

Monoclinic  
 $P2_1/n$   
 $a = 9.5279$  (12) Å  
 $b = 18.229$  (3) Å  
 $c = 12.7795$  (13) Å  
 $\beta = 107.313$  (9)°  
 $V = 2119.1$  (5) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.483$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Cell parameters from 25 reflections  
 $\theta = 10.0\text{--}15.0^\circ$   
 $\mu = 1.322$  mm<sup>-1</sup>  
 $T = 170$  (1) K  
 Prism  
 $0.30 \times 0.30 \times 0.16$  mm  
 Blue

#### Data collection

AFC-6S diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  
 $\psi$  scan (3 reflections)  
 (North *et al.*, 1968)  
 $T_{\min} = 0.63$ ,  $T_{\max} = 0.81$   
 5172 measured reflections  
 4892 independent reflections  
 1836 reflections with  
 $I > 2\sigma(I)$

$R_{\text{int}} = 0.061$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = 0 \rightarrow 12$   
 $k = 0 \rightarrow 23$   
 $l = -16 \rightarrow 15$   
 3 standard reflections  
 every 200 reflections  
 intensity decay: 0.43%

#### Refinement

Refinement on  $F^2$   
 $R(F) = 0.048$   
 $wR(F^2) = 0.167$   
 $S = 0.953$   
 4892 reflections  
 227 parameters  
 H atoms geometrically  
 idealized with C—H and  
 N—H = 0.95 Å

$w = 1/[\sigma^2(F_o^2) + (0.066P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.685$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.574$  e Å<sup>-3</sup>  
 Extinction correction: none  
 Scattering factors from  
*International Tables for*  
*Crystallography* (Vol. C)

Table 3. Selected geometric parameters (Å, °) for (2)

Co1—C11	2.2806 (19)	N1—C1	1.356 (8)
Co1—C12	2.2916 (18)	N1—C5	1.343 (8)
Co1—C13	2.2994 (16)	N2—C15	1.494 (7)
Co1—C14	2.2538 (18)	N2—C16	1.486 (7)
O1—C14	1.423 (7)	N2—C17	1.472 (8)
O1—C6	1.453 (7)		
C14—O1—C6	116.4 (4)	C17—N2—C15	111.4 (5)
C5—N1—C1	122.7 (6)	C17—N2—C16	110.8 (5)
C16—N2—C15	112.0 (5)		

Table 4. Hydrogen-bonding geometry (Å, °) for (2)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...Cl3	0.95	2.40	3.259 (7)	151
N2—H2...Cl2 <sup>i</sup>	0.95	2.43	3.221 (6)	141
N1—H1...O1	0.95	2.09	2.533 (8)	107
N2—H2...O1	0.95	2.33	2.747 (8)	106

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

For both (1) and (2), the space group  $P2_1/n$  was determined uniquely from the systematic absences.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989) for (1); *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988) for (2). Cell refinement: *CAD-4 Software* for (1); *MSC/AFC Diffractometer Control Software* for (2). For both compounds, data reduction: *TEXSAN* (Molecular Structure Corporation, 1994); program(s) used to solve structures: *SAPI91* (Fan, 1991); program(s) used to refine structures: *SHELXL97* (Sheldrick, 1997); molecular graphics: *TEXSAN*; software used to prepare material for publication: *SHELXL97*.

The authors thank the Natural Sciences and Engineering Research Council, Canada, for providing the diffractometer through an equipment grant to the University of Calgary, and the University of Calgary, for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1429). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). **C54**, 935–937

### Chloro[2,3-dimethylbutane-2,3-diolato(2-)-O,O'] [2,3-dimethylbutane-2,3-diolato(1-)-O,O']oxotungsten(VI)

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(Received 2 June 1997; accepted 12 January 1998)

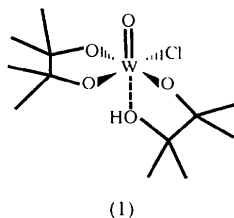
#### Abstract

The title compound,  $[\text{WOCl}(\text{C}_6\text{H}_{12}\text{O}_2)(\text{C}_6\text{H}_{13}\text{O}_2)]$ , is a neutral complex in which the central tungsten(VI) cation is bonded to one chloro, one oxo, one diolato

and one hydrogendiolato ligand resulting in a distorted octahedron around the W<sup>VI</sup> atom. Adjacent complex molecules are linked into dimers by O—H...Cl hydrogen bonds.

### Comment

Our studies of tungsten(VI)-diolato complexes have produced a new compound, chloro[2,3-dimethylbutane-2,3-diolato(2-)-O,O'] [2,3-dimethylbutane-2,3-diolato(1-)-O,O']oxotungsten(VI), (1), the crystal structure of which is reported here.



