

Tricarbonyl(3-carboxypropyl)( $\eta^5$ -cyclopentadienyl)tungsten(II)Holger B. Friedrich,<sup>a</sup> R. Alan Howie<sup>b\*</sup> and Martin O. Onani<sup>c</sup><sup>a</sup>School of Pure and Applied Chemistry, Howard College, University of KwaZulu–Natal, Durban 4041, South Africa, <sup>b</sup>Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, and <sup>c</sup>Department of Chemistry, University of Transkei, Private Bag X1, UNITRA, 5117 Umtata, South Africa

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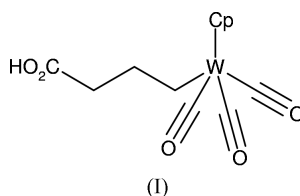
## Key indicators

Single-crystal X-ray study  
 $T = 295$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å  
 $R$  factor = 0.026  
 $wR$  factor = 0.072  
Data-to-parameter ratio = 13.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The bond lengths and angles in the title compound,  $[\text{W}(\text{C}_5\text{H}_5)(\text{C}_4\text{H}_7\text{O}_2)(\text{CO})_3]$ , are as expected for a molecule of this kind. The presence of the carboxylic acid group leads, however, to the creation of hydrogen-bonded dimers consisting of pairs of centrosymmetrically related molecules.

## Comment

The determination of the structure of the title compound, (I), was undertaken as part of our ongoing study of the chemistry of heterodinuclear compounds (Friedrich *et al.*, 2004). Fig. 1 is a drawing of the molecule and selected bond lengths and angles are given in Table 1. In both cases, the participation of the cyclopentadienyl (Cp) group in the coordination of W is represented, purely for convenience, by the notional bond  $\text{W}-\text{Cg1}$  where Cg1 is the centroid of the five-membered cyclopentadienyl ring. On this basis, W is effectively five-coordinate in a distorted square-pyramidal environment with apical Cp. This arrangement creates the appearance of a stool with Cp as its seat and the three carbonyl groups and the carboxy ligand as its feet, four in number.

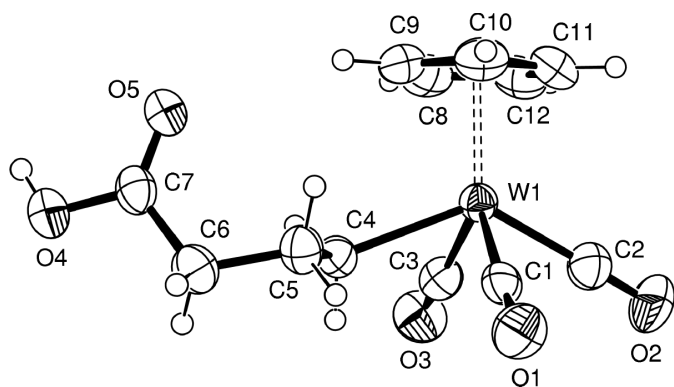


The bond lengths and angles given in Table 1 are unremarkable. The same is true for the  $\text{W}-\text{C}_{\text{Cp}}$  bonds in the range 2.312 (4)–2.376 (4) Å and the  $\text{C}-\text{C}$  bonds and  $\text{C}-\text{C}-\text{C}$  angles of Cp in the ranges 1.385 (7)–1.431 (7) Å and 107.3 (5)–108.4 (5)°, respectively. In the drawing of the unit-cell contents (Fig. 2) a notable feature is the presence of the hydrogen-bonded dimer involving a pair of centrosymmetrically related molecules. The hydrogen-bond parameters are given in Table 2. No other intermolecular contacts of any significance, other than van der Waals interactions, are present in the structure. Similar hydrogen-bonded dimers are present in the structures of the analogous compounds  $[\text{Cp}(\text{CO})_3\text{MoCH}_2\text{COOH}]$  and  $[\text{Cp}(\text{CO})_2\text{FeCH}_2\text{COOH}]$  (Ariyaratne *et al.*, 1969). Despite the limited quality of the refinements, these authors suggested that there was evidence to support some form of interaction between the metal (Mo or Fe) and the carboxylic acid group. There is no evidence for such an interaction in (I), which would be less likely in any case because of the length of the alkyl chain. There is perhaps a case for redetermining the earlier structures.

