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## Structure Reports

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# Poly[( $\mu_2$ -2-hydroxy-2-methylpropionato- $\kappa^3 O^1, O^2:O^1'$ )( $\mu_2$ -2-hydroxy-2-methylpropionato- $\kappa^2 O^1:\kappa O^1'$ )dioxido-uranium(VI)]

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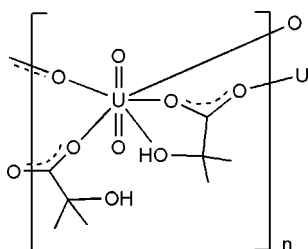
Received 15 February 2009; accepted 19 February 2009

 Key indicators: single-crystal X-ray study;  $T = 200$  K; mean  $\sigma(C-C) = 0.010$  Å;  $R$  factor = 0.029;  $wR$  factor = 0.100; data-to-parameter ratio = 18.4.

In the title compound,  $[UO_2(C_4H_7O_3)_2]_n$ , the dioxouranium(VI) units are linked by 2-hydroxy-2-methylpropionate ligands into a honeycomb structure. The U atom is seven-coordinate in a pentagonal-bipyramidal geometry. The uncoordinated hydroxy groups of the 2-hydroxy-2-methylpropionate ions interact with the O atom of the uranyl and with the coordinated hydroxy group of an adjacent 2-hydroxy-2-methylpropionate ion through O—H...O hydrogen bonds.

## Related literature

For related structures, see: Back *et al.* (2007); Bombieri *et al.* (1973, 1974); Jiang *et al.* (2002); Thuéry (2006, 2007a,b,c, 2008); Xie *et al.* (2003); Yokoyama *et al.* (1990).



## Experimental

## Crystal data

 $[U(C_4H_7O_3)_2O_2]$ 
 $M_r = 476.22$ 

 Monoclinic,  $P2_1/n$ 
 $a = 9.009$  (2) Å

 $b = 8.237$  (2) Å

 $c = 17.552$  (6) Å

 $\beta = 98.246$  (9)°

 $V = 1289.0$  (6) Å<sup>3</sup>
 $Z = 4$ 

 Mo  $K\alpha$  radiation

 $\mu = 12.62$  mm<sup>-1</sup>
 $T = 200$  K

 $0.20 \times 0.11 \times 0.03$  mm

## Data collection

Rigaku R-Axis RAPID Imaging

Plate diffractometer

Absorption correction: multi-scan

(ABSCOR; Higashi, 1995)

 $T_{\min} = 0.233$ ,  $T_{\max} = 0.685$ 

11887 measured reflections

2949 independent reflections

 2547 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.050$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.029$ 
 $wR(F^2) = 0.100$ 
 $S = 0.86$ 

2949 reflections

160 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.99$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -2.15$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

U1—O1	1.783 (5)	U1—O5 <sup>i</sup>	2.355 (4)
U1—O2	1.762 (6)	U1—O7	2.346 (5)
U1—O3	2.444 (5)	U1—O7	2.336 (5)
U1—O4	2.407 (5)		

 Symmetry code: (i)  $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$ .

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H13...O6	0.82	1.93	2.597 (6)	138
O6—H14...O1 <sup>ii</sup>	0.82	2.00	2.777 (6)	158

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Rigaku/MS, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *TEXSAN*.

The present study is the result of the efficient separation and analysis of nuclear fission products for reprocessing systems entrusted to Osaka University by the Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2546).

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**supplementary materials**

*Acta Cryst.* (2009). E65, m355-m356 [ doi:10.1107/S1600536809006059 ]

**Poly[( $\mu_2$ -2-hydroxy-2-methylpropionato- $\kappa^3 O^1, O^2:O^{1'}$ )( $\mu_2$ -2-hydroxy-2-methylpropionato- $\kappa^2 O^1:\kappa O^{1'}$ )dioxidouranium(VI)]**