A dimeric structural unit of the complex is shown in Fig. 1. The tungsten(VI) ion is bonded to one chloro ligand, one terminal oxo ligand, one diolato dianion and one hydrogendiolato monoanion. Both the diolato and hydrogendiolato ligands have a bidentate coordination mode. The chloro and oxo ligands are in *cis* positions. The weakest oxygen donor, *i.e.* the hydroxyl oxygen of the hydrogendiolato ligand, is arranged *trans* with respect to the multiple-bonded oxo ligand, as expected from the strong *trans* influence of the oxo group (Nugent & Mayer, 1988). The molecular structure of (1) resembles that found for the tungsten(VI) catecholato (cat) complex [WOCl(cat)(Hcat)].Et<sub>2</sub>O (Persson *et al.*, 1992). The coordination sphere of the W<sup>VI</sup> ion in (1) is also approximately similar to the corresponding values

found in the tungsten(VI)-centred units of dimeric [{WO(diol)(Hdiol)}<sub>2</sub>O] complexes, where H<sub>2</sub>diol is 2,3-dimethylbutane-2,3-diol, 1,2-cycloheptanediol or 1,2-cyclooctanediol (Lehtonen & Sillanpää, 1994).

Hydrogen bonding through second-sphere coordination plays a role in the crystallization of complex (1). The hydroxyl proton of the hydrogendiolato ligand is hydrogen bonded to the chloro ligand of an adjacent molecule and (1) occurs in the solid state as a dimeric species. In comparable tungsten(VI) complexes with bidentate monoanionic ligands, *e.g.* in [WOCl(cat)(Hcat)].Et<sub>2</sub>O and [WOCl<sub>3</sub>(Hbino)].Et<sub>2</sub>O (H<sub>2</sub>bino is 3,3'-dimethyl-1,1'-bi-2-naphthol), the H atoms of the hydroxyl groups are bonded to the O atoms of diethyl ether molecules (Persson *et al.*, 1992; Morton *et al.*, 1993).

The O—H...Cl hydrogen-bond parameters are H1...Cl<sup>i</sup> 2.45 (4) and O4...Cl<sup>i</sup> 3.182 (3) Å, with an almost linear O4—H1...Cl<sup>i</sup> angle of 170 (5)<sup>o</sup> [symmetry code: (i) -x, -y, -z]. Due to the inversion centre, dimeric units are formed. Similar dimers are found also in [W(η<sup>2</sup>-Ph<sub>2</sub>C<sub>2</sub>)Cl<sub>3</sub>(Hsal-5-Cl)]<sub>2</sub> (where Hsal is a salicylate monoanion; Baroni *et al.*, 1996), involving a stronger hydrogen bond, as seen from the O...Cl distance of 3.03 (2) Å. The carboxylic OH group generally forms stronger hydrogen bonds than alcoholic OH groups (Novak, 1974). Intermolecular hydrogen bonds of a similar size have been encountered in [{CuCl<sub>2</sub>(H<sub>2</sub>pin)}<sub>2</sub>] (H<sub>2</sub>pin is pinacol; Sillanpää, 1984), where the H...Cl distances vary from 3.082 (5) to 3.168 (5) Å.

### Experimental

2,3-Dimethylbutane-2,3-diol (2.0 mmol, 0.237 g) was added to a solution of WOCl<sub>3</sub> (1.0 mmol, 0.342 g) in diethyl ether (20 ml) and the reaction mixture was stirred under a slow stream of nitrogen. An immediate reaction ensued and the colour of the solution changed from orange-red to colourless. The nitrogen flow was continued until hydrogen chloride was evolved (1 h). Complex (1) separated as colourless crystals (0.3 g, 64% yield) over a period of 5 h at room temperature. One of these crystals was selected for X-ray analysis.

#### Crystal data

[WOCl(C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>)(C<sub>6</sub>H<sub>13</sub>O<sub>2</sub>)]

*M<sub>r</sub>* = 468.62

Triclinic

*P*1̄

*a* = 8.4200 (7) Å

*b* = 12.908 (2) Å

*c* = 7.9874 (9) Å

α = 94.902 (11)<sup>o</sup>

β = 93.311 (8)<sup>o</sup>

γ = 104.834 (8)<sup>o</sup>

*V* = 833.3 (2) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.868 Mg m<sup>-3</sup>