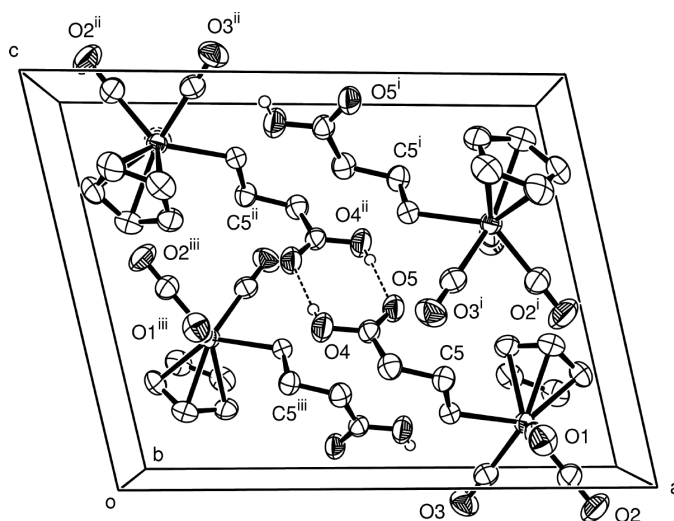
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**Figure 1**  
The molecule of (I). Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms as small circles of arbitrary radii. The dashed bond joins W and the centroid of the Cp ring.



**Figure 2**  
The unit-cell contents of (I). Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms as small circles of arbitrary radii. All H atoms other than those involved in hydrogen bonding (dashed lines) have been omitted. Selected atoms are labelled. [Symmetry codes: (i)  $x, \frac{1}{2} - y, \frac{1}{2} + z$ ; (ii)  $1 - x, 1 - y, 1 - z$ ; (iii)  $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$ .]

### Experimental

Compound (I) was obtained by hydrolysis brought about by the presence of water in a dichloromethane/hexane solution of  $[\text{Cp}(\text{CO})_3\text{W}(\text{CH}_2)_3\text{C}(\text{O})\text{Mo}(\text{CO})(\text{PMe}_3)(\text{PPh}_3)\text{Cp}]$  (Onani, 2002). Yellow crystals suitable for analysis were obtained after 5 d of slow diffusion of hexane into a dichloromethane solution of (I) kept at 278 K.

#### Crystal data

$[\text{W}(\text{C}_5\text{H}_5)(\text{C}_4\text{H}_7\text{O}_2)(\text{CO})_3]$   
 $M_r = 420.07$   
 Monoclinic,  $P2_1/c$   
 $a = 14.265(2) \text{ \AA}$   
 $b = 8.1540(11) \text{ \AA}$   
 $c = 11.229(2) \text{ \AA}$   
 $\beta = 102.800(15)^\circ$   
 $V = 1273.7(3) \text{ \AA}^3$   
 $Z = 4$

$D_x = 2.191 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 25 reflections  
 $\theta = 11.8\text{--}12.2^\circ$   
 $\mu = 9.08 \text{ mm}^{-1}$   
 $T = 295(2) \text{ K}$   
 Rhomb, yellow  
 $0.40 \times 0.30 \times 0.30 \text{ mm}$

#### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega$ - $2\theta$  scans  
 Absorption correction: refined from  $\Delta F$  (DIFABS; Walker & Stuart, 1983)  
 $T_{\min} = 0.011, T_{\max} = 0.066$   
 5154 measured reflections  
 2241 independent reflections

2053 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.035$   
 $\theta_{\max} = 25.0^\circ$   
 $h = -16 \rightarrow 16$   
 $k = -1 \rightarrow 9$   
 $l = -13 \rightarrow 13$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: 7%

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.072$   
 $S = 1.08$   
 2241 reflections  
 164 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.7777P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.00 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -1.17 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters ( $\text{\AA}, ^\circ$ ).

