C(1)	0.9750 (5)	−0.1129 (7)	0.2718 (2)	0.030 (1)
C(2)	1.0424 (5)	0.0921 (7)	0.2602 (2)	0.030 (1)
C(3)	1.0534 (5)	0.1594 (6)	0.2057 (2)	0.029 (1)
C(4)	0.9965 (5)	0.0244 (6)	0.1643 (1)	0.0246 (10)
C(5)	0.9313 (5)	−0.1788 (6)	0.1752 (2)	0.032 (1)
C(6)	0.9209 (5)	−0.2426 (7)	0.2293 (2)	0.034 (1)
C(7)	0.9568 (7)	−0.1862 (8)	0.3304 (2)	0.043 (1)

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

Co—O(4)	2.111 (3)	S—O(2)	1.463 (2)
Co—O(5)	2.085 (3)	S—O(3)	1.464 (2)
Co—O(6)	2.046 (3)	S—C(4)	1.776 (3)
S—O(1)	1.454 (2)	N—C(2)	1.387 (5)
O(4)—Co—O(5)	92.9 (1)	O(2)—S—C(4)	106.0 (2)
O(4)—Co—O(6)	90.5 (1)	O(3)—S—C(4)	107.5 (2)
O(5)—Co—O(6)	90.9 (1)	N—C(2)—C(1)	121.1 (4)
O(1)—S—O(2)	112.3 (2)	N—C(2)—C(3)	119.5 (4)
O(1)—S—O(3)	112.3 (2)	S—C(4)—C(3)	120.3 (3)
O(1)—S—C(4)	106.5 (2)	S—C(4)—C(5)	118.2 (3)
O(2)—S—O(3)	111.8 (2)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O(4)—H(9)···O(3^{i})	0.84 (4)	1.97 (4)	2.813 (4)	176 (4)
O(4)—H(10)···O(2^{ii})	0.80 (4)	1.97 (4)	2.759 (4)	168 (4)
O(5)—H(11)···O(1^{iii})	0.71 (4)	2.10 (4)	2.800 (4)	173 (5)
O(5)—H(12)···O(3^{iv})	0.86 (4)	1.98 (4)	2.838 (4)	174 (4)
O(6)—H(13)···O(1^{iv})	0.88 (4)	1.89 (4)	2.762 (4)	174 (3)
O(6)—H(14)···O(2^{i})	0.83 (4)	1.92 (4)	2.749 (4)	172 (4)

Symmetry codes: (i) $\frac{5}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{5}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iii) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iv) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

A statistical comparison of the intensities of 18 *hkl*/$\bar{h}$*kl* pairs prior to data collection revealed the absence of a mirror plane perpendicular to **a**, confirming the crystal system as monoclinic. Phenyl C—C distances were in the range 1.375 (5)–1.403 (5), with a mean of 1.389 (5) Å. All H atoms were located on difference electron-density maps and their positions refined with fixed isotropic *B* values equal to 1.2 times those of the attached atoms at the time of their inclusion. Bond distances were C—H 0.89 (3)–1.02 (4), N—H(7) 0.96 (4) and N—H(8) 0.85 (4) Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1991). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1983). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, least-squares-planes data, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FR1012). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[2,2,2-Crypt-Na]$_2$[Cr$_2$(CO)$_{10}$]: a Chromium(−1) Carbonyl Structure with a Very Weakly Coordinating Cation

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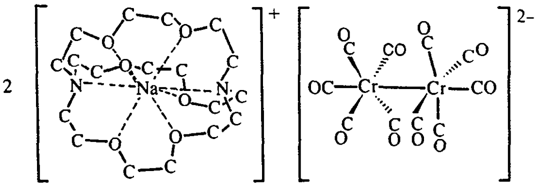
Abstract

