

19.02 (2),  $b = 11.67$  (2),  $c = 15.49$  (2) Å,  $\beta = 95.6$  (1)°,  $V = 3421$  Å<sup>3</sup>,  $Z = 8$ ;  $R_F = 0.149$  for 3638 Cu  $K\alpha$  Weissenberg data], which consists of four independent chains of similar molecular dimensions along the screw diads. The same type of polymeric structure has been found in Ph<sub>3</sub>SnNCO (Tarkova, Chuprunov, Simonov & Belov, 1977). In the presence of another ligand such as a Schiff base, Ph<sub>3</sub>SnNCS can form monomeric complexes exhibiting trigonal bipyramidal coordination geometry, for example, [L<sub>2</sub>H]<sup>+</sup>[Ph<sub>3</sub>Sn(NCS)<sub>2</sub>]<sup>-</sup> [ $L = 1$ -(salicylideneamino)-2-methoxybenzene] (Charland *et al.*, 1989) and Ph<sub>3</sub>Sn(NCS)L [ $L = 1$ -[(4'-methylphenylimino)methyl]-2-naphthol] (Khoo, Charland, Gabe & Smith, 1987).

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## Structure of Dimeric Lithium 2,6-Di-*tert*-butylphenoxide–Diethyl Ether (1/1)

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**Abstract.** {Li[OC<sub>6</sub>H<sub>3</sub>(C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>][O(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>]}<sub>2</sub>,  $M_r = 572.77$ , triclinic,  $P\bar{1}$ ,  $a = 10.188$  (8),  $b = 11.202$  (9),  $c = 9.220$  (5) Å,  $\alpha = 99.92$  (6),  $\beta = 113.39$  (5),  $\gamma = 68.31$  (7)°,  $V = 897.02$  Å<sup>3</sup>,  $Z = 1$  (one dimer),  $D_x = 1.06$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.61$  cm<sup>-1</sup>,  $F(000) = 316$ ,  $T = 230$  (5) K,  $R = 0.063$  for 1467 observed reflections with  $I > 2\sigma(I)$ . The structure is that of a centrosymmetric dimer, the phenoxide-O act as bridging atoms and the Li<sub>2</sub>( $\mu$ -O)<sub>2</sub> unit is planar. Each Li atom carries an additional ether ligand and thus shows the unusual coordination number of three.

**Experimental.** Reaction of [Lu(CH<sub>3</sub>)<sub>6</sub>][Li(TMEDA)]<sub>3</sub> (TMEDA = Me<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>NMe<sub>2</sub>) with cyclopentadiene and 2,6-di-*tert*-butylphenol in toluene at 195 K gave the title compound and other Lu-containing products. Crystals were obtained by extracting the white residue of the reaction mixture with ether/pentane (1/4) and keeping the extract at 253 K. As the compound rapidly loses ether at room temperature, all crystal manipulations had to be carried out at low temperature in a cold nitrogen stream.

Colourless crystal (0.42 × 0.28 × 0.25 mm) mounted on glass fibre and transferred to Enraf–Nonius

CAD-4 diffractometer equipped with low-temperature device. Lattice parameters derived from setting angles of 25 reflections,  $12 \leq 2\theta \leq 24^\circ$ . Intensity data were collected in the  $\omega$ - $2\theta$  scan mode with  $\Delta\omega = (0.90 + 0.35\tan\theta)^\circ$ , using graphite-monochromated Mo  $K\alpha$  radiation. Three reference reflections recorded every two hours showed only random variations. Intensity data for 3368 reflections with  $2\theta \leq 50^\circ$  ( $-11 \leq h \leq 12$ ,  $-13 \leq k \leq 13$ ,  $-10 \leq l \leq 0$ ); 2525 unique reflections ( $R_{\text{int}} = 0.018$ ), 1467 reflections with  $I > 2\sigma(I)$  used for refinement. Corrections for Lorentz and polarization effects. Structure solved with direct methods using *SHELXS86* (Sheldrick, 1986) and refined on  $F$  by full-matrix least squares with *SHELX76* (Sheldrick, 1976); non-H atoms refined with anisotropic thermal parameters, H atoms located from a difference Fourier map and refined isotropically, except the H atoms of the ether CH<sub>3</sub> groups. The C atoms of the ether methyl groups showed rather high temperature factors, indicating a small degree of disorder; therefore these CH<sub>3</sub> groups were treated as rigid groups with C—H bond lengths of 0.95 Å and a common isotropic temperature factor  $U = 0.08$  Å<sup>2</sup> for the H atoms. 290 parameters were refined,  $R = 0.063$ ,  $wR = 0.082$ ,  $w^{-1} = [\sigma^2(F_o) + 0.0027 F_o^2]$ ,  $S = 0.67$ ,  $(\Delta/\sigma)_{\text{max}} = 0.014$ ;  $|\Delta e| <$

