

Fig. 1. 50% probability ellipsoids of the cation showing the atom-numbering scheme.

thermal ellipsoids drawing (*SHELXTL-Plus*, Sheldrick, 1987) of the cation with the atom-labelling scheme is given in Fig. 1.

**Related literature.** For synthesis of related compounds refer to Lee, Gill, Iqbal, Azogu & Sutherland (1982) and Sutherland, Piórko, Chowdbury & Lee (1987). For structurally similar compounds refer to Abboud, Simonsen, Piórko & Sutherland (1990, 1991), Sutherland, Piórko, Lee, Simonsen & Lynch (1988), Lynch, Thomas, Simonsen, Piórko & Sutherland (1986) and Simonsen, Lynch, Sutherland & Piórko (1985).

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## Structure of Dicarbonyl(dimethylphosphonate)iodo( $\eta^5$ -pentamethylcyclopentadienyl)-rhenium(III)

BY F. W. B. EINSTEIN AND C. E. F. RICKARD

*Department of Chemistry, Simon Fraser University, Burnaby, British Columbia, Canada V5A 1S6*

AND H. KLAHN AND C. LEIVA

*Instituto Quimica, Universidad Catolica de Valparaiso, Avenida Brasil 2950, Casilla 4059, Valparaiso, Chile*

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**Abstract.**  $[\text{Re}(\text{C}_2\text{H}_6\text{O}_3\text{P})(\text{C}_{10}\text{H}_{15})(\text{CO})_2\text{I}]$ ,  $[\text{Cp}^*\text{-Re}(\text{CO})_2\{\text{PO}(\text{OMe})_2\}\text{I}]$  ( $\text{Cp}^*$  = pentamethylcyclopentadienyl),  $M_r = 613.4$ , trigonal axes,  $R\bar{3}$ ,  $a = 36.833$  (6)  $c = 7.535$  (2)  $\text{\AA}$ ,  $V = 8853$   $\text{\AA}^3$ ,  $Z = 18$ ,  $D_x = 2.071$   $\text{g cm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71069$   $\text{\AA}$ ,  $\mu = 79.1$   $\text{cm}^{-1}$ ,  $F(000) = 5183$ , room temperature, final  $R = 0.044$ ,  $wR = 0.060$  for 1608 observed reflections.

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The structure may be described as a five coordinate square pyramid with the Cp ring centroid occupying the unique apical site and the CO groups mutually *trans* in the square base. The ring is closely planar with the C atoms of the methyl groups displaced on average 0.18  $\text{\AA}$  away from the mean plane on the opposite side to the Re atom. There are only weak

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Table 1. Atomic coordinates for  $\text{Cp}^*\text{Re}(\text{CO})_2[\text{PO}(\text{OMe})_2]\text{I}$ 

	x	y	z	$B_{\text{iso}}(\text{\AA}^2)\dagger$
Re	0.08908 (3)	0.61435 (3)	0.0277 (1)	2.63
I	0.01978 (5)	0.59288 (5)	-0.1897 (2)	4.86
P	0.16382 (15)	0.6484 (2)	-0.0226 (7)	3.13
O1	0.1925 (4)	0.6845 (4)	0.087 (2)	4.0 (3)
O2	0.1747 (4)	0.6114 (4)	-0.016 (2)	4.5 (3)
O3	0.1700 (5)	0.6613 (4)	-0.229 (2)	4.8 (3)
O4	0.1082 (5)	0.6987 (5)	-0.132 (2)	5.3 (3)
O5	0.0950 (5)	0.5568 (5)	-0.249 (3)	6.3 (4)
C1	0.1103 (6)	0.6069 (6)	0.306 (3)	2.9 (4)
C2	0.0761 (6)	0.5668 (6)	0.260 (3)	3.2 (4)
C3	0.0394 (6)	0.5712 (6)	0.241 (3)	3.2 (4)
C4	0.0525 (6)	0.6146 (6)	0.272 (3)	3.5 (4)
C5	0.0968 (6)	0.6380 (6)	0.315 (3)	3.5 (4)
C6	0.1523 (6)	0.6147 (6)	0.374 (3)	3.3 (4)
C7	0.0780 (6)	0.5283 (6)	0.258 (3)	3.8 (4)
C8	-0.0044 (7)	0.5357 (7)	0.222 (4)	5.0 (5)
C9	0.0226 (7)	0.6316 (7)	0.283 (3)	5.0 (5)
C10	0.1211 (6)	0.6815 (6)	0.371 (3)	4.1 (4)
C11	0.2162 (9)	0.6202 (9)	0.002 (4)	7.1 (7)
C12	0.2083 (9)	0.6953 (9)	-0.291 (4)	7.1 (7)
C13	0.1024 (6)	0.6663 (6)	-0.079 (3)	3.8 (4)
C14	0.0932 (6)	0.5799 (7)	-0.156 (3)	4.1 (4)

$\dagger B_{\text{iso}}$  is the mean of the principal axes of the ellipsoid.

