Crystal structure of di[tris(hydroxymethylene)methaneammonium] oxalate monohydrate, $(C_4H_{12}NO_3)_2(C_2O_4) \cdot H_2O$

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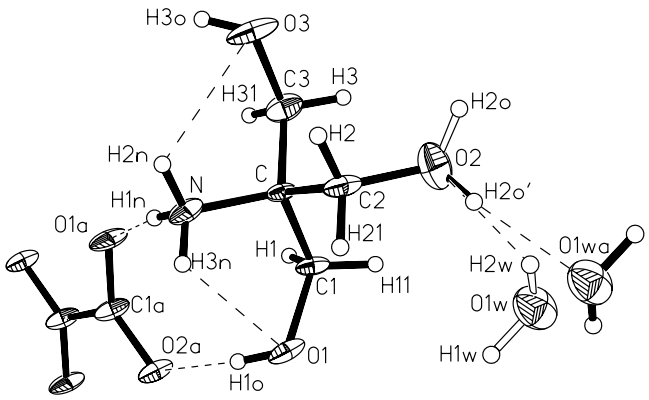

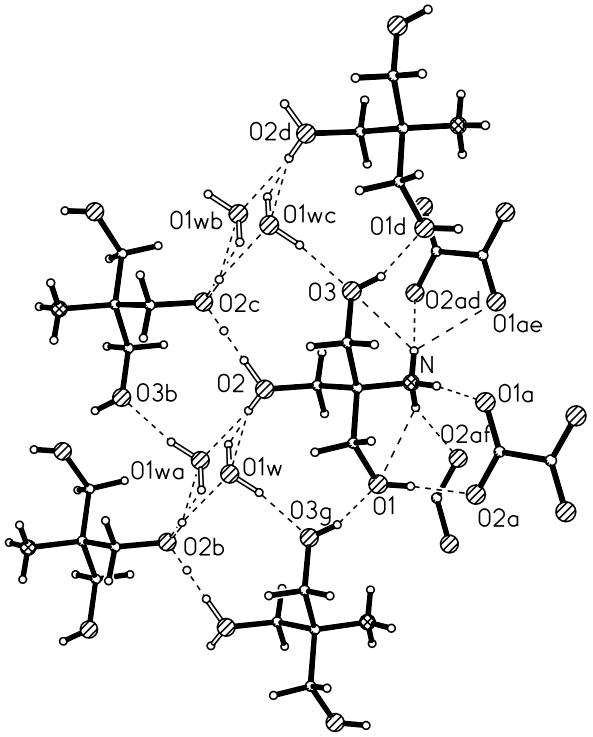

Abstract

$C_{10}H_{26}N_2O_{11}$, triclinic, $P\bar{1}$ (No. 2), $a = 6.235(9)$ Å, $b = 6.429(8)$ Å, $c = 10.92(1)$ Å, $\alpha = 82.16(9)°$, $\beta = 85.3(1)°$, $\gamma = 62.6(1)°$, $V = 384.6$ Å^3, $Z = 1$, $R_{gt}(F) = 0.056$, $wR_{ref}(F^2) = 0.150$, $T = 120$ K.

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Source of material

The title compound was obtained as a main oxidation product of L-ascorbic acid in a solution in the presence of tris(hydroxymethylene)aminomethane (TRIS). The crystallisation was carried out at 277 K from the reaction mixture in water/isopropanol (1:9).

Experimental details

The quality of the intensity data is low because the title compound is purely organic, the crystal studied was plate-like and Mo K_α radiation was used for the data collection. Although the diffraction data were measured at low temperature (120 K), the intensities did not get much stronger, as well as, the disorder was still present in the crystal structure.

Discussion

The TRIS cation is placed in the general position of the crystal lattice, while the oxalate anion C—C bond is lying on the center of symmetry at (1,1/2,1/2), and water molecule is disordered around another symmetry center, (0,1/2,0). The atom labelling is given in the upper figure. $TRIS^+$ adopts an unusual conformation with the torsion angles: ∠N–C–C1–O1 = –41.8(6)°, ∠N–C–C2–O2 = –178.3(5)° and ∠N–C–C3–O3 59.9°, and as a result two intramolecular N–H···O hydrogen bonds are formed. The geometry of intramolecular hydrogen bonds within the TRIS cation is: distances d(N···O1) = 2.716(8) Å and d(N···O3) = 2.862(8) Å; angles ∠N–H3n···O1= 104° and ∠N–H2n···O3 = 99°.

