metal-organic papers

Polymeric [Li(NO$_3$)(monoglyme)]$_n$

Wesley A. Henderson,[a] **Neil R. Brooks**[b] **and William H. Smyrl**[a]*

[a]Corrosion Research Center, Department of Chemical Engineering and Materials Science, University of Minnesota, 221 Church St. SE, Minneapolis, MN 55455, USA, and [b]X-Ray Crystallographic Laboratory, Department of Chemistry, University of Minnesota, 207 Pleasant St. SE, Minneapolis, MN 55455, USA

Correspondence e-mail: smyrl001@umn.edu

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.025
wR factor = 0.060
Data-to-parameter ratio = 13.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, *catena*-poly[[(1,2-dimethoxyethane)-lithium(I)]-μ-nitrato-*O:O'*], [Li(NO$_3$)(C$_4$H$_{10}$O$_2$)]$_n$, is a one-dimensional polymer. In the asymmetric unit, there are two Li$^+$ cations and two monoglyme molecules located on crystallographic twofold axes and one NO$_3^-$ anion on a general position. Both cations are coordinated by two ether O atoms from a single monoglyme. In addition, one cation is coordinated by two bidentate anions, making a six-coordinate Li$^+$, and the other by two monodentate anions, making a four-coordinate Li$^+$. Each NO$_3^-$ anion is coordinated to two Li$^+$ cations, to one in a monodentate fashion and to the other in a bidentate fashion. The one-dimensional Li(monoglyme)NO$_3$ chains are propagated by the 3$_1$ screw axis along the c axis, with alternating four- and six-coordinate Li$^+$ cations.

Comment

Structural characterization of the title compound, (I), was performed as part of a comprehensive study of lithium salt phase behavior with glyme ligands (Henderson, 2002). LiNO$_3$ tends to be highly associated into contact ion pair or aggregate solvate structures with ether solvents. During the course of preparing a phase diagram of the monoglyme–LiNO$_3$ system, it was found that addition of excess monoglyme to the salt resulted in the rapid formation of long needle-like single crystals of (I). These crystals were structurally characterized to determine the stoichiometry of the crystalline phase which had formed.

(I)
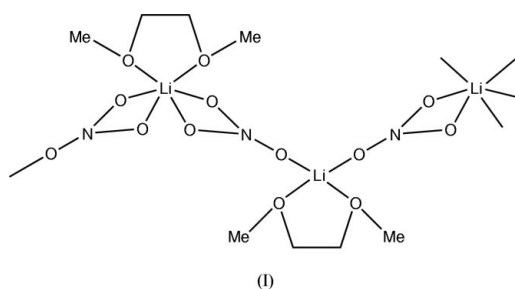
Experimental

Preparations were carried out in a dry room (<1% relative humidity). LiNO$_3$ (Aldrich) was dried at 393 K under high vacuum for 24 h. Anhydrous monoglyme (1,2-dimethoxyethane; 99.5%, Aldrich) was used as received. The title compound was formed by the addition of excess monoglyme to the salt. Single crystals grew rapidly at room temperature. A phase diagram of the monoglyme–LiNO$_3$ system could not be prepared, as heating the mixtures does not result in homogeneous liquids. Rather, the title compound melts and the salt, LiNO$_3$, precipitates out of the mixture.
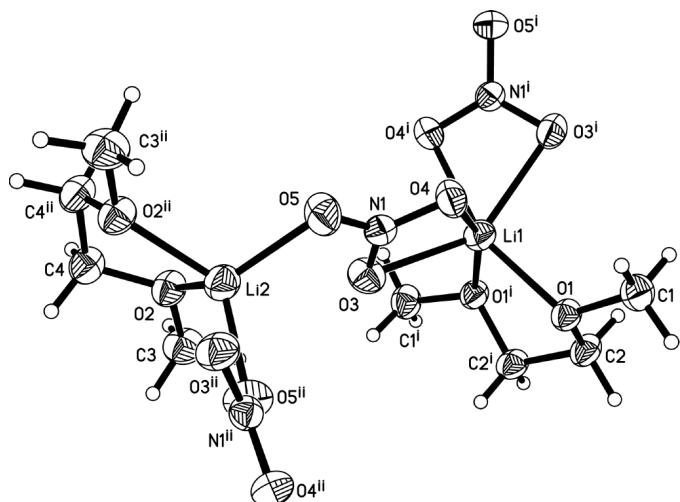
Figure 1
A view of the title compound showing the Li coordination environment and the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x, -x + y, 1/3 - z$; (ii) $y, x, -z$.]

Crystal data

[Li(NO$_3$)(C$_4$H$_{10}$O$_2$)]	Mo $K\alpha$ radiation
$M_r = 159.07$	Cell parameters from 2682
Trigonal, $P3_121$	reflections
$a = 7.4925$ (5) Å	$\theta = 3.1–23.8°$
$c = 23.989$ (3) Å	$\mu = 0.12$ mm^{-1}
$V = 1166.3$ (2) Å^3	$T = 173$ (2) K
$Z = 6$	Block, colourless
$D_x = 1.359$ Mg m^{-3}	$0.44 \times 0.16 \times 0.16$ mm

Data collection

Siemens CCD area-detector diffractometer	1367 independent reflections
φ and ω scans	1255 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Blessing, 1995; Sheldrick, 2000)	$R_{int} = 0.025$
	$\theta_{max} = 25.0°$
	$h = -8 \rightarrow 8$
	$k = -8 \rightarrow 8$
$T_{min} = 0.716$, $T_{max} = 0.846$	$l = -28 \rightarrow 25$
5711 measured reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.025$	$+ 0.0933P]$
$wR(F^2) = 0.060$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
1367 reflections	$\Delta\rho_{max} = 0.10$ e Å^{-3}
104 parameters	$\Delta\rho_{min} = -0.11$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL*97
	Extinction coefficient: 0.0059 (15)

Table 1
Selected geometric parameters (Å).

Li1—O1	2.021 (2)	Li2—O5	1.955 (2)
Li1—O4	2.123 (2)	Li2—O2	1.985 (3)
Li1—O3	2.2138 (14)		

The Friedel pairs were merged because the Flack (1983) parameter [-0.2 (11)] was meaningless due to the lack of atoms with large differences in anomalous scattering terms.
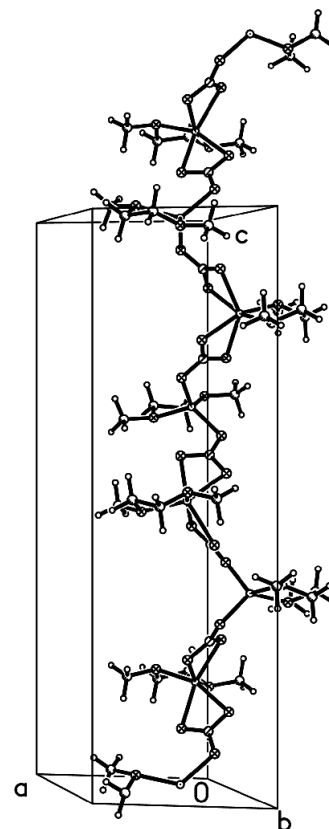

Figure 2
A view of the polymeric structure of the title compound. Key: C shaded, H open, Li dotted, O cross-hatched, and N hatched.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL/PC* and *PLATON* (Spek, 2001).

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