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Lithium orotate monohydrate

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Key indicators

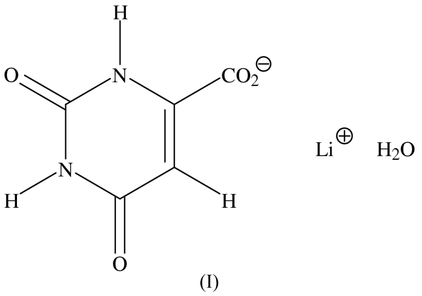
Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.079
Data-to-parameter ratio = 8.4

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

The X-ray crystal structure of the title compound, lithium 1,2,3,6-tetrahydro-2,6-dioxo-4-pyrimidinecarboxylate monohydrate, $Li^+ \cdot C_5H_3N_2O_4^- \cdot H_2O$, was redetermined at a temperature of 110 (2) K. It was now possible to locate all H atoms in the difference Fourier map. The hydrogen-bonding pattern can now be completely described, as well as the coordination mode of the water molecule to the lithium center.

Comment

The room-temperature structure of lithium orotate monohydrate, (I), was first reported by Bach *et al.* (1990). One H atom of the water molecule was missing in that publication, because it could not be located in the difference Fourier synthesis, neither could it be introduced in a calculated position.


(I)

With the present data, measured at a low temperature of 110 (2) K, all H-atom positions could be determined unambiguously from the difference Fourier map (Fig. 1). With this information, the crystal structure can be completely described. The lithium center links the orotate and the water molecules to form a two-dimensional layer which is defined by the base vectors [101] and [011]. Thereby the lithium is in a tetrahedral environment of four O atoms (Fig. 2). Two of these are carbonyl O atoms [Li—O 1.892 (3) and 1.950 (3) Å], one is from the carboxylate group [Li—O 1.881 (3) Å], and the fourth is the water molecule [Li—O 1.973 (4) Å].

The two-dimensional layers formed by the lithium coordination are linked by O—H···O and N—H···O hydrogen bonds (see Table 2) to build up a three-dimensional network. According to the geometrical definition of Jeffrey (1997), H2 and H4 are involved in bifurcated hydrogen bonds with angle sums at these H atoms of 351 and 359°, respectively.

As can be seen from the Li—O bond lengths, the donor ability of the two carbonyl groups is quite different. This effect is a consequence of the hydrogen bonding. While O4 accepts a strong hydrogen bond from a water molecule, O3 is not

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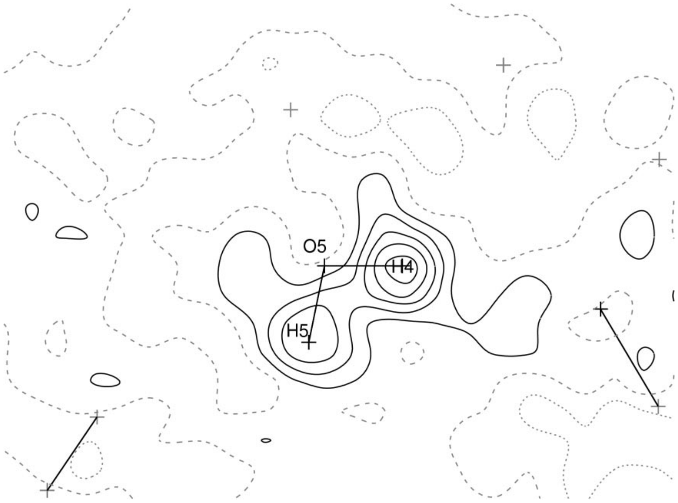

Figure 1
Difference Fourier map in the H_2O plane. Friedel pairs were merged. H4 and H5 were omitted from structure-factor calculations. Contour level: 0.1 e Å^{-3}.

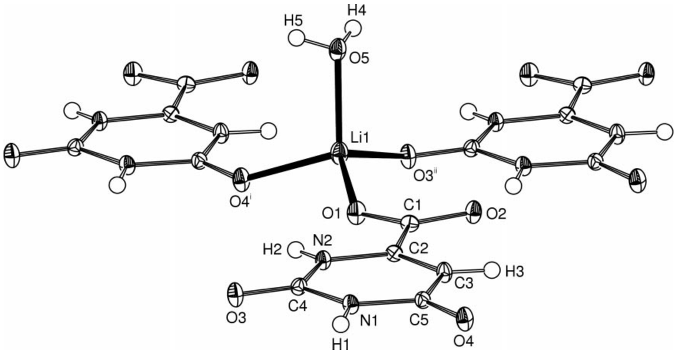

Figure 2
Coordination environment of the Li atom. Displacement ellipsoids drawn at the 50% probability level. [Symmetry codes: (i) x, $y-1$, $z-1$; (ii) $x+1$, $y-1$, z.]

involved in hydrogen bonding. The donor strength of O3 is therefore larger, leading to a shorter Li—O bond. Much smaller, but still significant, is this effect reflected in the C═O double-bond lengths of 1.225 (2) Å for O3 and 1.2425 (19) Å for O4.