The title compound, bis[(1,10-diaza-4,7,13,16,21,24-hexaoxabicyclo[8.8.8]hexacosane)sodium(+)] decacarbonyldichromate(*Cr*—*Cr*)(2−), [Na(C$_{18}$H$_{36}$N$_2$O$_6$)]$_2$[Cr$_2$(CO)$_{10}$], has been prepared by the reduction of [Cr(CO)$_6$] with Na$_2$Tl in dimethylformamide followed by the addition of the macrobicyclic ligand 2,2,2-crypt in ethylenediamine (en) solvent. The geometric parameters observed for the anion in the title salt differ significantly from those reported previously for the DME-solvated (DME = 1,2-dimethoxyethane) K$^+$ and

$[PPN]^+$ {$[PPN]^+$ = bis[bis(triphenylphospine)iminium]} salts, reflecting the weaker coordinating nature of the [2,2,2-crypt-Na]$^+$ cation.

Comment

The dichromium decacarbonyl anion of the title salt, (1), $[Cr_2(CO)_{10}]^{2-}$, is used extensively in the preparation of substituted chromium–carbonyl complexes and has been synthesized by the reduction of $[Cr(CO)_6]$ using $NaBH_4$ (Behrens & Haag, 1961), sodium (Hayter, 1966) and caesium (Ellis, Hentges, Kalina & Hagen, 1975) amalgams in dry tetrahydrofuran (THF), Na dissolved in 2,2′-bipyridine (Lindner, Behrens & Birkle, 1968), C_8K in dry deoxygenated THF (Ungurenasu & Palie, 1975) and KSi in DME/toluene (Hey-Hawkins & von Schnering, 1991). The most recent synthesis of $[Cr_2(CO)_{10}]^{2-}$ involved the alkali metal (Li^+, Na^+) naphthalenide reduction of $[Cr(CO)_5N(CH_3)_3]$ (Maher, Beatty & Cooper, 1985) followed by metathesis with [PPN].Cl to give the $[PPN]^+$ salt of the anion (Lee, Geib & Cooper, 1996). We have prepared the $[Cr_2(CO)_{10}]^{2-}$ anion by the reduction of $[Cr(CO)_6]$ using Na_2Tl followed by sequestering of the Na^+ cations with the macrobicyclic ligand 2,2,2-crypt, to give the title $[2,2,2\text{-crypt-Na}]_2[Cr_2(CO)_{10}]$ salt.



(1)

Compound (1) consists of discrete [2,2,2-crypt-Na]$^+$ and $[Cr_2(CO)_{10}]^{2-}$ ionic units and crystallizes in the *C*2/*c* space group. The structure of the [2,2,2-crypt-Na]$^+$ cation (Fig. 1) is similar to that determined previously in $[2,2,2\text{-crypt-Na}]_4[Te_4]$ (Devereux, Schrobilgen & Sawyer, 1985) and therefore will not be discussed. The $[Cr_2(CO)_{10}]^{2-}$ anion in (1) possesses a staggered geometry (D_{4d} point symmetry) (Fig. 2) which has been previously observed in both $K_2[Cr_2(CO)_{10}]$.2DME (Hey-Hawkins & von Schnering, 1991) and $[PPN]_2[Cr_2(CO)_{10}]$ (Lee, Geib & Cooper, 1996). The structural parameters for the $[Cr_2(CO)_{10}]^{2-}$ anion in (1), however, differ significantly from those observed both in the DME-solvated K^+ salt, which contains polymeric one-dimensional $K^+\cdots[Cr_2(CO)_{10}]^{2-}$ chains possessing strong anion–cation interactions, and in the $[PPN]^+$ salt, which has been reported to contain discrete anions. Interestingly, the geometric anion parameters observed in the $[PPN]^+$ salt were shown to be similar to those observed in the K^+ salt. The Cr—Cr bond in (1) has a length of 2.9761 (8) Å and is significantly shorter ($> 3\sigma$) than the corresponding bonds in the K^+ [2.999 (2) Å] and $[PPN]^+$ [2.995 (1) Å] salts. The axial Cr—C bond length in (1) [1.828 (2) Å] is significantly longer than the corresponding average bond distances observed in the previously characterized salts, *i.e.* K^+ 1.802 (6) Å and $[PPN]^+$ 1.792 (4) Å. The average equatorial Cr—C [1.876 (4) Å], axial C—O [1.160 (2) Å] and equatorial C—O [1.154 (4) Å] bond distances observed in (1) are similar at the 3σ level to the corresponding average distances observed in the K^+ [equatorial Cr—C 1.861 (5) Å, axial C—O 1.180 (8) Å and equatorial C—O 1.157 (5) Å] and $[PPN]^+$ [equatorial Cr—C 1.879 (5) Å, axial C—O 1.177 (4) Å and equatorial C—O 1.154 (5) Å] salts. The differences in the structural parameters between the present and the previously characterized $[Cr_2(CO)_{10}]^{2-}$ anions can be attributed to stronger cation–anion interactions in the K^+ and $[PPN]^+$ salts which perturb the anion electron density to a greater extent. The [2,2,2-crypt-Na]$^+$ cations in (1) are therefore significantly weaker in terms of their coordination.

