Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Tricarbonyl(η^5-cyclopentadienyl)(3-iodopropyl)-tungsten(II)

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Received 22 November 2004
Accepted 7 December 2004
Online 18 December 2004

In the title compound, $[W(C_5H_5)(C_3H_6I)(CO)_3]$, the W atom is coordinated by a cyclopentadienyl ligand, three carbonyl ligands and an iodoalkyl chain in a distorted square-pyramidal arrangement. In the crystal structure, the molecules pack with the iodoalkyl chains in non-interdigitated layers.

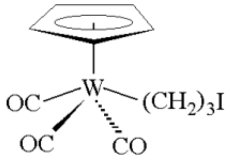
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.032
wR factor = 0.078
Data-to-parameter ratio = 27.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

This structure determination was undertaken as part of our ongoing study of halogenoalkyl transition metal compounds (Friedrich *et al.*, 2004). Reported structures of halogenoalkyl transition metal complexes are rare and, to our knowledge, only four structures are known where the halogenoalkyl group has a chain length exceeding one C atom (Friedrich *et al.*, 2004).



(I)

The title compound, $CpW(CO)_3(n\text{-}C_3H_6I)$, (I) (Cp = η^5-C_5H_5), is, to our knowledge, the first crystal structure reported for a tungsten iodopropyl molecule. The structures of the related tungsten halogenoalkyl compounds, $CpW(CO)_3(C_5H_{10}I)$, (II), and $CpW(CO)_3(C_3H_6Br)$, (III), have been reported previously (Friedrich *et al.*, 2001).

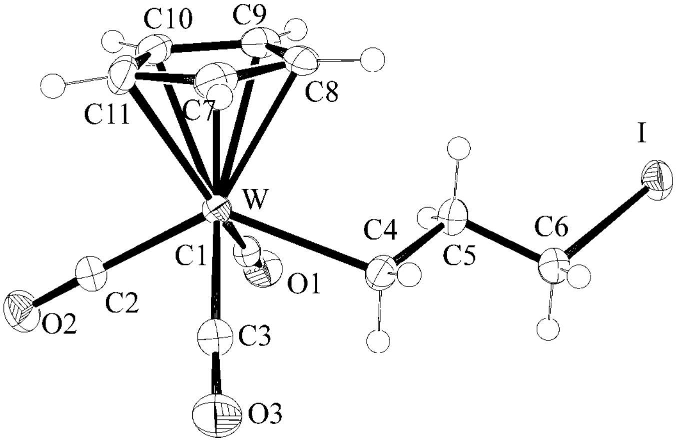


Figure 1
The molecular structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

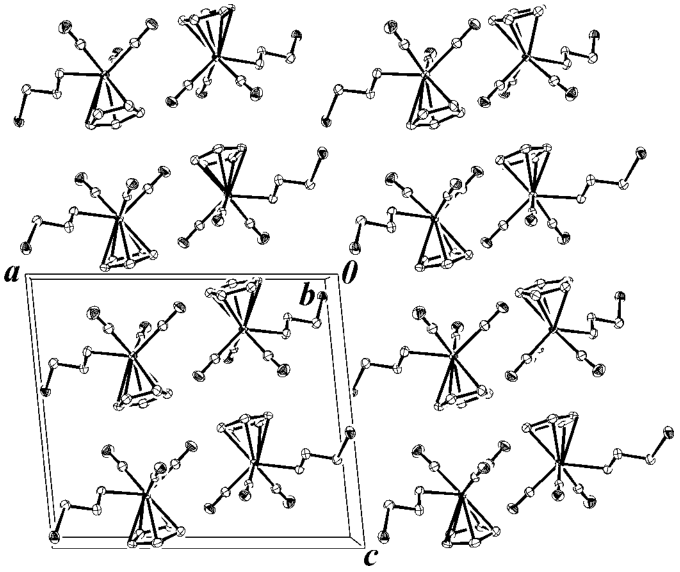


Figure 2
A packing diagram for (I), viewed down the *b* axis. H atoms have been omitted.

In (I), the W atom is coordinated in a distorted square-pyramidal fashion by three carbonyl ligands, an η^5-cyclopentadienyl ligand (assuming a single bond to the centroid) and a C-bonded iodoalkyl chain (Table 1). The molecular geometry and numbering scheme are illustrated in Fig. 1.