W1–Cg1	2.012 (2)	O3–C3	1.135 (5)
W1–C1	1.975 (5)	O4–C7	1.305 (5)
W1–C2	1.986 (5)	O5–C7	1.211 (5)
W1–C3	1.974 (5)	C4–C5	1.509 (6)
W1–C4	2.319 (4)	C5–C6	1.535 (5)
O1–C1	1.147 (6)	C6–C7	1.494 (6)
O2–C2	1.143 (6)		
Cg1–W1–C1	129.02 (15)	C1–W1–C3	106.84 (18)
Cg1–W1–C2	118.89 (18)	C1–W1–C4	75.15 (16)
Cg1–W1–C3	123.62 (16)	C2–W1–C3	78.1 (2)
Cg1–W1–C4	110.12 (13)	C2–W1–C4	130.95 (19)
C1–W1–C2	75.58 (19)	C3–W1–C4	73.91 (17)
C1–W1–C4–C5	50.8 (3)	W1–C4–C5–C6	167.4 (3)
C2–W1–C4–C5	106.0 (4)	C4–C5–C6–O4	−67.5 (6)
C3–W1–C4–C5	163.7 (4)	C4–C5–C6–O5	−82.2 (3)

Note: Cg1 is the centroid of the cyclopentadienyl ring.

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}, ^\circ$ ).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O4–H4 $\cdots$ O5 <sup>i</sup>	0.82	1.84	2.653 (5)	169

Symmetry code: (i)  $1 - x, 1 - y, 1 - z$ .

Presented here is a re-refinement, after suitable transformation of the unit-cell parameters and the atomic coordinates and reindexing of the intensity data, of a structure previously solved and fully refined in the space group  $P\bar{1}$ . The need for the re-refinement was clearly indicated by a *checkCIF* level A alert and the form it should take was revealed by recourse to the *ADDSYM* routine of *PLATON* (Spek, 2003). Close examination of a drawing of the content of the original, supposedly triclinic, unit cell fully confirmed the *ADDSYM* findings. Further support for the re-refinement reported here is the improvement in  $R [I > 2\sigma(I)]$  from 0.031 for the triclinic model to the value of 0.026 for the present refinement (with the number of refined parameters now half that of the triclinic refinement). In the final stages of the present refinement, H atoms were placed in calculated positions, with  $X\text{--}H = 0.82, 0.93$  and  $0.97 \text{ \AA}$  for hydroxyl, cyclopentadienyl and methylene H atoms, respectively, and refined using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$  or  $1.2U_{\text{eq}}(\text{C})$ , as appropriate for the nature of  $X$ . The position of the hydroxyl group in terms of its rotation about

the C—O bond was also refined. The highest residual electron-density peak is 0.99 Å from atom W1. The deepest residual electron-density hole lies 0.69 Å from atom W1.

Data collection: *CAD-4/PC* (Enraf–Nonius, 1994); cell refinement: *CAD-4/PC*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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## supporting information

*Acta Cryst.* (2004). E60, m1641–m1643 [https://doi.org/10.1107/S1600536804025371]

## Tricarbonyl(3-carboxypropyl)( $\eta^5$ -cyclopentadienyl)tungsten(II)

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### Tricarbonyl(3-carboxypropyl)( $\eta^5$ -cyclopentadienyl)tungsten(II)

#### Crystal data

[W(C<sub>3</sub>H<sub>5</sub>)(C<sub>4</sub>H<sub>7</sub>O<sub>2</sub>)(CO)<sub>3</sub>]

$M_r = 420.07$

Monoclinic,  $P2_1/c$

Hall symbol: P -2ybc

$a = 14.265$  (2) Å

$b = 8.1540$  (11) Å

$c = 11.229$  (2) Å

$\beta = 102.800$  (15)°

$V = 1273.7$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 792$

$D_x = 2.191$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 25 reflections

$\theta = 11.8$ – $12.2$ °

$\mu = 9.08$  mm<sup>-1</sup>

$T = 295$  K

Rhomb, yellow

$0.40 \times 0.30 \times 0.30$  mm

#### Data collection

Enraf–Nonius CAD-4  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$ – $2\theta$  scans

Absorption correction: part of the refinement  
model ( $\Delta F$ )

(DIFABS; Walker & Stuart, 1983)

$T_{\min} = 0.011$ ,  $T_{\max} = 0.066$

5154 measured reflections

2241 independent reflections

2053 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 25.0$ °,  $\theta_{\min} = 2.9$ °

$h = -16 \rightarrow 16$

$k = -1 \rightarrow 9$

$l = -13 \rightarrow 13$

3 standard reflections every 120 min

intensity decay: 7%

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.072$

$S = 1.08$

2241 reflections

164 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.7777P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.00$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -1.17$  e Å<sup>-3</sup>

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes. Distance (1 entry) and angles (4 entries) involving Cg1 (the centroid of the cyclopentadienyl ring) have been entered by hand into the relevant sections of the cif for ease of reference.