**T. Yoshimura, H. Kikunaga and A. Shinohara**

**Comment**

Structural chemistry of uranyl(VI) complexes with hydroxycarboxylate or alkoxycarboxylate has been extensively studied (Back *et al.* (2007); Bombieri *et al.* (1973, 1974); Jiang *et al.* (2002); Thuéry (2006, 2007*a,b,c*, 2008); Xie *et al.* (2003); Yokoyama *et al.* (1990)). The crystals of the title compound (**I**) suitable for single-crystal X-ray analysis were obtained by the reaction of bis(acetato)dioxouranium dihydrate with an excess amount of 2-hydroxy-2-methylpropionic acid in water. Herein, we report on the crystal structure of **I**. Uranium(VI) atom is seven-coordinate in a pentagonal-bipyramidal structure. The two oxygen atoms are located at the axial positions with nearly linear O(1)—U(1)—O(2) angle (178.3 (2)°). The equatorial positions are coordinated by five oxygen atoms of 2-hydroxy-2-methylpropionate (HIB) ligands. Two kinds of HIB ligands exist in the asymmetric unit. One of the HIB ligands links two uranium atoms by the carboxyl group. The other chelates one uranium atom through the hydroxy and carboxyl groups, moreover the carboxyl group bridges the neighboring uranium atom. As a result, a two-dimensional honeycomb structure is formed. The IR spectrum of **I** shows stretching bands of the carboxyl group of HIB at 1614 and 1561  $\text{cm}^{-1}$ .

**Experimental**

2-Hydroxy-2-methylpropionic acid (150 mg, 1.45 mmol) was added to a solution of bis(acetato)dioxouranium dihydrate (50 mg, 0.12 mmol) in 3 ml of water. The resulting yellow solution was left for several days at room temperature to give yellow crystals, which were filtered off, washed with a small amount of water, and then dried in air.

**Refinement**

H atoms bonded to C and O atoms were placed at calculated positions [C—H = 0.96 and O—H = 0.82] and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.0 U_{\text{eq}}(\text{C}, \text{O})$ . The deepest hole is 0.68 Å from atom U(1).

**Figures**

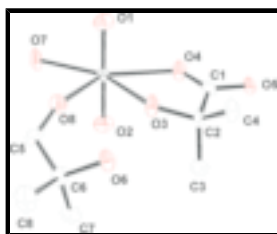


Fig. 1. The asymmetric unit of **I**, with the atom-numbering scheme and displacement ellipsoids drawn at 50% probability level. Hydrogen atoms are omitted for clarity.

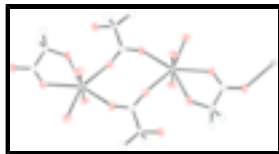


Fig. 2. Fragment of the polymeric structure. Hydrogen atoms are omitted for clarity.

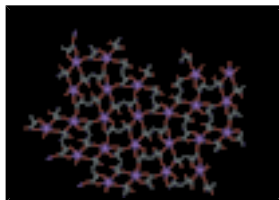


Fig. 3. View of the polymeric structure. Hydrogen atoms are omitted for clarity.

**Poly[( $\mu_2$ -2-hydroxy-2-methylpropionato- $\kappa^3 O^1, O^2: O^{1'}$ )( $\mu_2$ -2-hydroxy-2-methylpropionato- $\kappa^2 O^1: \kappa O^{1'}$ )dioxidouranium(VI)]**

*Crystal data*

[U(C<sub>4</sub>H<sub>7</sub>O<sub>3</sub>)<sub>2</sub>O<sub>2</sub>]

$M_r = 476.22$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 9.009 (2) \text{ \AA}$

$b = 8.237 (2) \text{ \AA}$

$c = 17.552 (6) \text{ \AA}$

$\beta = 98.246 (9)^\circ$

$V = 1289.0 (6) \text{ \AA}^3$

$Z = 4$

$F_{000} = 872.00$

$D_x = 2.454 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.7107 \text{ \AA}$

Cell parameters from 8716 reflections

$\theta = 3.0\text{--}27.5^\circ$

$\mu = 12.62 \text{ mm}^{-1}$

$T = 200 \text{ K}$

Platelet, yellow

$0.20 \times 0.11 \times 0.03 \text{ mm}$

*Data collection*

Rigaku R-Axis RAPID Imaging Plate diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution:  $10.00 \text{ pixels mm}^{-1}$

