

- Melnik, A. C., Kildahl, N. K., Rendina, A. R. & Busch, D. H. (1979). *J. Am. Chem. Soc.* **101**, 3232–3240.
- Norman, R. E., Yan, S., Que, L. Jr, Backes, G., Ling, J., Sanders-Loehr, J., Zhang, J. H. & O'Connor, C. J. (1990). *J. Am. Chem. Soc.* **112**, 1554–1562.
- Rabion, A., Chen, S., Wang, J., Buchanan, R. M., Seris, J.-L. & Fish, R. H. (1995). *J. Am. Chem. Soc.* **117**, 12356–12357.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1996a). *SADABS. Absorption Correction Program*. University of Göttingen, Germany.
- Sheldrick, G. M. (1996b). *SHELXL96. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Siemens (1995). *SMART and SAINT. Data Collection and Processing software for the SMART System*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1997). **C53**, 1387–1388

catena-Poly[[1,4-dioxane-*O*¹]iodolithium]- μ -(1,4-dioxane-*O*¹:*O*⁴)

COLIN EABORN*, ADAM FAROOK, PETER B. HITCHCOCK AND J. DAVID SMITH*

School of Chemistry, Physics and Environmental Science, University of Sussex, Brighton BN1 9QJ, England. E-mail: c.eaborn@sussex.ac.uk

(Received 8 April 1997; accepted 6 June 1997)

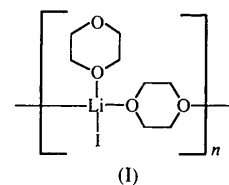
Abstract

In the title compound, $[\text{LiI}(\text{C}_4\text{H}_8\text{O}_2)(\mu\text{-C}_4\text{H}_8\text{O}_2)]_n$, obtained by recrystallization of LiI from 1,4-dioxane, the ligand is present in both monodentate and bridging modes, the latter resulting in a linear polymeric structure.

Comment

A few structures are known in which 1,4-dioxane is coordinated to Li (Bartlett *et al.*, 1986; Evans, Dominguez, Levan & Doedens, 1985; Veith, Ruloff, Huch & Töllner, 1988; Belzner, Dehnert & Stalke, 1994; Müller & Krause, 1972; Taube, Windisch, Görlitz & Schumann, 1993; Andrews *et al.*, 1995; Uhl, Klinkhammer, Layh & Massa, 1991; Cramer, Bruck & Gilje, 1986; Nöth, Thomas & Schmidt, 1996; West, Sohn, Powell, Müller & Apeloig, 1996). In a small number of these, the dioxane bridges through its two O atoms between two Li centres (Evans *et al.*, 1985), in some cases to give polymers (Taube *et al.*, 1993; Andrews *et al.*, 1995; Nöth *et al.*, 1996), and there are two rather complex species in which there is both bridging and monodentate coordination (Cramer *et al.*, 1986; Uhl *et al.*, 1991). The

title compound, (I), provides a rather striking example of such dual functionality in which the bridging mode gives rise to a very simple linear polymeric structure aligned along a twofold screw axis.



We initially isolated crystals of the title compound, (I), unexpectedly as a consequence of using 1,4-dioxane during work-up of a product mixture formed from a reaction of Sm with $(\text{Me}_2\text{NMe}_2\text{Si})_3\text{Cl}$ that had evidently retained a little of the LiI generated during its preparation. Having identified the solvated salt by determination of its crystal structure, we then made it simply by recrystallizing LiI from 1,4-dioxane, as described in the *Experimental* section, confirming by determination of the unit cell that the crystals were identical to those examined previously.

The structure is shown in Fig. 1, and bond lengths and angles are listed in Table 1. As can be seen from Table 1, there is no significant difference between the Li—O bonds to the bridging and terminal 1,4-dioxane ligands or between the geometries of the two types of dioxane rings, which are in chair conformations.

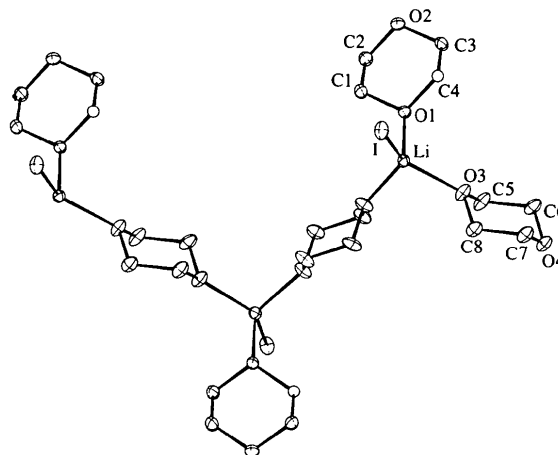


Fig. 1. The structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

Experimental

To ensure the absence of traces of water, LiI was generated by adding a solution of BuLi (3.55 mmol) in hexane (1.42 ml) dropwise to a stirred solution of $\text{ICH}_2\text{CH}_2\text{I}$ (3.55 mmol) in heptane (30 ml). A white precipitate separated immediately

but the mixture was stirred for 1 h before the solvent was taken off under vacuum. The white solid residue was taken up in hot 1,4-dioxane and crystals (0.43 g, 91%) suitable for the crystallographic study separated on cooling.