The five W—C bonds to the cyclopentadienyl C atoms range from 2.313 (5) Å to 2.382 (5) Å. In the halogenoalkyl chain, the C atoms are in the energetically favoured all-*trans* conformation, and are almost coplanar with the atoms W, O3 and C3 (r.m.s deviation 0.0132 Å). The I atom, however, is 0.278 (1) Å out of this plane. The C—I bond length of 2.169 (5) Å is similar to the value of 2.121 (10) Å observed for (II), and the W—C4 bond of 2.329 (5) Å compares favourably with values of 2.348 (10) Å reported for (II), and 2.347 (15) Å and 2.336 (14) Å reported for (III).

The molecular packing in (I), as viewed down the *b* axis, is shown in Fig. 2. In the crystal structure, the molecules assemble into layers such that the non-interdigitated haloalkyl chains face each other on one side of the layer, with the rest of each molecule on the other. The plane through the C atoms of the haloalkyl chain is tilted by 66.6 (4)° relative to the (100) plane, and the cyclopentadienyl plane (r.m.s. deviation 0.0049 Å) is tilted by 80.82 (14)° relative to this (100) plane. A short contact of 3.6028 (13) Å is observed between neighbouring I atoms.

The structure of compound (II), where the iodopropyl chain in (I) is substituted by a longer iodopentyl chain, is different from the structures of the iodopropyl-containing compounds. In this material, a layered molecular packing is not observed, and pairs of molecules pack with interdigitated iodopentyl chains.

Experimental

The title compound was prepared according to the procedure of Friedrich *et al.*, 2001) and crystals were grown by slow evaporation of a mixture of dichloromethane and hexane (1:4) at 278 K.

Crystal data

$[W(C_5H_5)(C_3H_6I)(CO)_3]$
$M_r = 501.95$
Monoclinic, $P2_1/c$
$a = 14.509$ (5) Å
$b = 6.9285$ (19) Å
$c = 12.806$ (3) Å
$\beta = 95.62$ (4)°
$V = 1281.1$ (6) Å^3
$Z = 4$

$D_x = 2.602$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 624 reflections
$\theta = 2.2$–31.4°
$\mu = 11.42$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.20 × 0.20 × 0.10 mm

Data collection

Oxford Excalibur2 CCD diffractometer
$\omega/2\theta$ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.123$, $T_{\max} = 0.319$
8459 measured reflections

3934 independent reflections
3333 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\max} = 31.9°$
$h = -21 \rightarrow 13$
$k = -10 \rightarrow 10$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.078$
$S = 1.00$
3934 reflections
145 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.045P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 2.13$ e Å^{-3}
$\Delta\rho_{\min} = -2.58$ e Å^{-3}
Extinction correction: none

Table 1
Selected bond lengths (Å).

W—C1	1.983 (5)	W—C8	2.381 (5)
W—C2	1.987 (5)	W—C9	2.339 (5)
W—C3	1.987 (5)	W—C10	2.313 (5)
W—C4	2.329 (5)	W—C11	2.320 (5)
W—C7	2.382 (5)		

During this study, it was established that the structure of (III), $CpW(CO)_3(C_3H_6Br)$ (Friedrich *et al.*, 2001), was reported in a space group of too low a symmetry (triclinic, space group $P\overline{1}$), and is actually monoclinic and isostructural with the title compound. Substitution of the Br atom in (III) by the larger I atom in (I) results in a slight expansion of two of the unit-cell parameters and a slight contraction of the third. All H atoms were placed in calculated positions, with C—H distances of 0.96–0.97 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C), and were refined using a riding model. The maximum and minimum electron-density peaks are 0.72 Å and 0.78 Å, respectively, from W at (0.3173, 0.7293, 0.6985) and (0.2722, 0.6244, 0.6624).

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlisCCD*; data reduction: *CrysAlisRED* (Oxford Diffraction, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

Acta Cryst. (2005). E**61**, m144–m146 Friedrich *et al.* • $[W(C_5H_5)(C_3H_6I)(CO)_3]$ **m145**

We thank the NRF (South Africa) and DAAD (Germany) for support, and the University of Natal for a post-doctoral fellowship (MOO). We also thank Ms Z. Mabuza for assistance in the laboratory.

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