Least-squares planes ( $x, y, z$  in crystal coordinates) and deviations from them (\* indicates atom used to define plane)  
 4.4564 (0.0323)  $x$  + 4.7000 (0.0174)  $y$  + 7.4911 (0.0205)  $z$  = 8.9230 (0.0227)

\* -0.0042 (0.0029) C8 \* 0.0026 (0.0030) C9 \* 0.0001 (0.0031) C10 \* -0.0027 (0.0032) C11 \* 0.0043 (0.0030) C12 - 2.0105 (0.0021) W1 - 3.2724 (0.0062) C1 - 2.9105 (0.0073) C2 - 3.0909 (0.0066) C3

Rms deviation of fitted atoms = 0.0032

2.8996 (0.0447)  $x$  + 4.0968 (0.0144)  $y$  + 8.6965 (0.0214)  $z$  = 4.3963 (0.0356)

Angle to previous plane (with approximate e.s.d.) = 9.01 (0.38)

\* 0.0000 (0.0000) C1 \* 0.0000 (0.0000) C2 \* 0.0000 (0.0000) C3 1.1472 (0.0028) W1 3.1471 (0.0060) C8 3.3253 (0.0063) C9 3.2498 (0.0064) C10 3.0270 (0.0066) C11 2.9704 (0.0066) C12 - 0.6372 (0.0067) O1 - 0.6798 (0.0093) O2 - 0.6125 (0.0071) O3

Rms deviation of fitted atoms = 0.0000

3.1651 (0.0224)  $x$  + 4.0974 (0.0075)  $y$  + 8.5980 (0.0115)  $z$  = 3.9634 (0.0183)

Angle to previous plane (with approximate e.s.d.) = 1.10 (0.31)

\* 0.0000 (0.0000) O1 \* 0.0000 (0.0000) O2 \* 0.0000 (0.0000) O3

Rms deviation of fitted atoms = 0.0000

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

H atoms placed in calculated positions with  $X-H$  0.82, 0.93 and 0.97 Å for hydroxyl, cyclopentadienyl and methylene H, respectively, and refined with a riding model with  $U_{iso}(H) = 1.5U_{eq}(O)$  or  $1.2U_{eq}(C)$  as appropriate for the nature of  $X$ . The position of the hydroxyl group in terms of its rotation about the C—O bond was also refined.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{iso}^*/U_{eq}$
W1	0.800314 (12)	0.48662 (2)	0.141360 (15)	0.03491 (11)
O1	0.8107 (2)	0.1070 (5)	0.1116 (3)	0.0607 (9)
O2	0.9002 (3)	0.4342 (6)	-0.0773 (4)	0.0759 (11)
O3	0.6469 (3)	0.6263 (5)	-0.0757 (3)	0.0655 (9)
O4	0.4419 (2)	0.3506 (5)	0.3892 (3)	0.0621 (9)
H4	0.4325	0.4221	0.4365	0.093*
O5	0.5900 (3)	0.4493 (5)	0.4377 (3)	0.0567 (8)
C1	0.8043 (3)	0.2463 (6)	0.1213 (4)	0.0394 (9)
C2	0.8645 (4)	0.4546 (7)	0.0031 (5)	0.0523 (12)
C3	0.7026 (3)	0.5712 (6)	0.0022 (4)	0.0465 (10)
C4	0.6532 (3)	0.3944 (5)	0.1705 (4)	0.0408 (9)
H4A	0.6202	0.4871	0.1966	0.049*
H4B	0.6154	0.3596	0.0918	0.049*
C5	0.6527 (3)	0.2565 (5)	0.2599 (4)	0.0439 (10)
H5A	0.7003	0.2791	0.3343	0.053*
H5B	0.6710	0.1555	0.2254	0.053*
C6	0.5543 (3)	0.2328 (5)	0.2916 (4)	0.0471 (10)
H6A	0.5050	0.2374	0.2167	0.057*