Crystal data

[LiI(C₄H₈O₂)₂]

$M_r = 310.05$

Monoclinic

$P2_1/n$

$a = 7.726(2) \text{ \AA}$

$b = 10.220(2) \text{ \AA}$

$c = 15.169(3) \text{ \AA}$

$\beta = 99.67(2)^\circ$

$V = 1180.7(4) \text{ \AA}^3$

$Z = 4$

$D_x = 1.744 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 7-10^\circ$

$\mu = 2.698 \text{ mm}^{-1}$

$T = 173(2) \text{ K}$

Block

$0.30 \times 0.30 \times 0.25 \text{ mm}$

Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction:

ψ scan (MolEN; Fair, 1990)

$T_{\min} = 0.46$, $T_{\max} = 0.51$

3030 measured reflections

2837 independent reflections

2178 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\text{max}} = 27.97^\circ$

$h = 0 \rightarrow 10$

$k = 0 \rightarrow 13$

$l = -20 \rightarrow 19$

2 standard reflections

frequency: 120 min

intensity decay: 0.5%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.084$

$S = 1.005$

2837 reflections

123 parameters

H atoms riding

$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2]$

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

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

$\Delta\rho_{\text{max}} = 1.157 \text{ e \AA}^{-3}$ (at centre of a dioxane ring)

$\Delta\rho_{\text{min}} = -0.634 \text{ e \AA}^{-3}$

Extinction correction:

SHELXL93

Extinction coefficient:

0.0091 (6)

Scattering factors from

International Tables for Crystallography (Vol. C)

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

Li—O1	2.640 (6)	Li—O1	1.923 (6)
Li—O3	1.944 (6)	Li—O4 ⁱ	1.940 (6)
O1—Li—O1	111.1 (3)	O3—Li—O1	112.3 (3)
O4 ⁱ —Li—O1	119.5 (3)	O1—Li—O3	109.0 (3)
O1—Li—O4 ⁱ	101.4 (3)	O4 ⁱ —Li—O3	102.6 (3)

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

Data collection: CAD-4 Software (Enraf-Nonius, 1989). Cell refinement: CAD-4 Software. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: CAMERON (Watkin & Pearce, 1993). Software used to prepare material for publication: SHELXL93.

We thank the EPSRC for support of this work and the Universiti Sains Malaysia for the award of a postgraduate scholarship to AF.

Supplementary data for this paper are available from the IUCR electronic archives (Reference: CF1181). Services for accessing these data are described at the back of the journal.

References

- Andrews, P. C., Armstrong, D. R., Baker, D. R., Mulvey, R. E., Clegg, W., Horsburgh, L., O'Neill, P. A. & Reed, D. (1995). *Organometallics*, **14**, 427–439.
- Bartlett, R. A., Dias, H. V. R., Hope, H., Murray, B. D., Olmstead, M. M. & Power, P. P. (1986). *J. Am. Chem. Soc.* **108**, 6921–6926.
- Belzner, J., Dehnert, U. & Stalke, D. (1994). *Angew. Chem. Int. Ed. Engl.* **33**, 2450–2452.
- Cramer, R. E., Bruck, M. A. & Gilje, J. W. (1986). *Organometallics*, **5**, 1496–1499.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Evans, W. J., Dominguez, R., Levan, K. R. & Doedens, R. J. (1985). *Organometallics*, **4**, 1836–1841.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf-Nonius, Delft, The Netherlands.
- Müller, B. & Krause, J. (1972). *J. Organomet. Chem.* **44**, 141–159.
- Nöth, H., Thomas, S. & Schmidt, M. (1996). *Chem. Ber.* **129**, 451–458.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Taube, R., Windisch, H., Görlitz, F. H. & Schumann, H. (1993). *J. Organomet. Chem.* **445**, 85–91.
- Uhl, W., Klinkhammer, K.-W., Layh, M. & Massa, W. (1991). *Chem. Ber.* **124**, 279–284.
- Veith, M., Ruloff, C., Huch, V. & Töllner, F. (1988). *Angew. Chem. Int. Ed. Engl.* **27**, 1381–1382.
- Watkin, D. J. & Pearce, L. J. (1993). *CAMERON. Chemical Crystallography Laboratory*. University of Oxford, England.
- West, R., Sohn, H., Powell, D. R., Müller, T. & Apeloig, Y. (1996). *Angew. Chem. Int. Ed. Engl.* **35**, 1002–1004.

Acta Cryst. (1997). **C53**, 1388–1390

Aquatetrachlorotris(tetrahydrofuran-O)-thorium(IV) Tetrahydrofuran Solvate (1/1)

MARCUS P. SPRY, WILLIAM ERRINGTON AND GERALD R. WILLEY

Department of Chemistry, University of Warwick, Coventry CV4 7AL, England. E-mail: w.errington@warwick.ac.uk

(Received 17 February 1997; accepted 4 June 1997)

Abstract

The title compound, [ThCl₄(C₄H₈O)₃(H₂O)].C₄H₈O, is eight-coordinate with a distorted square-antiprismatic metal-atom geometry; pairs of O or Cl atoms are located at opposite vertices of the square planes.