$T = 200 \text{ K}$

$\omega$  scans

Absorption correction: multi-scan (ABSCOR; Higashi, 1995)

$T_{\min} = 0.233$ ,  $T_{\max} = 0.685$

11887 measured reflections

2949 independent reflections

2547 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\max} = 27.5^\circ$

$\theta_{\min} = 3.0^\circ$

$h = 12 \rightarrow 11$

$k = -9 \rightarrow 10$

$l = -22 \rightarrow 22$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$$R[F^2 > 2\sigma(F^2)] = 0.029$$

$$wR(F^2) = 0.100$$

$$S = 0.86$$

2949 reflections

160 parameters

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.079P)^2 + 5.354P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.99 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -2.15 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

### 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.

**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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
U(1)	0.63772 (2)	0.19668 (3)	0.62425 (1)	0.0180 (1)
O(1)	0.7538 (6)	0.3101 (5)	0.5695 (3)	0.028 (1)
O(2)	0.5188 (6)	0.0891 (6)	0.6778 (3)	0.032 (1)
O(3)	0.4753 (5)	0.4342 (5)	0.6250 (3)	0.026 (1)
O(4)	0.6920 (5)	0.3869 (6)	0.7286 (3)	0.029 (1)
O(5)	0.6435 (5)	0.6018 (6)	0.7991 (3)	0.0252 (10)
O(6)	0.2338 (5)	0.4208 (5)	0.5248 (3)	0.0241 (10)
O(7)	0.7009 (6)	-0.0415 (6)	0.5630 (3)	0.034 (1)
O(8)	0.4514 (6)	0.1975 (5)	0.5169 (3)	0.030 (1)
C(1)	0.6115 (7)	0.5015 (8)	0.7435 (3)	0.022 (1)
C(2)	0.4611 (7)	0.5295 (8)	0.6929 (4)	0.021 (1)
C(3)	0.3351 (8)	0.463 (1)	0.7324 (4)	0.036 (2)
C(4)	0.4392 (10)	0.7064 (7)	0.6704 (5)	0.030 (2)
C(5)	0.3241 (7)	0.1601 (8)	0.4820 (4)	0.020 (1)
C(6)	0.1889 (7)	0.2626 (8)	0.4949 (4)	0.021 (1)
C(7)	0.1191 (10)	0.1790 (9)	0.5588 (5)	0.039 (2)
C(8)	0.0781 (10)	0.280 (1)	0.4211 (5)	0.038 (2)
H(1)	0.3277	0.5253	0.7779	0.0356*
H(2)	0.2423	0.4698	0.6980	0.0356*
H(3)	0.3555	0.3516	0.7462	0.0356*
H(4)	0.4388	0.7711	0.7159	0.0297*
H(5)	0.3454	0.7192	0.6374	0.0297*
H(6)	0.5196	0.7409	0.6438	0.0297*
H(7)	0.0321	0.2384	0.5684	0.0386*