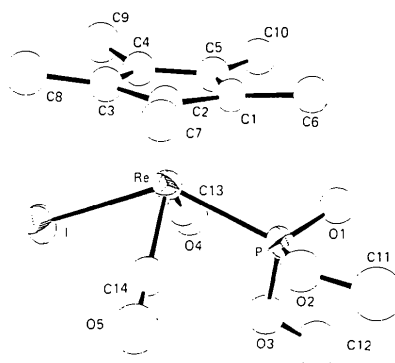


Fig. 1. The structure of  $\text{Cp}^*\text{Re}(\text{CO})_2[\text{PO}(\text{OMe})_2]\text{I}$  showing the atomic numbering.

interactions between the H atoms of the  $\text{Cp}^*$  methyl groups and the diphenylphosphate O atoms, O1—H6b 2.38 and O2—H6a 2.49 Å.

**Experimental.** Pale yellow crystals of the title compound obtained from dichloromethane were mounted on glass fibres. Crystal dimensions  $0.07 \times 0.11 \times 0.41$  mm. Cell parameters were obtained from the four-circle coordinates of 25 reflections in the range  $16 < 2\theta < 24^\circ$ . The unique data set was measured in the range  $4 < 2\theta < 47^\circ$  ( $-35 < h < 35$ ,  $0 < k < 41$ ,  $0 < l < 8$ ) using graphite-monochromated Mo  $K\alpha$  radiation on an Enraf-Nonius CAD-4 diffractometer. Intensities measured using  $\omega$ -scan technique. Two standard reflections monitored every hour of data collection showed no systematic variation. The data were corrected for Lorentz and polarization

Table 2. Interatomic distances (Å) for  $\text{Cp}^*\text{Re}(\text{CO})_2[\text{PO}(\text{OMe})_2]\text{I}$ 

Re—I	2.7942 (17)	O3—C12	1.42 (3)
Re—P	2.417 (5)	O4—C13	1.17 (3)
Re—C1	2.301 (18)	O5—C14	1.13 (3)
Re—C2	2.349 (19)	C1—C2	1.42 (3)
Re—C3	2.357 (19)	C1—C5	1.46 (3)
Re—C4	2.284 (19)	C1—C6	1.52 (3)
Re—C5	2.295 (20)	C2—C3	1.45 (3)
Re—C13	1.899 (21)	C2—C7	1.46 (3)
Re—C14	1.936 (22)	C3—C4	1.44 (3)
P—O1	1.471 (14)	C3—C8	1.49 (3)
P—O2	1.600 (15)	C4—C5	1.45 (3)
P—O3	1.607 (15)	C4—C9	1.52 (3)
O2—C11	1.40 (3)	C5—C10	1.45 (3)

effects and an analytical absorption correction applied, min. and max. transmission coefficients were 0.438 and 0.584. 2907 unique reflections were measured of which 1608 were considered observed,  $I > 2.5\sigma(I)$ .

The coordinates of the Re and I atoms were obtained by direct methods and the remaining atoms from subsequent difference electron density maps. The H atoms were fixed in calculated positions oriented with one H atom of each methyl group opposite the metal and thermal parameters 10% greater than those of the C atom to which they were attached. Refinement was by full-matrix least squares minimizing  $\sum w(|F_o| - |F_c|)^2$  with weights  $w = 1/[\sigma^2(F) + 0.002F^2]$ . The heavier atoms were assigned anisotropic thermal parameters but allowing C and O atoms to assume anisotropic motion did not produce a significant improvement in the agreement. At convergence the maximum shift/e.s.d. was 0.05 and the largest peak in a final difference map was  $1.3(2) e \text{ \AA}^{-3}$ , on the threefold axis. Final  $R = 0.044$ ,  $wR = 0.060$  for 1608 observed reflections. Programs used for data reduction, solution and structure refinement were from the *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989) system running on a MicroVAX II computer. Scattering factors for neutral atoms (including anomalous dispersion) were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The final positional and equivalent isotropic thermal parameters are given in Table 1.\* The structure, with the atomic numbering, is shown in Fig. 1 and interatomic distances are given in Table 2.

**Related literature.** Distances within the dimethylphosphonate ligand are comparable to those found in other complexes, P=O 1.51, P—O 1.62 Å

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53602 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Schubert, Werner, Zinner & Werner, 1983) and P=O 1.488, P—O 1.595 Å (Fernanda, Carvalho, Pombeiro, Hughes & Richards, 1987).