H6B	0.5522	0.1243	0.3263	0.057*
C7	0.5312 (3)	0.3564 (5)	0.3790 (4)	0.0425 (9)
C8	0.8027 (4)	0.7247 (6)	0.2584 (4)	0.0564 (12)
H8	0.7539	0.8020	0.2378	0.068*
C9	0.8052 (3)	0.5954 (6)	0.3390 (4)	0.0512 (11)
H9	0.7585	0.5715	0.3825	0.061*
C10	0.8902 (5)	0.5065 (5)	0.3438 (6)	0.0542 (15)
H10	0.9095	0.4135	0.3907	0.065*
C11	0.9411 (3)	0.5829 (7)	0.2652 (5)	0.0563 (12)
H11	0.9998	0.5488	0.2510	0.068*
C12	0.8885 (4)	0.7185 (6)	0.2124 (5)	0.0584 (12)
H12	0.9059	0.7914	0.1574	0.070*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
W1	0.03475 (17)	0.03509 (14)	0.03487 (16)	0.00048 (5)	0.00766 (11)	-0.00016 (5)
O1	0.063 (2)	0.047 (2)	0.072 (2)	0.0105 (16)	0.0141 (17)	-0.0033 (17)
O2	0.078 (3)	0.096 (3)	0.066 (2)	-0.004 (3)	0.044 (2)	-0.008 (2)
O3	0.064 (2)	0.071 (2)	0.055 (2)	0.0085 (19)	-0.0007 (17)	0.0166 (18)
O4	0.0410 (18)	0.081 (3)	0.068 (2)	-0.0133 (17)	0.0219 (16)	-0.0192 (19)
O5	0.0394 (19)	0.072 (2)	0.060 (2)	-0.0114 (18)	0.0157 (17)	-0.016 (2)
C1	0.031 (2)	0.040 (3)	0.046 (2)	0.0026 (16)	0.0054 (17)	-0.0030 (17)
C2	0.052 (3)	0.054 (3)	0.053 (3)	-0.003 (2)	0.017 (3)	-0.001 (2)
C3	0.053 (3)	0.043 (2)	0.045 (2)	0.000 (2)	0.014 (2)	0.001 (2)
C4	0.035 (2)	0.049 (2)	0.038 (2)	-0.0004 (18)	0.0072 (17)	0.0001 (18)
C5	0.042 (2)	0.041 (2)	0.050 (2)	0.0016 (18)	0.014 (2)	0.0023 (19)
C6	0.045 (2)	0.043 (2)	0.053 (2)	-0.0043 (19)	0.012 (2)	0.0041 (19)
C7	0.038 (2)	0.048 (2)	0.042 (2)	-0.0002 (19)	0.0117 (18)	0.0103 (19)
C8	0.063 (3)	0.041 (2)	0.061 (3)	0.001 (2)	0.004 (2)	-0.012 (2)
C9	0.054 (3)	0.057 (3)	0.043 (2)	-0.007 (2)	0.011 (2)	-0.014 (2)
C10	0.059 (4)	0.057 (3)	0.042 (3)	-0.001 (2)	0.001 (3)	-0.0020 (18)
C11	0.039 (2)	0.070 (3)	0.056 (3)	-0.010 (2)	0.000 (2)	-0.008 (3)
C12	0.062 (3)	0.046 (3)	0.064 (3)	-0.022 (2)	0.006 (2)	-0.004 (2)

*Geometric parameters (Å, °)*

W1—Cg1	2.012 (2)	C4—H4B	0.9700
W1—C1	1.975 (5)	C5—C6	1.535 (5)
W1—C2	1.986 (5)	C5—H5A	0.9700
W1—C3	1.974 (5)	C5—H5B	0.9700
W1—C11	2.312 (4)	C6—C7	1.494 (6)
W1—C12	2.313 (4)	C6—H6A	0.9700
W1—C4	2.319 (4)	C6—H6B	0.9700
W1—C8	2.340 (4)	C8—C9	1.385 (7)
W1—C10	2.355 (6)	C8—C12	1.431 (7)
W1—C9	2.376 (4)	C8—H8	0.9300
O1—C1	1.147 (6)	C9—C10	1.403 (8)