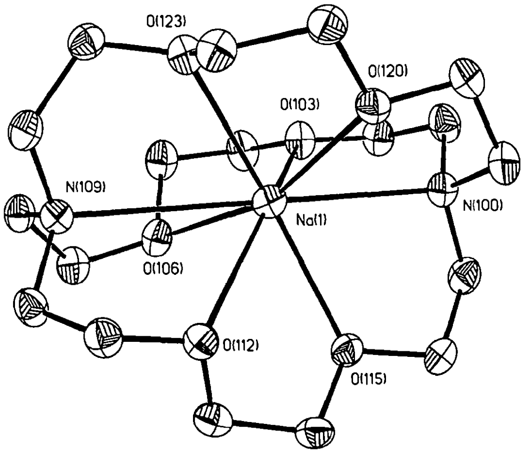


Fig. 1. *ORTEP*II (Johnson, 1976) view of the [2,2,2-crypt-Na]$^+$ cation in (1) with displacement ellipsoids drawn at the 50% probability level.

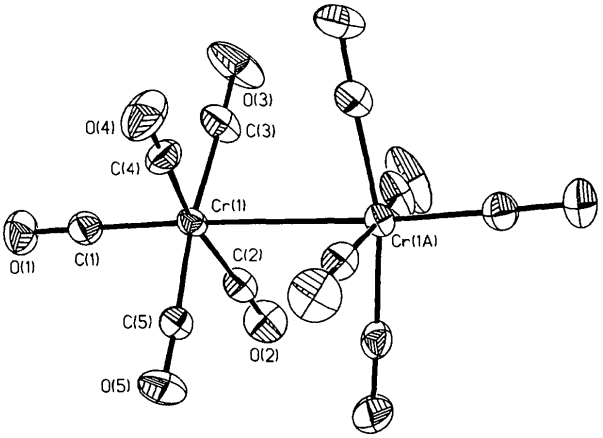


Fig. 2. *ORTEP*II (Johnson, 1976) view of the $[Cr_2(CO)_{10}]^{2-}$ anion in (1) with displacement ellipsoids drawn at the 50% probability level.

In all structures, the axial Cr—C bonds are shorter than the equatorial Cr—C bonds, which can be attributed to greater electron back donation from the metal centers onto the axial C atoms. The axial C—O bond distances are consequently longer than the equatorial ones.

It should be noted that the $[Cr_2(CO)_{10}]^{2-}$ anion has also been structurally characterized by X-ray crystallography in the CH_2Cl_2-solvated $[PPN]^+$ salt (Handy, Ruff & Dahl, 1970). Structural comparisons with the anion in this salt were not made, however, since the structure contained a poorly defined solvent molecule, lacked anisotropic thermal refinement and the crystal underwent a 19% decay.