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## Structure of Di- $\mu$ -chloro-bis[trichlorobis(tetrahydrofuran)uranium(IV)]

BY J. REBIZANT, M. R. SPIRLET\* AND C. APOSTOLIDIS

*Joint Research Centre, Institute for Transuranium Elements, Postfach 2340, D-7500 Karlsruhe, Germany*

G. VAN DEN BOSSCHE†

*Cristallographie B5, Université de Liège au Sart Tilman, B-4000 Liège, Belgium*

AND B. KANELAKOPOULOS,

*Institut für Heisse Chemie, Kernforschungszentrum, Karlsruhe, Postfach 3640, D-7500 Karlsruhe, Germany*

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**Abstract.** [ $\{\text{UCl}_4(\text{C}_4\text{H}_8\text{O})_2\}_2$ ],  $M_r = 1048.12$ , monoclinic,  $P2_1/c$ ,  $a = 10.756$  (5),  $b = 12.372$  (5),  $c = 12.293$  (6) Å,  $\beta = 113.31$  (3)°,  $V = 1502$  (2) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.317$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 109.486$  cm<sup>-1</sup>,  $F(000) = 960$ ,  $T = 295$  (1) K,  $R = 0.037$  for 1287 observed unique reflections. The dimeric complex, in which two  $\text{UCl}_4(\text{THF})_2$  moieties are bridged by two Cl atoms, lies on a centre of symmetry. Each U atom of the dimer is seven coordinated in a pentagonal-bipyramidal geometry. The maximum deviation from the best plane through the U atom and its five equatorial ligand atoms [Cl(1), Cl(1)<sup>i</sup>, Cl(4) and the O atoms of the two THF] is 0.05 (1) Å. Cl(2)—U—Cl(3) is almost linear and perpendicular to this plane.

**Experimental.** The title complex was prepared by reaction of uranium tetrachloride with tetrahydrofuran in *n*-pentane at room temperature. Extraction and recrystallization with a 10% THF solution in *n*-pentane gave single crystals. The selected specimen (0.30 × 0.25 × 0.30 mm) was sealed in a thin-walled glass capillary under an inert atmosphere. Enraf-Nonius CAD-4 diffractometer was used with graphite-monochromated radiation. Unit-cell dimensions and standard deviations were obtained by least-squares fit to 25 reflections in the range 16–30°

( $2\theta$ ). The space group was unequivocally established from systematic absences. Intensity data were collected by  $\theta$ - $2\theta$  scans in the range  $4 < 2\theta < 46^\circ$ ,  $h\ 0 \rightarrow 12$ ,  $k\ -14 \rightarrow 14$ ,  $l\ -14 \rightarrow 14$ . In total 3829 reflections were measured to give 2081 unique reflections ( $R_{\text{int}} = 0.023$ ) of which 1287 were considered observed [ $I \geq 3\sigma(I)$ ] and used in refinement ( $h\ 0 \rightarrow 10$ ,  $k\ 0 \rightarrow 12$ ,  $l\ -12 \rightarrow 11$ ). Intensities of three standard reflections were measured at 30 min intervals, anisotropic decay correction was applied (total decay 3.9%). The data were corrected for Lorentz and polarization effects.  $\psi$  scans were used for the absorption correction (North, Phillips & Mathews, 1968), the min. and max. relative transmission factors were 73.32 and 99.95% respectively. The structure was solved by direct methods and refined by full-matrix least-squares techniques which minimized  $\sum w(\Delta F)^2$ ;  $w = 1/[\sigma(F_o)]^2$  with  $\sigma(F_o) = \sigma(F_o^2)/2F_o$  and  $\sigma(F_o^2) = [\sigma^2(I) + (A/I)^2]^{1/2}/Lp$  where  $A$ , the ignorance factor, is 0.06. Anisotropic temperature factors were used for non-H atoms. A secondary-extinction coefficient was refined to  $g = 8.9 \times 10^{-8} \{F_c = F_c/[1 + g(F_c)^2 Lp]\}$ . The final discrepancy indices were  $R = 0.037$ ,  $wR = 0.066$ ,  $S = 2.09$ . 137 variables were refined with maximum shift/e.s.d. in final cycle 0.03. H atoms were included in final structure factor calculations in idealized positions with  $B_{\text{iso}} = 4$  Å<sup>2</sup>. Largest positive and negative peaks on final  $\Delta\rho$  map had heights +1.16 and -1.39 e Å<sup>-3</sup>, respectively, with all substantial peaks close to U. Atomic scattering factors and

\* On leave from the University of Liège, Belgium.

† To whom correspondence should be addressed.