O2—C2	1.143 (6)	C9—H9	0.9300
O3—C3	1.135 (5)	C10—C11	1.406 (8)
O4—C7	1.305 (5)	C10—H10	0.9300
O4—H4	0.8200	C11—C12	1.392 (7)
O5—C7	1.211 (5)	C11—H11	0.9300
C4—C5	1.509 (6)	C12—H12	0.9300
C4—H4A	0.9700		
Cg1—W1—C1	129.02 (15)	W1—C4—H4B	107.7
Cg1—W1—C2	118.89 (18)	H4A—C4—H4B	107.1
Cg1—W1—C3	123.62 (16)	C4—C5—C6	112.8 (3)
Cg1—W1—C4	110.12 (13)	C4—C5—H5A	109.0
C1—W1—C2	75.58 (19)	C6—C5—H5A	109.0
C1—W1—C3	106.84 (18)	C4—C5—H5B	109.0
C1—W1—C4	75.15 (16)	C6—C5—H5B	109.0
C2—W1—C3	78.1 (2)	H5A—C5—H5B	107.8
C2—W1—C4	130.95 (19)	C7—C6—C5	114.5 (4)
C3—W1—C4	73.91 (17)	C7—C6—H6A	108.6
C3—W1—C11	136.4 (2)	C5—C6—H6A	108.6
C1—W1—C11	111.26 (19)	C7—C6—H6B	108.6
C2—W1—C11	91.5 (2)	C5—C6—H6B	108.6
C3—W1—C12	102.8 (2)	H6A—C6—H6B	107.6
C1—W1—C12	145.43 (17)	O5—C7—O4	123.2 (4)
C2—W1—C12	93.8 (2)	O5—C7—C6	123.5 (4)
C11—W1—C12	35.04 (19)	O4—C7—C6	113.3 (4)
C11—W1—C4	135.87 (17)	C9—C8—C12	108.0 (5)
C12—W1—C4	131.05 (18)	C9—C8—W1	74.4 (3)
C3—W1—C8	94.28 (18)	C12—C8—W1	71.1 (3)
C1—W1—C8	152.86 (17)	C9—C8—H8	126.0
C2—W1—C8	126.6 (2)	C12—C8—H8	126.0
C11—W1—C8	58.51 (19)	W1—C8—H8	120.4
C12—W1—C8	35.81 (18)	C8—C9—C10	108.3 (5)
C4—W1—C8	95.24 (17)	C8—C9—W1	71.5 (3)
C3—W1—C10	151.43 (19)	C10—C9—W1	71.9 (3)
C1—W1—C10	98.92 (16)	C8—C9—H9	125.8
C2—W1—C10	121.1 (2)	C10—C9—H9	125.8
C11—W1—C10	35.1 (2)	W1—C9—H9	122.4
C12—W1—C10	58.17 (18)	C9—C10—C11	108.0 (5)
C4—W1—C10	101.69 (19)	C9—C10—W1	73.6 (3)
C8—W1—C10	57.57 (17)	C11—C10—W1	70.8 (3)
C3—W1—C9	118.59 (18)	C9—C10—H10	126.0
C1—W1—C9	118.74 (17)	C11—C10—H10	126.0
C2—W1—C9	148.82 (19)	W1—C10—H10	121.3
C11—W1—C9	57.98 (17)	C12—C11—C10	108.4 (5)
C12—W1—C9	58.12 (18)	C12—C11—W1	72.5 (3)
C4—W1—C9	80.20 (16)	C10—C11—W1	74.1 (3)
C8—W1—C9	34.14 (17)	C12—C11—H11	125.8
C10—W1—C9	34.51 (19)	C10—C11—H11	125.8

C7—O4—H4	109.5	W1—C11—H11	119.4
O1—C1—W1	177.1 (4)	C11—C12—C8	107.3 (5)
O2—C2—W1	178.8 (5)	C11—C12—W1	72.5 (3)
O3—C3—W1	177.1 (4)	C8—C12—W1	73.1 (3)
C5—C4—W1	118.3 (3)	C11—C12—H12	126.4
C5—C4—H4A	107.7	C8—C12—H12	126.4
W1—C4—H4A	107.7	W1—C12—H12	119.9
C5—C4—H4B	107.7		
C1—W1—C4—C5	50.8 (3)	W1—C4—C5—C6	167.4 (3)
C2—W1—C4—C5	106.0 (4)	C4—C5—C6—O4	-67.5 (6)
C3—W1—C4—C5	163.7 (4)	C4—C5—C6—O5	-82.2 (3)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O4—H4 $\cdots$ O5 <sup>i</sup>	0.82	1.84	2.653 (5)	169

Symmetry code: (i)  $-x+1, -y+1, -z+1$ .