Experimental

The extreme air- and moisture-sensitive natures of the compounds used required all manipulations to be carried out under rigorously anhydrous conditions and in the absence of oxygen both in a two-station nitrogen-atmosphere drybox (Vacuum Atmospheres Model DLX, with moisture and oxygen levels < 0.1 p.p.m.; for general solid and crystal handling) on a general-purpose grease-free glass vacuum line equipped with Pyrex glass/Teflon stopcocks (J. Young Scientific Glassware) and in a glovebag (for solution handling) which had been purged with dry nitrogen for at least 12 h prior to use. The known binary Na_2Tl alloy (Hansen & Smith, 1967) was prepared as previously described (Burns, Devereux, Granger & Schrobilgen, 1985) by fusion of the elements [Na (0.6248 g, 27.177 mmol) and Tl (2.8429 g, 13.910 mmol)] inside a thick-walled Pyrex glass tube. The finely crushed alloy (0.0759 g, 0.303 mmol) was then allowed to react with $[Cr(CO)_6]$ (0.0551 g, 0.251 mmol) in DMF over a period of one week in a sealed reactor under 1 atm of dry nitrogen. The resulting yellow solution was pipetted onto an ethylenediamine (en) solution of 2,2,2-crypt (0.1160 g, 0.308 mmol) contained in one arm of a two-arm Pyrex glass vessel. After 2 d, an excess of THF (1:2 *v*/*v*) was vacuum distilled at 273 K into the second arm of the vessel. Orange prisms of (1) were obtained by vapor-phase diffusion of the THF into the en/DMF solution under static vacuum.

Crystal data

$[Na(C_{18}H_{36}N_2O_6)]_2$-$[Cr_2(CO)_{10}]$
$M_r = 1183.06$
Monoclinic
$C2/c$
$a = 21.393\,(4)$ Å
$b = 11.586\,(2)$ Å
$c = 22.635\,(5)$ Å
$\beta = 90.09\,(3)°$
$V = 5610.2\,(19)$ Å^3
$Z = 4$
$D_x = 1.401$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 1131 reflections
$\mu = 0.482$ mm^{-1}
$T = 150\,(2)$ K
Prism
0.38 × 0.21 × 0.15 mm
Orange

Data collection

Stoe Imaging Plate Diffractometer System (IPDS)
Oscillation scans
Absorption correction: none
21 950 measured reflections

6860 independent reflections
$R_{int} = 0.0256$
$\theta_{max} = 29°$
$h = -30 \rightarrow 30$
$k = -14 \rightarrow 14$
$l = -27 \rightarrow 32$

