

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

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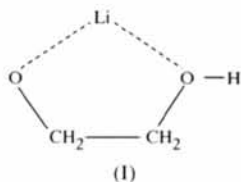
(Received 4 November 1997; accepted 3 February 1998)

**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-



hedron, which shares an edge and two corners with other tetrahedra, forming a layer (Fig. 2). The hydroxyl group (O2) forms hydrogen bonds with O atoms (O1) within the layer, but since the methylene groups of the ligand make both surfaces of the layer hydrophobic (Fig. 1), only van der Waals forces hold the layers together.

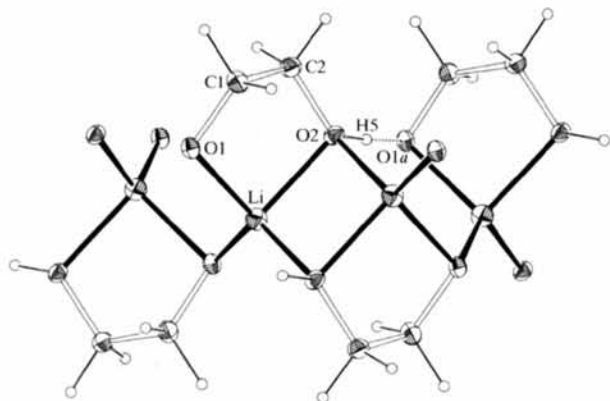


Fig. 1. A layer of Li(OCH<sub>2</sub>CH<sub>2</sub>OH), viewed approximately down the *a* axis. Displacement ellipsoids are shown at 50% probability and H atoms are drawn as small circles.

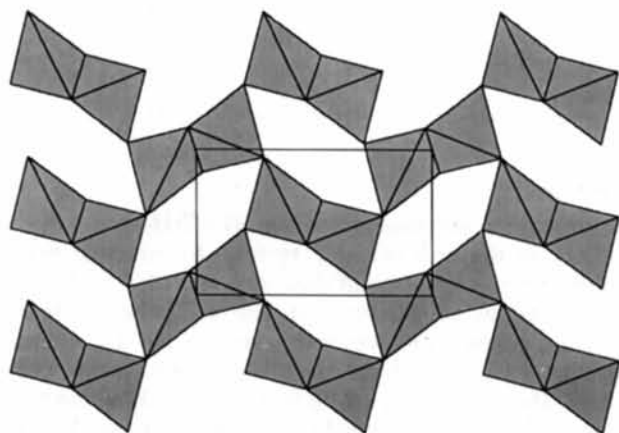


Fig. 2. Packing of the Li tetrahedra viewed down the *c* axis.

## Experimental

MnO<sub>2</sub>, LiOH and ethylene glycol (HOCH<sub>2</sub>CH<sub>2</sub>OH), in a ratio of 1:10:360, were reacted hydrothermally at 463 K for 3 d in an attempt to synthesize layered manganese oxide. Two kinds of colorless crystals were found in the post-reaction mixture. One of them, a crystal of irregular shape, lost its transparency

and turned into powder in the air, even while covered with glue. The second kind, with sharper faces, was more stable, and its crystal structure has been determined.

## Crystal data

[Li(C<sub>2</sub>H<sub>5</sub>O<sub>2</sub>)]

*M<sub>r</sub>* = 68.01

Orthorhombic

*Pbca*

*a* = 5.0283 (3) Å

*b* = 8.0427 (5) Å

*c* = 16.030 (1) Å

*V* = 648.3 (1) Å<sup>3</sup>

*Z* = 8

*D<sub>x</sub>* = 1.3935 Mg m<sup>-3</sup>

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

Mo *K*α radiation

λ = 0.71069 Å

Cell parameters from 509 reflections

θ = 5–28°

μ = 0.115 mm<sup>-1</sup>

*T* = 295 K

Prism

0.32 × 0.26 × 0.13 mm

Colorless

## Data collection

Siemens SMART CCD

diffractometer

ω scans

Absorption correction: none

4116 measured reflections

945 independent reflections

772 reflections with

*F<sub>o</sub>* > 4σ(*F<sub>o</sub>*)

*R<sub>int</sub>* = 0.029

θ<sub>max</sub> = 28.2°

*h* = -6 → 5

*k* = -10 → 6

*l* = -20 → 17

Intensity decay: 0.2%

## Refinement

Refinement on *F*

*R* = 0.036

*wR* = 0.055

*S* = 1.63

772 reflections

66 parameters

All H atoms refined

*w* = 1/[σ(*F<sub>o</sub>*)<sup>2</sup> + 0.002*F<sub>o</sub>*<sup>2</sup>]

(Δ/σ)<sub>max</sub> = 0.01

Δρ<sub>max</sub> = 0.21 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.27 e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from *International Tables for X-ray*

*Crystallography* (Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

	$U_{eq} = (1/3)\sum_i \sum_j U^{ij} a^i a^j \mathbf{a}_i \cdot \mathbf{a}_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
Li	0.1331 (3)	0.6367 (2)	-0.01412 (1)	0.0165 (5)
O1	-0.04666 (1)	0.78876 (8)	0.06592 (4)	0.0137 (3)
O2	0.15516 (1)	0.46838 (8)	0.07685 (4)	0.0143 (3)
C1	-0.0941 (2)	0.69042 (1)	0.13803 (6)	0.0158 (3)
C2	0.1252 (2)	0.56298 (1)	0.15127 (5)	0.0155 (3)

