| C16 | 0.2247 (18) | 0.0824 (7) | 0.7898 (17) | 0.021 (4) |
| C17 | 0.0564 (17) | 0.1525 (7) | 0.7363 (16) | 0.018 (4) |
| C18 | 0.0532 (17) | 0.0562 (7) | 0.6265 (15) | 0.015 (4) |
| Cl1 | 0.8082 (7) | 0.0716 (3) | −0.1473 (6) | 0.066 (2) |
| Cl2 | 0.6430 (10) | 0.0466 (4) | 0.0421 (9) | 0.110 (4) |
| Cl3 | 0.9223 (13) | 0.0538 (5) | 0.0812 (11) | 0.146 (5) |
| C1S | 0.7932 (28) | 0.0386 (12) | −0.0140 (23) | 0.062 (8) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Os1—Os3 | 2.7890 (12) | Os5—C15 | 1.88 (2) |
| Os1—Os4 | 2.8313 (11) | Os6—C16 | 1.89 (2) |
| Os1—Os6 | 2.8043 (12) | Os6—C17 | 1.89 (2) |
| Os2—Os3 | 2.8249 (11) | Os6—C18 | 1.90 (2) |
| Os2—Os4 | 2.8137 (12) | O1—C1 | 1.15 (2) |
| Os2—Os5 | 2.7829 (12) | O2—C2 | 1.16 (2) |
| Os3—Os4 | 2.7605 (11) | O3—C3 | 1.18 (2) |
| Os3—Os5 | 2.7875 (11) | O4—C4 | 1.16 (2) |
| Os3—Os6 | 2.8159 (12) | O5—C5 | 1.15 (2) |
| Os4—Os5 | 2.8050 (12) | O6—C6 | 1.12 (2) |
| Os4—Os6 | 2.8006 (11) | O7—C7 | 1.14 (2) |
| Os5—Os6 | 2.7395 (11) | O8—C8 | 1.14 (2) |
| Os1—C1 | 1.88 (2) | O9—C9 | 1.16 (2) |
| Os1—C2 | 1.90 (2) | O10—C10 | 1.18 (2) |
| Os1—C3 | 1.84 (2) | O11—C11 | 1.11 (2) |
| Os2—C4 | 1.87 (2) | O12—C12 | 1.17 (2) |
| Os2—C5 | 1.87 (2) | O13—C13 | 1.14 (2) |
| Os2—C6 | 1.92 (2) | O14—C14 | 1.13 (2) |
| Os3—C7 | 1.95 (2) | O15—C15 | 1.17 (2) |
| Os3—C8 | 1.93 (2) | O16—C16 | 1.17 (2) |
| Os3—C9 | 1.93 (2) | O17—C17 | 1.14 (2) |
| Os4—C10 | 1.89 (2) | O18—C18 | 1.14 (2) |
| Os4—C11 | 1.94 (2) | Cl1—C1S | 1.76 (3) |
| Os4—C12 | 1.89 (2) | Cl2—C1S | 1.67 (3) |
| Os5—C13 | 1.89 (2) | Cl3—C1S | 1.72 (3) |
| Os5—C14 | 1.95 (2) | | |
| Os3—Os1—Os6 | 60.46 (3) | Os3—Os4—Os2 | 60.89 (3) |
| Os3—Os1—Os4 | 58.83 (3) | Os6—Os4—Os2 | 109.10 (3) |
| Os6—Os1—Os4 | 59.59 (3) | Os5—Os4—Os2 | 59.38 (3) |
| Os5—Os2—Os4 | 60.16 (3) | Os3—Os4—Os1 | 59.82 (3) |
| Os5—Os2—Os3 | 59.61 (3) | Os6—Os4—Os1 | 59.72 (3) |
| Os4—Os2—Os3 | 58.62 (2) | Os5—Os4—Os1 | 107.97 (4) |
| Os4—Os3—Os5 | 60.74 (3) | Os2—Os4—Os1 | 115.13 (3) |
| Os4—Os3—Os1 | 61.35 (2) | Os6—Os5—Os2 | 111.83 (4) |
| Os5—Os3—Os1 | 109.69 (4) | Os6—Os5—Os3 | 61.25 (3) |
| Os4—Os3—Os6 | 60.28 (2) | Os2—Os5—Os3 | 60.95 (3) |
| Os5—Os3—Os6 | 58.53 (3) | Os6—Os5—Os4 | 60.66 (3) |
| Os1—Os3—Os6 | 60.04 (3) | Os2—Os5—Os4 | 60.47 (3) |
| Os4—Os3—Os2 | 60.48 (3) | Os3—Os5—Os4 | 59.15 (3) |
| Os5—Os3—Os2 | 59.45 (3) | Os5—Os6—Os4 | 60.83 (3) |
| Os1—Os3—Os2 | 116.13 (3) | Os5—Os6—Os1 | 110.64 (4) |