Refinement

Refinement on F^2
$R(F)$ $[I > 2\sigma(I)] = 0.0341$
$wR(F^2) = 0.0879$
$S = 1.085$
6768 reflections
343 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 3.7329P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.003$
$\Delta\rho_{max} = 0.318$ e Å^{-3}
$\Delta\rho_{min} = -0.433$ e Å^{-3}
Extinction correction: none
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 equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cr1	0.01994 (1)	0.50458 (2)	0.18705 (1)	0.02159 (7)
C1	0.03908 (8)	0.50120 (14)	0.10839 (8)	0.0301 (3)
O1	0.04975 (7)	0.49875 (13)	0.05816 (6)	0.0443 (3)
C2	−0.06179 (8)	0.44865 (15)	0.17537 (7)	0.0301 (3)
O2	−0.11183 (6)	0.41476 (13)	0.16676 (7)	0.0451 (3)
C3	0.05028 (10)	0.3557 (2)	0.20234 (8)	0.0389 (4)
O3	0.06944 (11)	0.26441 (14)	0.21084 (7)	0.0725 (6)
C4	0.09467 (7)	0.5685 (2)	0.21604 (8)	0.0298 (3)
O4	0.13922 (6)	0.61243 (14)	0.23412 (7)	0.0467 (4)
C5	−0.00757 (7)	0.65735 (14)	0.18572 (7)	0.0270 (3)
O5	−0.02049 (7)	0.75413 (11)	0.18493 (7)	0.0431 (3)
Na1	0.16005 (3)	0.99615 (5)	−0.03903 (3)	0.02524 (13)
N100	0.15721 (6)	1.00196 (11)	0.08087 (6)	0.0243 (3)
C101	0.11426 (8)	1.09428 (15)	0.09840 (8)	0.0312 (3)
C102	0.12650 (8)	1.20701 (14)	0.06638 (7)	0.0303 (3)
O103	0.12590 (5)	1.18589 (9)	0.00463 (5)	0.0276 (2)
C104	0.13575 (9)	1.29016 (14)	−0.02794 (8)	0.0332 (4)
C105	0.13970 (8)	1.26018 (15)	−0.09199 (8)	0.0324 (3)
O106	0.19482 (6)	1.19332 (10)	−0.10159 (5)	0.0297 (2)
C107	0.21207 (8)	1.18708 (14)	−0.16231 (7)	0.0296 (3)
C108	0.16762 (7)	1.11434 (13)	−0.19963 (7)	0.0260 (3)
N109	0.16382 (6)	0.99499 (11)	−0.17867 (6)	0.0223 (2)
C110	0.21791 (7)	0.92820 (14)	−0.19840 (7)	0.0269 (3)
C111	0.22955 (7)	0.82393 (14)	−0.15928 (7)	0.0274 (3)
O112	0.23867 (5)	0.85302 (10)	−0.09871 (5)	0.0269 (2)
C113	0.29701 (7)	0.90970 (15)	−0.08729 (8)	0.0287 (3)
C114	0.30948 (7)	0.90051 (15)	−0.02223 (8)	0.0292 (3)
O115	0.26294 (5)	0.96523 (10)	0.00817 (5)	0.0256 (2)
C116	0.26943 (7)	0.95114 (15)	0.07040 (7)	0.0284 (3)
C117	0.22132 (8)	1.0260 (2)	0.10077 (7)	0.0299 (3)
C118	0.13553 (8)	0.89062 (15)	0.10339 (7)	0.0298 (3)
C119	0.07921 (8)	0.84410 (15)	0.07042 (7)	0.0290 (3)
O120	0.09424 (5)	0.83958 (10)	0.00949 (5)	0.0256 (2)
C121	0.04400 (7)	0.79236 (14)	−0.02436 (7)	0.0288 (3)
C122	0.05783 (8)	0.81395 (13)	−0.08833 (7)	0.0277 (3)
O123	0.06012 (5)	0.93594 (9)	−0.09724 (5)	0.0263 (2)
C124	0.05080 (7)	0.96998 (14)	−0.15725 (7)	0.0276 (3)
C125	0.10517 (7)	0.93993 (14)	−0.19746 (7)	0.0265 (3)

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

Cr1—C1	1.828 (2)	C5—O5	1.155 (2)
Cr1—C2	1.883 (2)	Na1—N100	2.716 (2)
Cr1—C3	1.875 (2)	Na1—O103	2.5190 (13)
Cr1—C4	1.879 (2)	Na1—O106	2.7893 (14)
Cr1—C5	1.865 (2)	Na1—N109	3.162 (2)
Cr1—Cr1^{i}	2.9761 (8)	Na1—O112	2.7222 (14)
C1—O1	1.160 (2)	Na1—O115	2.4715 (13)
C2—O2	1.157 (2)	Na1—O120	2.5461 (14)
C3—O3	1.150 (2)	Na1—O123	2.6046 (14)
C4—O4	1.155 (2)		