## supplementary materials

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H(8)	0.1908	0.1757	0.6048	0.0386*
H(9)	0.0907	0.0703	0.5432	0.0386*
H(10)	-0.0100	0.3359	0.4322	0.0383*
H(11)	0.0507	0.1746	0.4007	0.0383*
H(12)	0.1237	0.3413	0.3841	0.0383*
H(13)	0.4210	0.4724	0.5878	0.0255*
H(14)	0.2178	0.4883	0.4902	0.0241*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
U(1)	0.0192 (2)	0.0158 (2)	0.0185 (2)	0.00083 (8)	0.0005 (1)	-0.00114 (8)
O(1)	0.022 (3)	0.030 (3)	0.032 (3)	0.007 (2)	0.004 (2)	0.007 (2)
O(2)	0.029 (3)	0.025 (2)	0.044 (3)	0.005 (2)	0.009 (2)	0.007 (2)
O(3)	0.034 (3)	0.022 (2)	0.017 (2)	0.008 (2)	-0.008 (2)	-0.005 (2)
O(4)	0.029 (3)	0.025 (2)	0.030 (2)	0.007 (2)	-0.004 (2)	-0.006 (2)
O(5)	0.028 (2)	0.027 (2)	0.020 (2)	-0.008 (2)	0.001 (2)	-0.010 (2)
O(6)	0.030 (2)	0.016 (2)	0.023 (2)	0.002 (2)	-0.005 (2)	-0.001 (2)
O(7)	0.032 (3)	0.027 (3)	0.038 (3)	0.009 (2)	-0.008 (2)	-0.018 (2)
O(8)	0.023 (3)	0.033 (3)	0.033 (3)	0.005 (2)	-0.002 (2)	-0.008 (2)
C(1)	0.023 (3)	0.024 (3)	0.017 (3)	-0.007 (3)	0.000 (2)	-0.001 (3)
C(2)	0.027 (3)	0.016 (3)	0.020 (3)	0.000 (3)	0.000 (2)	-0.008 (2)
C(3)	0.032 (4)	0.042 (4)	0.035 (4)	-0.012 (3)	0.009 (3)	-0.013 (3)
C(4)	0.040 (4)	0.015 (3)	0.032 (4)	0.004 (3)	-0.001 (3)	-0.004 (3)
C(5)	0.022 (3)	0.019 (3)	0.020 (3)	0.009 (3)	0.001 (2)	0.002 (3)
C(6)	0.021 (3)	0.018 (3)	0.022 (3)	0.013 (3)	-0.003 (2)	0.003 (3)
C(7)	0.035 (4)	0.036 (4)	0.048 (5)	-0.015 (3)	0.017 (4)	-0.005 (3)
C(8)	0.035 (4)	0.035 (4)	0.040 (4)	0.014 (3)	-0.012 (4)	-0.004 (4)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

U(1)—O(1)	1.783 (5)	C(2)—C(4)	1.514 (9)
U(1)—O(2)	1.762 (6)	C(3)—H(1)	0.960
U(1)—O(3)	2.444 (5)	C(3)—H(2)	0.960
U(1)—O(4)	2.407 (5)	C(3)—H(3)	0.960
U(1)—O(5) <sup>i</sup>	2.355 (4)	C(4)—H(4)	0.960
U(1)—O(7)	2.346 (5)	C(4)—H(5)	0.960
U(1)—O(8)	2.336 (5)	C(4)—H(6)	0.960
O(3)—C(2)	1.449 (8)	C(5)—C(6)	1.525 (10)
O(3)—H(13)	0.820	C(6)—C(7)	1.53 (1)
O(4)—C(1)	1.241 (8)	C(6)—C(8)	1.523 (10)
O(5)—C(1)	1.280 (8)	C(7)—H(7)	0.960
O(6)—C(6)	1.441 (8)	C(7)—H(8)	0.960
O(6)—H(14)	0.820	C(7)—H(9)	0.960
O(7)—C(5) <sup>ii</sup>	1.257 (8)	C(8)—H(10)	0.960
O(8)—C(5)	1.259 (8)	C(8)—H(11)	0.960
C(1)—C(2)	1.528 (8)	C(8)—H(12)	0.960
C(2)—C(3)	1.51 (1)		

