Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Lithium hydrogen iodofumarate monohydrate

Heinrich Billetter, Ingo Pantenburg and Uwe Ruschewitz*

Institut für Anorganische Chemie, Universität zu Köln, Greinstrasse 6, D-50939 Köln, Germany

Correspondence e-mail:
uwe.ruschewitz@uni-koeln.de

Received 18 August 2005
Accepted 19 August 2005
Online 27 August 2005

Key indicators

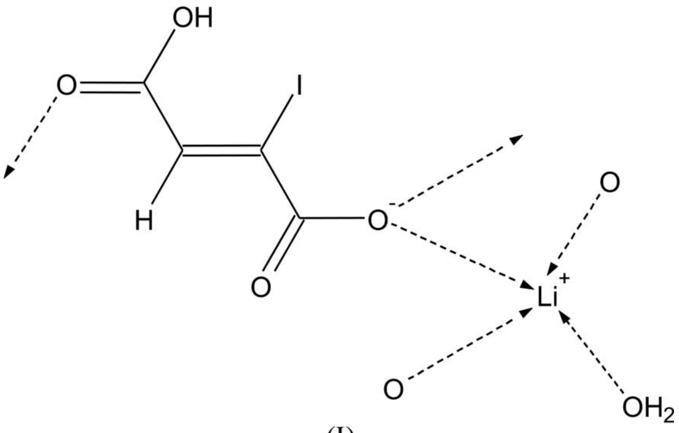
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.034
wR factor = 0.091
Data-to-parameter ratio = 15.4

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

Yellow crystals of the title compound, poly[[aqualithium(I)]-μ_3-iodofumarato], [Li(HOOC—CH═CI—COO)(H$_2$O)]$_n$, crystallized unexpectedly from an aqueous solution containing acetylenedicarboxylic acid and LiI. The lithium cation is coordinated tetrahedrally by four O atoms of three iodofumarate anions and one water molecule. These tetrahedra are connected such that a layered coordination polymer is formed.

Comment

During our ongoing studies of coordination polymers based on acetylenedicarboxylic acid (*e.g.* Billetter *et al.*, 2004) we unexpectedly obtained yellow crystals of the title compound, (I), as a single-phase product. The crystal structure of (I) (Fig. 1) is composed of lithium ions tetrahedrally coordinated by four O atoms stemming from three iodofumarate anions and one water molecule [Li—O = 1.915 (7)–1.977 (8) Å]. Two such tetrahedra share a common edge to form a dimeric unit with Li$\cdots$Li$^{\rm iv}$ = 2.773 (15) Å [symmetry code: (iv) 3 − x, −y, 1 − z]. These dimeric units are connected by the bifunctional iodofumarate anions to form a two-dimensional coordination network. Surprisingly, no inter-layer hydrogen bonds were found so that weak interatomic forces, *i.e.* van der Waals interactions, must be assumed to hold the layers together (Fig. 2).



(I)

It is surprising that (I) was obtained from a solution containing acetylenedicarboxylic acid and LiI. Thus, a *trans* addition of HI to the triple bond of acetylenedicarboxylic acid must have occurred under the conditions of crystallization. We have observed this unusual reaction in a few other experiments, details of which will be published in the near future.

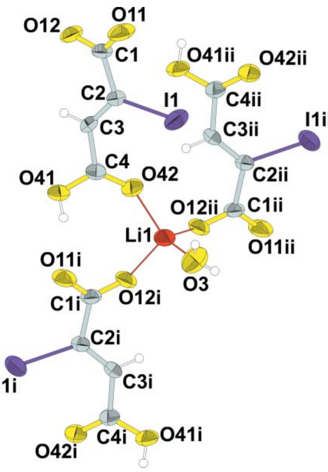


Figure 1
A view of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H-atom radii are arbitrary. [Symmetry codes: (i) $\frac{1}{2} + x, -\frac{1}{2} - y, \frac{1}{2} + z$; (ii) $\frac{5}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.]

Experimental

Acetylenedicarboxylic acid (1.71 g, 15 mmol) was dissolved in deionized water (30 ml). A solution of LiI (1.34 g, 10 mmol) in deionized water (10 ml) was added. The beaker was sealed with a perforated foil. After approximately 100 days, yellow plate-like crystals of (I) were obtained. Analysis calculated for $C_4H_4ILiO_5$ (265.91): C 18.07, H 1.52%; found: C 17.93, H 1.52%. No yield was determined. DTA/TG data show that (I) loses its water molecule at about 363 K. This is followed by a multi-step decomposition at 473 K, which cannot be resolved any further.