C1—Cr1—C2	93.74 (8)	O103—Na1—O106	64.04 (4)
C1—Cr1—C3	94.71 (8)	O103—Na1—O112	154.97 (4)
C1—Cr1—C4	99.02 (8)	O103—Na1—O120	106.96 (5)
C1—Cr1—C5	94.35 (7)	O103—Na1—O123	101.18 (4)
C1—Cr1—Cr1^{i}	176.10 (5)	O106—Na1—N109	59.25 (5)
C2—Cr1—C4	166.89 (7)	N109—Na1—N100	178.80 (5)
C2—Cr1—Cr1^{i}	82.36 (6)	O112—Na1—O106	94.67 (4)
C3—Cr1—C2	91.74 (9)	O112—Na1—N109	58.98 (5)
C3—Cr1—C4	90.22 (8)	O115—Na1—N100	65.97 (5)
C3—Cr1—Cr1^{i}	85.57 (6)	O115—Na1—O103	102.45 (4)
C4—Cr1—Cr1^{i}	84.87 (6)	O115—Na1—O106	95.74 (4)
C5—Cr1—C2	91.81 (7)	O115—Na1—O112	64.89 (4)
C5—Cr1—C3	170.03 (7)	O115—Na1—O120	101.71 (4)
C5—Cr1—C4	84.26 (7)	O115—Na1—O123	155.46 (5)
C5—Cr1—Cr1^{i}	85.67 (5)	O120—Na1—N100	64.75 (4)
O1—C1—Cr1	178.4 (2)	O120—Na1—O106	161.90 (4)
O2—C2—Cr1	178.4 (2)	O120—Na1—O112	97.03 (4)
O3—C3—Cr1	178.8 (2)	O120—Na1—O123	64.76 (4)
O4—C4—Cr1	176.9 (2)	O123—Na1—N100	119.52 (5)
O5—C5—Cr1	175.46 (14)	O123—Na1—O106	100.48 (4)
N100—Na1—O106	119.57 (4)	O123—Na1—N109	61.03 (4)
N100—Na1—O112	121.72 (5)	O123—Na1—O112	95.34 (4)
O103—Na1—N100	65.12 (4)		

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

Crystal data were collected on a single crystal sealed under dry nitrogen in a 0.2 mm Lindemann glass capillary. Data were collected in two stages: (*a*) 433 exposures (5 min per exposure) were obtained at 50 mm with $40 \leq \varphi \leq 169.9°$ and with the crystal oscillated through 0.3° in φ; (*b*) 160 exposures (3 min per exposure) were obtained at 125 mm with $40 \leq \varphi \leq 200°$ and with the crystal oscillated through 1° in φ. The two data sets were merged and the final data set contained 92% of the calculated number of unique reflections. Computations were carried out on a Silicon Graphics Model 4600 PC workstation. During refinement, 92 reflections were either omitted if $F^2 < 0$ or flagged for potential systematic errors. H-atom positions were calculated and C—H was fixed to 0.96 Å, with $U_{iso}(H) = 1.2U_{eq}$ of the parent atom.

Data collection: *IPDS* (Stoe, 1995). Cell refinement: *IPDS*. Data reduction: *IPDS*. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BR1160). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bisphenylbis[tetracarbonyl(hydrido)osmio]germane, $Ph_2Ge[Os(CO)_4H]_2$

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Abstract

The title compound, octacarbonyl-1κ^4C,2κ^4C-dihydrido-1κH,2κH-bis(phenyl-3κC)germaniumdiosmium(2 *Ge—Os*), $[Os_2(H)_2(CO)_8\{Ge(C_6H_5)_2\}]$, is the first cluster containing a single Os—*E*—Os (*E* = Ge, Sn or Pb) chain to be structurally characterized. The molecule possesses approximate C_2 symmetry. The Os—Ge—Os metal chain is bent and the Os—Ge bonds [2.599 (2) and 2.614 (2) Å] are the longest yet known.

Comment

The title compound, (I), was obtained in low yield as a by-product from the preparation of $[Ph_2SnOs(CO)_4]_2$ by the reaction of equimolar amounts of Ph_2GeCl_2 and $Na_2Os(CO)_4$. We believe that its formation resulted from partial hydrolysis of the very moisture-sensitive $Na_2Os(CO)_4$ to $NaHOs(CO)_4$, which subsequently attacked the Ph_2GeCl_2.

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