O(1)—U(1)—O(2)	178.3 (2)	C(1)—C(2)—C(4)	111.6 (6)
O(1)—U(1)—O(3)	88.9 (2)	C(3)—C(2)—C(4)	112.9 (6)
O(1)—U(1)—O(4)	89.9 (2)	C(2)—C(3)—H(1)	109.5
O(1)—U(1)—O(5) <sup>i</sup>	88.5 (2)	C(2)—C(3)—H(2)	109.5
O(1)—U(1)—O(7)	89.5 (2)	C(2)—C(3)—H(3)	109.5
O(1)—U(1)—O(8)	88.5 (2)	H(1)—C(3)—H(2)	109.5
O(2)—U(1)—O(3)	89.4 (2)	H(1)—C(3)—H(3)	109.5
O(2)—U(1)—O(4)	89.7 (2)	H(2)—C(3)—H(3)	109.5
O(2)—U(1)—O(5) <sup>i</sup>	93.0 (2)	C(2)—C(4)—H(4)	109.5
O(2)—U(1)—O(7)	91.7 (2)	C(2)—C(4)—H(5)	109.5
O(2)—U(1)—O(8)	90.6 (2)	C(2)—C(4)—H(6)	109.5
O(3)—U(1)—O(4)	62.1 (1)	H(4)—C(4)—H(5)	109.5
O(3)—U(1)—O(5) <sup>i</sup>	135.7 (2)	H(4)—C(4)—H(6)	109.5
O(3)—U(1)—O(7)	148.9 (2)	H(5)—C(4)—H(6)	109.5
O(3)—U(1)—O(8)	68.9 (2)	O(7) <sup>ii</sup> —C(5)—O(8)	124.4 (6)
O(4)—U(1)—O(5) <sup>i</sup>	73.7 (2)	O(7) <sup>ii</sup> —C(5)—C(6)	116.7 (6)
O(4)—U(1)—O(7)	149.0 (2)	O(8)—C(5)—C(6)	119.0 (6)
O(4)—U(1)—O(8)	131.0 (2)	O(6)—C(6)—C(5)	111.4 (5)
O(5) <sup>i</sup> —U(1)—O(7)	75.3 (2)	O(6)—C(6)—C(7)	105.3 (5)
O(5) <sup>i</sup> —U(1)—O(8)	155.1 (2)	O(6)—C(6)—C(8)	109.8 (6)
O(7)—U(1)—O(8)	80.0 (2)	C(5)—C(6)—C(7)	106.3 (6)
U(1)—O(3)—C(2)	124.2 (3)	C(5)—C(6)—C(8)	111.5 (6)
U(1)—O(3)—H(13)	126.3	C(7)—C(6)—C(8)	112.2 (6)
C(2)—O(3)—H(13)	109.5	C(6)—C(7)—H(7)	109.5
U(1)—O(4)—C(1)	126.5 (4)	C(6)—C(7)—H(8)	109.5
U(1) <sup>iii</sup> —O(5)—C(1)	136.7 (4)	C(6)—C(7)—H(9)	109.5
C(6)—O(6)—H(14)	109.5	H(7)—C(7)—H(8)	109.5
U(1)—O(7)—C(5) <sup>ii</sup>	154.9 (4)	H(7)—C(7)—H(9)	109.5
U(1)—O(8)—C(5)	153.0 (5)	H(8)—C(7)—H(9)	109.5
O(4)—C(1)—O(5)	125.3 (6)	C(6)—C(8)—H(10)	109.5
O(4)—C(1)—C(2)	119.3 (5)	C(6)—C(8)—H(11)	109.5
O(5)—C(1)—C(2)	115.4 (6)	C(6)—C(8)—H(12)	109.5
O(3)—C(2)—C(1)	102.7 (5)	H(10)—C(8)—H(11)	109.5
O(3)—C(2)—C(3)	109.9 (5)	H(10)—C(8)—H(12)	109.5
O(3)—C(2)—C(4)	109.3 (5)	H(11)—C(8)—H(12)	109.5
C(1)—C(2)—C(3)	109.9 (5)		

Symmetry codes: (i)  $-x+3/2, y-1/2, -z+3/2$ ; (ii)  $-x+1, -y, -z+1$ ; (iii)  $-x+3/2, y+1/2, -z+3/2$ .

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

<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>
O(3)—H(13) $\cdots$ O(6)	0.820	1.927	2.597 (6)	138.188
O(6)—H(14) $\cdots$ O(1) <sup>iv</sup>	0.820	1.999	2.777 (6)	158.201

Symmetry codes: (iv)  $-x+1, -y+1, -z+1$ .