Crystal data

$[Li(C_4H_2IO_4)(H_2O)]$
$M_r = 265.91$
Monoclinic, $P2_1/n$
$a = 7.5649$ (15) Å
$b = 6.7833$ (8) Å
$c = 15.502$ (3) Å
$\beta = 101.98$ (2)°
$V = 778.2$ (2) Å^3
$Z = 4$
$D_x = 2.270$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1058 reflections
$\theta = 1.9$–28.2°
$\mu = 4.08$ mm^{-1}
$T = 295$ (2) K
Plate, yellow
0.50 × 0.20 × 0.05 mm

Data collection

Stoe IPDS-I diffractometer
φ scans
Absorption correction: numerical [*X-RED* (Stoe & Cie, 2001); the crystal shape was optimized by *X-SHAPE* (Stoe & Cie, 1999)]
$T_{\min} = 0.202$, $T_{\max} = 0.608$
10677 measured reflections
1740 independent reflections
1523 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.050$
$\theta_{\max} = 28.0°$
$h = -9 \rightarrow 10$
$k = -8 \rightarrow 8$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.091$
$S = 1.08$
1740 reflections
113 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 1.7418P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.12$ e Å^{-3}
$\Delta\rho_{\min} = -1.52$ e Å^{-3}

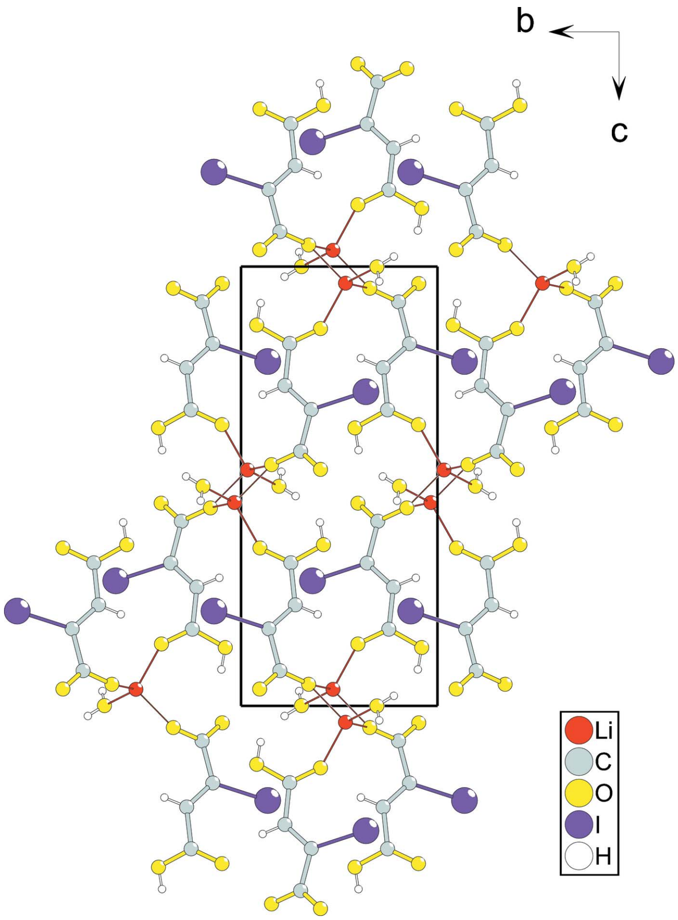


Figure 2
View of the crystal structure of (I) in a projection along [100].

Table 1
Selected geometric parameters (Å, °).

Li1—O42	1.915 (7)	Li1—O12^{ii}	1.952 (8)
Li1—O3	1.917 (10)	Li1—O12^{i}	1.977 (8)
O42—Li1—O3	103.4 (4)	O42—Li1—O12^{i}	112.5 (4)
O42—Li1—O12^{ii}	115.8 (4)	O3—Li1—O12^{i}	117.2 (4)
O3—Li1—O12^{ii}	118.2 (4)	O12^{ii}—Li1—O12^{i}	90.2 (3)

Symmetry codes: (i) $x + \frac{1}{2}, -y - \frac{1}{2}, z + \frac{1}{2}$; (ii) $-x + \frac{5}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O41—H41$\cdots$O11^{i}	0.82 (2)	1.82 (3)	2.618 (4)	164 (6)
O3—H31$\cdots$O11^{iii}	0.81 (2)	2.14 (4)	2.896 (6)	154 (8)

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

The position of H3 of the iodofumarate anion was positioned geometrically and allowed to ride on its parent atom C3, with C—H = 0.93 Å and $U_{\rm iso}$(H) = 0.044 Å^2. The H atoms of the water molecule and the OH group of the iodofumarate anion were located in

m1858 Billetter *et al.* • $[Li(C_4H_2IO_4)(H_2O)]$ *Acta Cryst.* (2005). E**61**, m1857–m1859

difference Fourier maps at the end of the refinement and refined isotropically with restrained O—H distances of 0.82 (2) Å. The highest peak and deepest hole are located 0.72 and 0.83 Å, respectively, from atom I1.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2001); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Gualardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.

Billetter, H., Pantenburg, I. & Ruschewitz, U. (2004). *Z. Naturforsch. Teil B*, **59**, 903–909.

Brandenburg, K. (2001). *DIAMOND*. Release 2.1.e. Crystal Impact GbR, Bonn, Germany.

Sheldrick, G. M. (1997). *SHELXL97*. Release 97-2. University of Göttingen, Germany.

Stoe & Cie (1999). *X-SHAPE*. Version 1.06. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (2001). *X-AREA* (MainMenu Version 1.15) and *X-RED* (Version 1.22). Stoe & Cie GmbH, Darmstadt, Germany.

Acta Cryst. (2005). E**61**, m1857–m1859 Billetter *et al.* • $[Li(C_4H_2IO_4)(H_2O)]$ **m1859**