*D<sub>m</sub>* not measured

Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 25

reflections

θ = 24.8–25.0<sup>o</sup>

μ = 7.10 mm<sup>-1</sup>

*T* = 293 (2) K

Prism

0.18 × 0.12 × 0.10 mm

Colourless

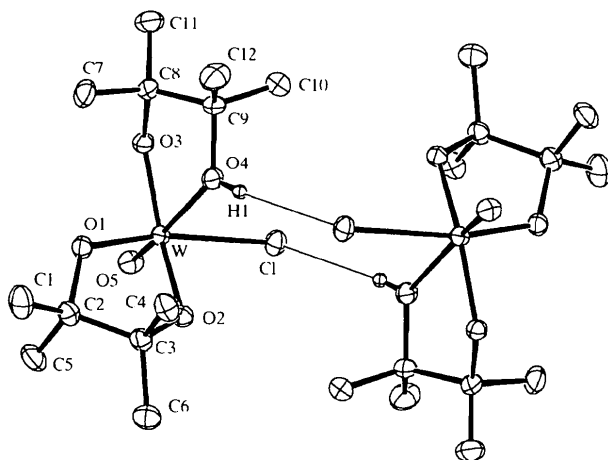


Fig. 1. ORTEP (Johnson, 1976) plot of dimeric (1) with the numbering scheme of the asymmetric unit. Displacement ellipsoids are drawn at the 20% probability level and H atoms, except for H1, have been omitted.

**Data collection**

Rigaku AFC-5S diffractometer  
 $\omega$ -2 $\theta$  scans  
 Absorption correction: empirical via  $\psi$  scans (North *et al.*, 1968)  
 $T_{\min} = 0.342$ ,  $T_{\max} = 0.491$   
 3157 measured reflections  
 2936 independent reflections

2755 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.013$   
 $\theta_{\max} = 25^\circ$   
 $h = 0 \rightarrow 10$   
 $k = -15 \rightarrow 15$   
 $l = -10 \rightarrow 10$   
 3 standard reflections every 150 reflections  
 intensity decay: -1%

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.020$   
 $wR(F^2) = 0.045$   
 $S = 1.096$   
 2936 reflections  
 175 parameters  
 H atoms: see below  
 $w = 1/[\sigma^2(F_o^2) + (0.0154P)^2 + 1.403P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 1.070 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.734 \text{ e } \text{\AA}^{-3}$   
 Extinction correction: none  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

W—O5	1.716 (3)	O1—C2	1.454 (5)
W—O3	1.874 (3)	O2—C3	1.452 (5)
W—O1	1.900 (3)	O3—C8	1.462 (5)
W—O2	1.911 (3)	O4—C9	1.470 (5)
W—O4	2.293 (3)	C2—C3	1.563 (6)
W—Cl	2.4212 (11)		
O5—W—O3	98.88 (13)	O5—W—Cl	90.25 (11)
O5—W—O1	101.17 (14)	O3—W—Cl	101.19 (9)
O3—W—O1	91.69 (12)	O1—W—Cl	161.32 (9)
O5—W—O2	106.32 (13)	O2—W—Cl	84.58 (9)
O3—W—O2	154.16 (12)	O4—W—Cl	81.19 (8)
O1—W—O2	78.08 (12)	C2—O1—W	119.6 (3)
O5—W—O4	164.97 (13)	C3—O2—W	121.2 (2)
O3—W—O4	70.99 (11)	C8—O3—W	128.9 (3)
O1—W—O4	90.44 (12)	C9—O4—W	116.5 (2)
O2—W—O4	85.27 (11)		

An error in the crystal size measurement caused difficulties in absorption correction. Heavy atoms were refined with anisotropic displacement parameters. The hydroxyl H atom was refined with a displacement parameter 1.2 times that of the host O atom. The CH H atoms were refined as riding on their host atoms.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1995). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1506). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). **C54**, 937–939

**Layered Structure of Lithium Ethylene Glycolate, Li(OCH<sub>2</sub>CH<sub>2</sub>OH)**

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(Received 4 November 1997; accepted 3 February 1998)

**Abstract**

Monolithium ethylene glycolate, [Li(C<sub>2</sub>H<sub>5</sub>O<sub>2</sub>)], crystallizes in the orthorhombic system. Its structure was solved from single-crystal data with a final  $R = 0.036$ . The O atoms of the bridging and chelating ethylene glycol ligands form tetrahedra around the Li atoms, and these tetrahedra form layers by sharing corners and edges. Hydroxyl groups form hydrogen bonds within the layer and the layers are held together by van der Waals forces only.

**Comment**

Ethylene glycolate, HOCH<sub>2</sub>CH<sub>2</sub>O<sup>-</sup>, behaves as a chelating and bridging ligand. In the title compound, (I), it coordinates one Li atom by both O atoms, and these O atoms coordinate one additional Li atom each (Fig. 1). Thus, the Li coordination polyhedron is a tetra-