Fig. 1

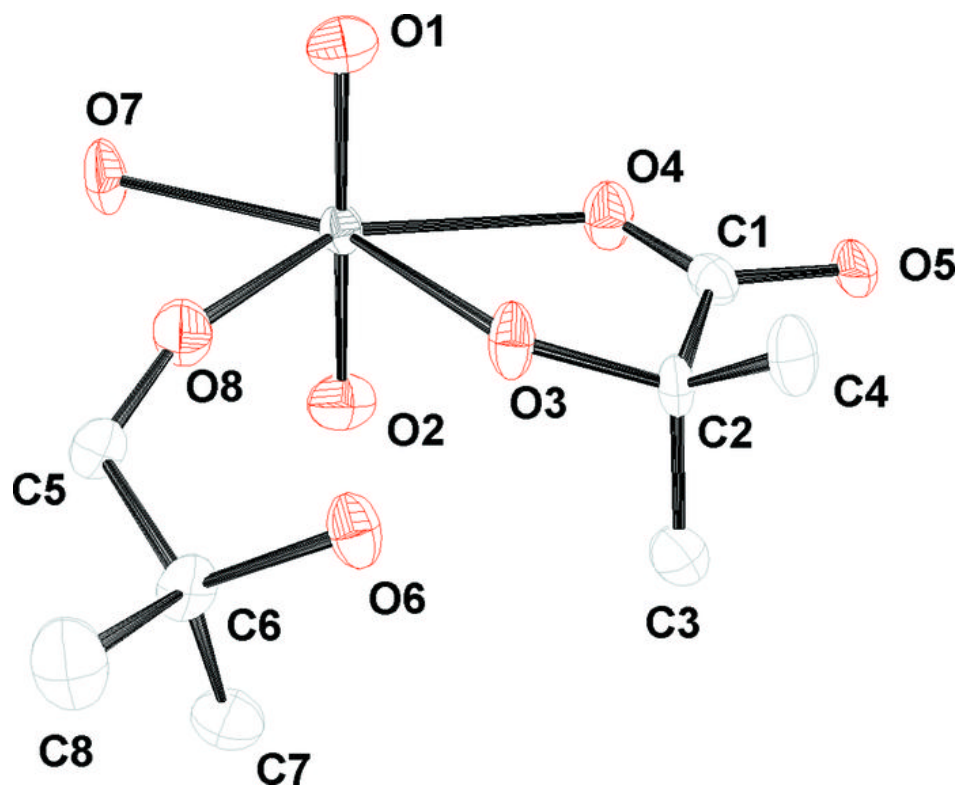


Fig. 2

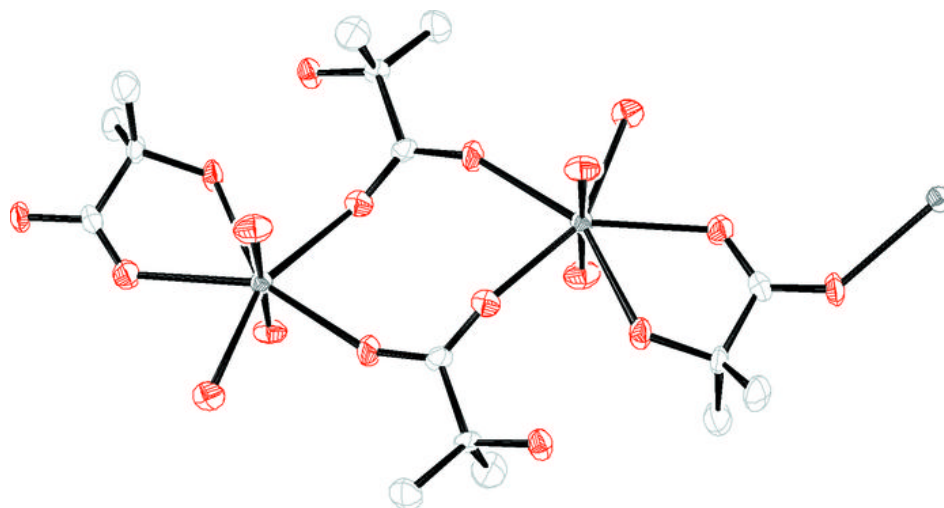


Fig. 3