An extensive network of intermolecular hydrogen bonds is observed (lower figure). The most characteristic feature of this network is the presence of two homodromic chains [1] of the O–H···O hydrogen bonds along the [$\bar{1}$10] direction. The flip-flop of the cation O2–H2o and O2–H2o′ bonds and the disorder of the water molecule create antiparallel chains, each of the population of 0.5. The sequences of interactions within the chains are: ···O2b–H2o′···O1w–H2w···O2-H2o···O2c–H2o′···O1wc-, and ···H2o–O2b···H2w–O1wa···H2o′–O2···H2o–O2c.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.05 × 0.35 × 0.55 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.37 cm^{-1}
Diffractometer, scan mode:	KM4, $\omega/2\theta$
$2\theta_{max}$:	42.14°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1662, 831
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 435
$N(param)_{refined}$:	115
Programs:	SHELXS-97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1o)	2*i*		0.4825	0.5845	0.3555	0.027
H(2o)	2*i*	0.50	0.2829	0.2613	0.0507	0.051
H(2o′)	2*i*	0.50	0.4751	0.0616	0.0225	0.051
H(3o)	2*i*		1.0696	–0.2739	0.2583	0.043
H(1n)	2*i*		0.7818	0.1484	0.4047	0.05(3)
H(2n)	2*i*		0.7182	–0.0390	0.3935	0.07(3)
H(3n)	2*i*		0.5297	0.1962	0.4115	0.12(5)
H(1)	2*i*		0.7406	0.4577	0.2160	0.021
H(11)	2*i*		0.5370	0.4949	0.1272	0.021
H(2)	2*i*		0.4936	–0.0330	0.2196	0.027
H(21)	2*i*		0.3034	0.2274	0.2365	0.027
H(3)	2*i*		0.8855	0.0762	0.0904	0.028
H(31)	2*i*		1.0196	0.0653	0.2087	0.028
H(1w)	2*i*	0.50	0.0072	0.6011	0.0954	0.054
H(2w)	2*i*	0.50	0.0538	0.3881	0.0341	0.054

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*i*		0.4168(7)	0.5793(7)	0.2947(4)	0.013(2)	0.010(2)	0.039(3)	–0.001(2)	–0.005(2)	–0.001(2)
O(2)	2*i*		0.4298(8)	0.1825(8)	0.0680(4)	0.040(3)	0.050(3)	0.027(3)	–0.027(3)	–0.006(2)	–0.007(3)
O(3)	2*i*		0.9642(8)	–0.2144(7)	0.2062(5)	0.017(3)	0.013(3)	0.066(4)	0.001(2)	0.004(3)	–0.004(2)
N	2*i*		0.673(1)	0.1146(9)	0.3760(5)	0.022(3)	0.007(3)	0.031(4)	–0.005(3)	–0.004(3)	0.002(3)
C	2*i*		0.657(1)	0.1781(9)	0.2404(6)	0.007(3)	0.008(4)	0.024(4)	–0.002(3)	–0.001(3)	–0.005(3)
C(1)	2*i*		0.595(1)	0.440(1)	0.2110(6)	0.009(4)	0.012(4)	0.029(4)	–0.002(3)	0.004(3)	–0.006(3)
C(2)	2*i*		0.455(1)	0.131(1)	0.1961(6)	0.014(4)	0.013(4)	0.034(5)	0.000(3)	0.001(4)	–0.007(3)
C(3)	2*i*		0.897(1)	0.033(1)	0.1792(6)	0.020(4)	0.015(4)	0.034(5)	–0.009(3)	0.003(3)	0.001(3)
C(1a)	2*i*		0.910(1)	0.470(1)	0.4707(6)	0.016(4)	0.008(4)	0.037(5)	–0.004(3)	0.000(3)	–0.001(3)
O(1a)	2*i*		0.9878(7)	0.2608(7)	0.4491(4)	0.017(3)	0.008(3)	0.047(3)	–0.003(2)	–0.010(2)	–0.007(2)
O(2a)	2*i*		0.7016(8)	0.6375(7)	0.4461(4)	0.012(2)	0.011(2)	0.038(3)	–0.002(2)	–0.005(2)	–0.003(2)
O(1w)	2*i*	0.50	0.041(2)	0.535(2)	0.024(1)	0.036(7)	0.047(7)	0.041(8)	–0.024(6)	0.001(6)	–0.015(6)

References

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