| | | | |
|---|---|---|---|
| Os6—Os3—Os2 | 108.35 (3) | Os4—Os6—Os1 | 60.68 (3) |
| Os3—Os4—Os6 | 60.84 (3) | Os5—Os6—Os3 | 60.21 (3) |
| Os3—Os4—Os5 | 60.11 (3) | Os4—Os6—Os3 | 58.88 (3) |
| Os6—Os4—Os5 | 58.51 (3) | Os1—Os6—Os3 | 59.50 (3) |

An absorption correction based on $\psi$-scan data was applied, followed by an empirical absorption correction at isotropic convergence (*DIFABS*; Walker & Stuart, 1983). Data collection and cell refinement: *DIF*4 (Stoe & Cie, 1988*a*). Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*).

We thank BP Chemicals Ltd for support (JGMN) and the SERC for the provision of a diffractometer.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1094). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1994). C**50**, 1054–1057

# Sodium Cyclododecyl Sulfate Trihydrate, $Na^+.C_{12}H_{23}O_4S^-.3H_2O$

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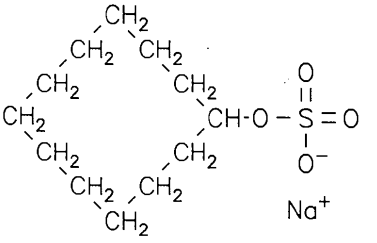
(*Received* 13 *September* 1993; *accepted* 10 *December* 1993)

## Abstract

The conformation of the cyclododecyl ring has slightly distorted 422 symmetry. The molecules are packed in a head-to-head arrrangement. The hydrophilic parts of neighbouring molecular layers are connected *via* coordination to $Na^+$ and hydrogen bonds.

## Comment

The continuing interest in the structural analogies of three-dimensional crystals of amphiphiles and their two-dimensional mono- or bilayers is based on comparable intermolecular interactions within the layer planes. In a systematic study of the effect of structural elements there are different possibilities for modifying the amphiphilic nature of a model surfactant, such as sodium dodecyl sulfate. The important role of counter ions in the structural arrangement can be demonstrated by a comparison of the crystal structures of sodium dodecyl sulfate (Sundell, 1977) and butylammonium dodecyl sulfate (Szulzewsky, Schulz & Vollhardt, 1983). However, no systematic information is available on the structural effect of the hydrophobic part of the amphiphilic molecule. Therefore, the long linear hydrocarbon chain of sodium *n*-dodecyl sulfate has been replaced by a large ring hydrocarbon with the same number of C atoms. The asymmetric unit of the title compound, (I), with the atomic numbering scheme is illustrated in Fig. 1.




(I)

As observed previously in different sodium dodecyl sulfate hydrate crystals (Sundell, 1977; Coiro, Mazza & Pochetti, 1986; Coiro, Manigrasso, Mazza & Pochetti, 1987), the three bonds S—O2, S—O3, S—O4 (average length 1.447 Å) are shorter than the ester bond S—O1 [1.571 (4) Å] and the three O—S—O bond angles involving O1 (average bond angle 105.4°) are smaller than the

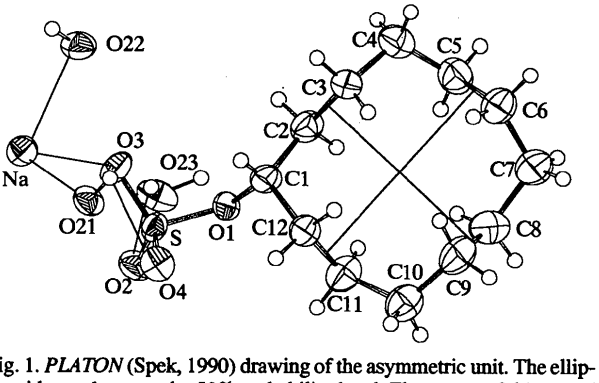



Fig. 1. *PLATON* (Spek, 1990) drawing of the asymmetric unit. The ellipsoids are drawn at the 50% probability level. The two twofold axes of the ring, the $Na^+\cdots O$ coordinations and some of the hydrogen bonds are indicated by thin lines. Three water H atoms could not be determined and are therefore not shown.