Table 1. Atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ ), with *e.s.d.*'s in parentheses
$$B_{eq} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}$
Li	0.0457 (8)	0.4178 (6)	0.4111 (8)	3.14
O(1)	-0.0473 (3)	0.5913 (2)	0.3869 (3)	2.38
O(2)	0.1146 (4)	0.2773 (3)	0.2798 (4)	3.71
C(1)	-0.0865 (4)	0.7044 (3)	0.3237 (4)	2.19
C(2)	-0.2386 (4)	0.7639 (4)	0.2196 (4)	2.36
C(3)	-0.2784 (5)	0.8868 (4)	0.1729 (5)	3.00
C(4)	-0.1747 (6)	0.9514 (5)	0.2215 (6)	3.80
C(5)	-0.0283 (6)	0.8908 (4)	0.3119 (5)	3.27
C(6)	0.0217 (4)	0.7665 (4)	0.3604 (4)	2.46
C(7)	-0.3561 (4)	0.6948 (4)	0.1582 (5)	2.81
C(8)	-0.2972 (7)	0.5675 (5)	0.0771 (7)	3.52
C(9)	-0.3963 (7)	0.6766 (6)	0.2951 (7)	3.87
C(10)	-0.5039 (6)	0.7738 (6)	0.0350 (7)	4.33
C(11)	0.1910 (5)	0.6985 (4)	0.4525 (5)	3.11
C(12)	0.2519 (7)	0.5745 (5)	0.3657 (8)	3.85
C(13)	0.2884 (7)	0.7811 (6)	0.4682 (8)	4.25
C(14)	0.2185 (7)	0.6716 (6)	0.6208 (6)	3.65
C(15)	0.1961 (8)	0.1500 (5)	0.3350 (8)	4.76
C(16)	0.3425 (9)	0.0950 (7)	0.3081 (10)	8.75
C(17)	0.0565 (8)	0.2809 (6)	0.1074 (7)	5.48
C(18)	-0.0687 (9)	0.2233 (7)	0.0321 (7)	7.67

Table 2. Selected bond distances ( $\text{\AA}$ ), bond angles and torsion angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

Li—Li <sup>i</sup>	2.427 (14)	C(4)—C(5)	1.356 (6)
Li—O(1)	1.830 (7)	C(5)—C(6)	1.382 (6)
Li—O(1)	1.880 (7)	C(6)—C(11)	1.538 (5)
Li—O(2)	1.904 (7)	C(7)—C(8)	1.519 (6)
O(1)—C(1)	1.342 (5)	C(7)—C(9)	1.540 (1)
O(2)—C(15)	1.422 (6)	C(7)—C(10)	1.531 (6)
O(2)—C(17)	1.461 (7)	C(11)—C(12)	1.526 (7)
C(1)—C(2)	1.430 (4)	C(11)—C(13)	1.540 (1)
C(1)—C(6)	1.415 (7)	C(11)—C(14)	1.527 (8)
C(2)—C(3)	1.377 (6)	C(15)—C(16)	1.490 (1)
C(2)—C(7)	1.532 (7)	C(17)—C(18)	1.510 (1)
C(3)—C(4)	1.386 (8)		
O(1)—Li—O(2)	134.7 (5)	C(5)—C(6)—C(11)	120.3 (5)
Li—O(1)—C(1)	155.1 (4)	C(2)—C(7)—C(8)	110.3 (4)
Li—O(2)—C(15)	122.5 (4)	C(2)—C(7)—C(9)	109.8 (4)
Li—O(2)—C(17)	123.5 (3)	C(2)—C(7)—C(10)	111.4 (4)
C(15)—O(2)—C(17)	112.2 (4)	C(8)—C(7)—C(9)	111.8 (5)
O(1)—C(1)—C(2)	120.2 (4)	C(8)—C(7)—C(10)	106.7 (4)
O(1)—C(1)—C(6)	120.5 (3)	C(9)—C(7)—C(10)	106.8 (5)
C(2)—C(1)—C(6)	119.4 (4)	C(6)—C(11)—C(12)	110.6 (3)
C(1)—C(2)—C(3)	118.1 (4)	C(6)—C(11)—C(13)	112.1 (4)
C(1)—C(2)—C(7)	122.1 (4)	C(6)—C(11)—C(14)	111.2 (4)
C(3)—C(2)—C(7)	119.8 (3)	C(12)—C(11)—C(13)	105.8 (5)
C(2)—C(3)—C(4)	121.8 (4)	C(12)—C(11)—C(14)	110.7 (4)
C(3)—C(4)—C(5)	119.5 (5)	C(13)—C(11)—C(14)	106.3 (4)
C(4)—C(5)—C(6)	122.3 (5)	O(2)—C(15)—C(16)	111.9 (7)
C(1)—C(6)—C(5)	118.4 (4)	O(2)—C(15)—C(17)	111.7 (7)
C(1)—C(6)—C(11)	121.3 (4)		
O(1)—Li—O(2)—C(15)	175.2 (6)	Li—O(1)—C(1)—C(2)	108.1 (8)
O(1)—Li—O(2)—C(17)	-21.4 (9)	Li—O(1)—C(1)—C(6)	-72.4 (9)
O(2)—Li—O(1)—C(1)	-24.5 (13)		