Table 2. Selected bond lengths (Å)

Li—O1 <sup>i</sup>	1.908 (2)	O1—C1	1.421 (1)
Li—O2 <sup>ii</sup>	1.956 (2)	O2—C2	1.423 (1)
Li—O1	1.990 (2)	C1—C2	1.520 (1)
Li—O2	1.993 (2)		

Symmetry codes: (i)  $\frac{1}{2} + x, \frac{1}{2} - y, -z$ ; (ii)  $-x, 1 - y, -z$ .

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O2—H5...O1 <sup>i</sup>	1.18 (2)	1.28 (2)	2.448 (1)	170 (2)

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

The crystal structure was solved using direct methods. All atoms were found from the *E* map (H atoms from the difference map). Final refinement was conducted in an anisotropic (H atoms in an isotropic) approximation.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *CSD* (Akselrud *et al.*, 1993). Program(s) used to refine structure: *CSD*. Molecular graphics: *CrystalDesigner* (Crystal Structure Design, 1997) and *ORTEP3* (Farrugia, 1997). Software used to prepare material for publication: *CSD*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1011). Services for accessing these data are described at the back of the journal.

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## Di- $\mu$ -chloro-bis[chlorodimethyl(3-methyl-4-nitropyridine *N*-oxide-*O*)tin]

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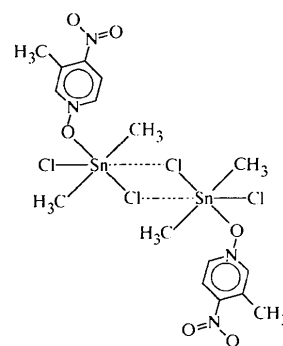
### Abstract

The title compound,  $[\text{Sn}_2\text{Cl}_4(\text{CH}_3)_4(\text{C}_6\text{H}_6\text{N}_2\text{O}_3)_2]$ , crystallizes as a six-coordinate *trans*- $\text{C}_2\text{SnOCl}_3$  octahedral chlorine-bridged centrosymmetric dimer; some geometric details are Sn—O 2.399 (2), Sn—Cl 2.3583 (7) and 2.4774 (7), Sn—Cl<sub>bridge</sub> 3.9341 (8) Å and C—Sn—C 137.7 (2)°.

### Comment

3-Methyl-4-nitropyridine *N*-oxide (POM), a material for non-linear optics (Sigelle *et al.*, 1982), forms complexes

with a number of inorganic salts. These complexes exhibit strong second harmonic generation (SHG) effects, as exemplified by  $\text{CdBr}_2 \cdot 2\text{POM}$ , the SHG of which is 18 times that of potassium dihydrogen phosphate (Li *et al.*, 1992; Hu *et al.*, 1994). This SHG property implies that the complexes must be non-centrosymmetric (Dougherty & Kurtz, 1976). On the other hand, POM is an O-donor ligand that should yield complexes with Lewis basic dimethyltin dichloride, which forms 1:2 complexes with pyridine *N*-oxide (Blom *et al.*, 1969) and quinoline *N*-oxide (Ng, 1994), but a 1:1 complex with 2,6-dimethylpyridine *N*-oxide (Ng *et al.*, 1983). The dimethyltin dichloride–POM adduct was expected to crystallize in a non-centrosymmetric space group, but the crystal structure determination shows that the adduct, (I), packs across a center of inversion as a chlorine-bridged  $[\text{Sn}—\text{Cl} \ 3.9341(8) \ \text{\AA}]$  six-coordinate dimeric compound.



(I)

The Sn—Cl bridge is much weaker than that [3.399 (1) Å] in the 2,6-dimethylpyridine *N*-oxide complex, as well as that [3.561 (1) Å] in the 1,2-diphenylcyclopropanone complex (Ng *et al.*, 1982). With only one possible exception, that of the dimethyl sulfoxide complex (Ng & Rheingold, 1989), the 1:1 adducts of dimethyltin dichloride show sixfold coordination at tin in the solid state (Ng & Zuckerman, 1982). The bridging distance is also longer than that [3.54 (5) Å] found in the parent Lewis base, the sixfold-coordinated geometry of which is supported by Mössbauer measurements (Davies *et al.*, 1970).

The Sn atom in the complex shows distorted  $\text{C}_2\text{SnOCl}_3$  octahedral coordination geometry [C—Sn—C 137.7 (2) Å]. The C—Sn—C angle has been opened somewhat by the long chlorine bridge. The complex adopts five-coordinate geometry in solution, as suggested by the magnitudes of the coupling constants in the NMR spectra. Bond distances involving the POM moiety are similar to those found in POM itself (Shiro *et al.*, 1977; Baert *et al.*, 1988). However, the nitro plane is twisted out of the pyridyl plane by 24.7 (2)° in the complex, whereas in POM, the twist is only 16.7° (Shiro *et al.*, 1977).