The carboxylate O atoms have different environments; while the first one coordinates to the lithium, the second one acts as an acceptor for two nearly linear hydrogen bonds. Surprisingly, this difference is not reflected in the bond distances of C1—O1 1.250 (2) Å and C1—O2 1.252 (2) Å.

The water molecule coordinates to the lithium *via* the tetrahedral 'lone pair' direction. The β-angle between the H_2O plane and the oxygen–metal direction (Ptasiewicz-Bak *et al.*, 1999), amounts to 58.7°. The second 'lone pair' is an acceptor for a hydrogen bond.

Experimental

Lithium orotate monohydrate as obtained from Fluka Chemie AG (Buchs, Switzerland) was dissolved in water. The solution was then evaporated at a temperature of 330 K until crystal formation started.

Crystal data

$Li^{+}\cdot C_5H_3N_2O_4^{-}\cdot H_2O$
$M_r = 180.05$
Triclinic, $P1$
$a = 4.9745$ (2) Å
$b = 5.3035$ (2) Å
$c = 6.7548$ (2) Å
$\alpha = 89.733$ (2)°
$\beta = 102.717$ (2)°
$\gamma = 99.6012$ (13)°
$V = 171.305$ (11) Å^3

$Z = 1$
$D_x = 1.745$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2473 reflections
$\theta = 3.1$–30.0°
$\mu = 0.15$ mm^{-1}
$T = 110$ (2) K
Plate, colourless
0.15 × 0.15 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans with 2° scan width and a crystal–detector distance of 40 mm
4219 measured reflections
994 independent reflections

948 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.050$
$\theta_{\rm max} = 30.0°$
$h = -6 \rightarrow 6$
$k = -7 \rightarrow 7$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.079$
$S = 1.09$
994 reflections
118 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.0193P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.031$
$\Delta\rho_{\rm max} = 0.43$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.250 (2)	O3—Li1^{i}	1.892 (3)
O1—Li1	1.881 (3)	O4—C5	1.2425 (19)
O2—C1	1.252 (2)	O4—Li1^{ii}	1.950 (3)
O3—C4	1.225 (2)	O5—Li1	1.973 (4)
O2—C1—C2—C3	−4.2 (2)		

Symmetry codes: (i) $x-1, 1+y, z$; (ii) $x, 1+y, 1+z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.81	2.10	2.8910 (19)	165
N2—H2···O1	0.80	2.26	2.6326 (18)	109
N2—H2···O5^{ii}	0.80	2.14	2.8941 (19)	157
O5—H4···O1^{iii}	0.80	2.56	2.9738 (17)	114
O5—H4···O4^{iv}	0.80	2.09	2.8797 (17)	168
O5—H5···O2^{v}	0.80	1.91	2.6928 (18)	166

Symmetry codes: (i) $x-1, 1+y, z$; (ii) $x-1, y, z$; (iii) $1+x, y, z$; (iv) $1+x, y-1, z-1$; (v) $x, y, z-1$.

The absolute structure could not be determined reliably. Friedel pairs were therefore merged in the refinement.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL*2000 (Otwinowski & Minor, 1997); data reduction: *HKL*2000 (Otwinowski & Minor, 1997); program(s) used to solve structure: coordinates taken from literature (Bach *et al.*, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001); software used to prepare material for publication: manual editing of *SHELXL*97 output.

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References

Bach, I., Kumberger, O. & Schmidbaur, H. (1990). *Chem. Ber.* **123**, 2267–2271.

Jeffrey, G. A. (1997). *An Introduction to Hydrogen Bonding*. New York: Oxford University Press.

Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.

Ptasiewicz-Bak, H., Olovsson, I. & McIntyre, G. J. (1999). *Acta Cryst.* B**55**, 830–840.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (2001). *PLATON*. Utrecht University, The Netherlands.