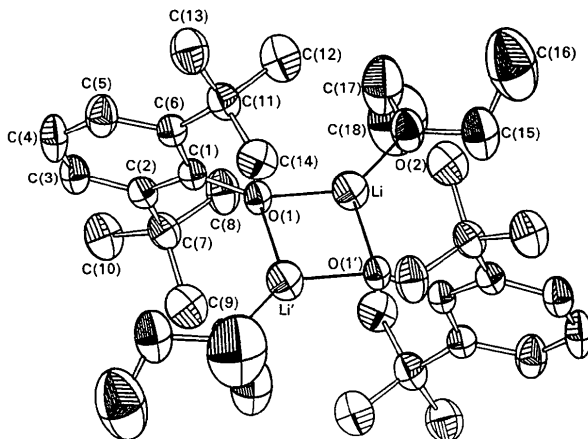
Symmetry code: (i)  $-x, 1-y, 1-z$ .

Fig. 1. ORTEP (Johnson, 1976) drawing of the dimer with 50% probability ellipsoids, showing the atom-numbering scheme.

$0.3 \text{ e \AA}^{-3}$ . Scattering factors for Li from Cromer & Mann (1968), for C, O, and H as supplied by SHELX76. Final positional and equivalent isotropic thermal parameters are given in Table 1.\* Fig. 1 shows the numbering of the atoms. Bond lengths and angles are listed in Table 2.

The compound is dimeric and has the symmetry  $C_2 - \bar{1}$ . The O atoms of the 2,6-di-*tert*-butylphenoxide groups act as bridging atoms, forming a slightly unsymmetric bridge with two significantly different bond lengths Li—O(1) 1.830 (7)  $\text{\AA}$ , and Li<sup>i</sup>—O(1)

1.880 (7)  $\text{\AA}$ . The Li<sub>2</sub>( $\mu$ -O)<sub>2</sub> unit is planar. The planes of the bulky 2,6-di-*tert*-butylphenoxide ligands are almost perpendicular (88°) to this plane. There is a slight out of plane deviation for the O atoms of the ether groups (3°) and the atom C(1) of the phenoxy groups (8°). The *tert*-butyl groups are bent out of the ring plane (5°) of the phenoxy groups. The Li atoms show coordination number three. The Li—O(2) bond to the ether groups is significantly longer [1.904 (7)  $\text{\AA}$ ] than the Li—O(phenoxy) bonds, indicating a weaker bonding; this reflects the thermal instability of the compound, due to an easy loss of the ether groups.

**Related literature.** The title compound was obtained in the course of studying the reactions of the very reactive hexamethyl lutetium (Schumann, Müller, Bruncks, Lauke, Pickardt, Schwarz & Eckart, 1984) with cyclopentadiene and 2,6-di-*tert*-butylphenoxide. The Li—O distances found for the title compound compare favourably with distances found in similar complexes, *e.g.* [Li(2,6-di-*tert*-butyl-4-methylphenoxide)(Et<sub>2</sub>O)]<sub>2</sub> (Çetinkaya, Gümrükçü & Lappert, 1980), [Li(OC<sup>t</sup>Bu<sub>3</sub>)(THF)]<sub>2</sub> (Power, Hvovslev, Hope & Murray, 1983) and [Li(2,6-di-*tert*-butylphenoxide)-(THF)]<sub>2</sub> (Huffman, Geerts & Caulton, 1984).

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\* 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 54407 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of $\text{pzB}(\text{pz})_3\text{Mo}(\text{CO})_2(\eta^2\text{-OCNMe}_2)$

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**Abstract.** Dicarbonyl(dimethylaminocarbonyl)(1-pyrazolyl)tris(pyrazolylborane)molybdenum,  $\text{C}_{17}\text{H}_{18}\text{BMoN}_9\text{O}_3$ ,  $M_r = 503.14$ , monoclinic,  $C2/c$ ,  $a = 24.568$  (3),  $b = 9.089$  (2),  $c = 18.616$  (2) Å,  $\beta = 95.03$  (1)°,  $V = 4141$  (2) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.61$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 6.6$  cm<sup>-1</sup>,  $F(000) = 2032$ ,  $T = 294$  K, final  $R = 0.029$  for 2967 unique observed reflections. Crystals of the title compound  $\text{pzB}(\text{pz})_3\text{Mo}(\text{CO})_2(\eta^2\text{-OCNMe}_2)$  (I) (pz = 1-pyrazolyl) were isolated in low yield from the reaction of an excess of diethylamine with  $[\text{pzB}(\text{pz})_3(\text{CO})_2\text{-Mo}(\eta^2\text{-SMcCNMe}_2)]\text{BF}_4$ . An attempt to achieve a rational synthesis of the complex *via* the reaction of  $\text{Me}_2\text{NCOCl}$  with  $[\text{pzB}(\text{pz})_3\text{Mo}(\text{CO})_3]^-$  was not successful. Compound (I) is isostructural (but not isomorphous) with  $\text{pzB}(\text{pz})_3\text{Mo}(\text{CO})_2(\eta^2\text{-SCNMe}_2)$  (II) [Desmond, Lalor, O'Sullivan & Ferguson (1990). *J. Organomet. Chem.* **381**, C33–C37] with the sulfur atom replaced by an oxygen in (I). As in (II) the  $\eta^2\text{-OCN}(\text{CH}_3)_2$  is situated between two carbonyl ligands and one pyrazolyl ring, but close to the C5—O5 group; the orientation of the dihapto ligand is specified by two torsion angles N21—Mo—O1—C1 [ $-75.7$  (2)°] and C5—Mo—O1—C1 [ $33.9$  (2)°]; the corresponding values in (II) are  $-74.9$  (2) and  $42.3$  (2)°, respectively. The conformation of the two molecules differs only in the orientation of the uncomplexed pyrazolyl ring (N41—C45), presumably

due to differences in crystal packing. The coordination about the Mo atom is distorted octahedral with the tridentate ligand [Mo—N11 2.181 (3), Mo—N21 2.294 (3), Mo—N31 2.218 (3) Å] and two carbonyl groups [Mo—C4 1.933 (4), Mo—C5 1.970 (3) Å] occupying five sites and the  $\eta^2\text{-OCN}(\text{CH}_3)_2$  ligand in the sixth position [Mo—O1 2.207 (2), Mo—C1 2.050 (3) Å]. The packing is due mainly to van der Waals interactions and all intermolecular contacts agree with those predicted from radii-sum rules.

**Experimental.** Three-dimensional intensity data were collected on an Enraf–Nonius CAD-4 diffractometer using a dark-red needle-shaped crystal  $0.18 \times 0.11 \times 0.38$  mm and graphite-monochromated Mo  $K\alpha$  radiation; lattice parameters were refined using 25 reflections in the range  $8 < \theta < 20^\circ$ ; reflections were measured using  $\omega/2\theta$  scan ( $2 < 2\theta < 54^\circ$ );  $\omega$ -scan width ( $0.6 + 0.35\tan\theta$ )°; range of  $hkl$ :  $h$  0 to 31,  $k$  0 to 8,  $l$   $-17$  to  $17$ ; 4928 reflections were measured of which only 3026 had  $I \geq 3\sigma I$  and were labelled observed. Three reflections (10,0,4, 408 and 134) were measured periodically throughout the data collection and showed less than 2% variation. After averaging equivalent reflections ( $R_{\text{int}} = 0.009$ ), 2967 reflections were retained and used in the analysis. Lorentz, polarization and absorption corrections [Gaussian integration; Coppens, Leiserowitz & Rabinovich (1965)] were applied to the data; range of transmission coefficients 0.890 to 0.934. Structure was solved by the heavy-atom method; a Fourier map calculated on the basis of the position of the Mo atom revealed the entire molecule. Structural

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