

5206 measured reflections
 5164 independent reflections
 4646 reflections with
 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

3 standard reflections
 every 200 reflections
 frequency: 120 min
 intensity decay: none

Refinement

Refinement on F^2
 $R(F) = 0.058$
 $wR(F^2) = 0.157$
 $S = 1.054$
 5133 reflections
 422 parameters
 H atoms riding
 $w = 1/[\sigma^2(F_o^2) + (0.115P)^2 + 1.50P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.003$
 $\Delta\rho_{\text{max}} = 0.445 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.222 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)
 Absolute structure: Flack
 (1983)
 Flack parameter = $-0.1(3)$

Based on the systematic absences of hkl , $h + k = 2n + 1$, packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be C2 (No. 5). Friedel pairs (2467) were collected to a θ_{max} of 60° and were not merged. A methoxymethyl group attached to C6 was disordered and its atoms were allowed to refine over two locations, C22 and C22A, with site-occupancy factors of 0.50. The phenyl ring was refined as a regular hexagon. The H atoms were included at geometrically idealized positions with C—H and O—H distances of 0.95 Å. A half molecule of methanol solvate per asymmetric unit was found close to a twofold axis and was disordered; its H atoms were ignored. The non-H atoms of chasmaconitine were given anisotropic displacement parameters, while the methanol C and O atoms were given isotropic displacement parameters.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI91* (Fan, 1991). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976) in *TEXSAN*. Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1327). Services for accessing these data are described at the back of the journal. A packing diagram has also been deposited.

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Acta Cryst. (1998). **C54**, 238–240

Citraconic Acid

ELAINE BATCHELOR AND WILLIAM JONES

Department of Chemistry, University of Cambridge, Lensfield Road, Cambridge CB2 1EW, England. E-mail: eb205@cus.cam.ac.uk

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Abstract

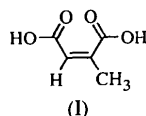
Molecules of the title compound, C₅H₆O₄, form extended tapes via O—H···O interactions. The tapes assemble via C—H···O interactions giving rise to planar sheets.

Comment

Carboxylic acids have been used successfully in the supramolecular design of organic crystalline solids (Pedireddi *et al.*, 1996; Zhao *et al.*, 1990). Their potential to form strong and predictable hydrogen-bonded motifs is particularly useful. Leiserowitz (1976) reported that carboxylic acids predominantly interact via a centrosymmetric cyclic motif.

Molecules of the title compound, (I), assemble via two crystallographically distinct cyclic hydrogen-bond motifs [O1···O2 2.609(3) Å and O1···H2O—O2(1-x, 1-y, 1-z) 175(2)^o; O3···O4 2.714(3) Å and O3···H4O—O4(-x, 1-y, -z) 175(1)^o] giving rise to an extended molecular tape. The C—O bond lengths for the O1—C1—O2 carboxyl group [C=O 1.260(3) and C—O 1.270(3) Å] lie outside the expected ranges

for such bonds and indicate a degree of disorder (Dunitz & Strickler, 1968). The dihedral angle between the planes of the acid groups (O1, O2, C1, C2 and O3, O4, C4, C3) is $81.4(1)^\circ$. Hence, no intramolecular hydrogen bond is observed. This is in contrast, for example, to maleic acid (James & Williams, 1974).



The tapes are joined by C—H...O interactions [$C2 \cdots O1$ 3.314(4) Å and $C2-H2 \cdots O1$ 159.7(1) $^\circ$] to form sheets parallel to the $(\bar{1}01)$ planes (Fig. 2). A similar packing arrangement is observed in maleic acid (James & Williams, 1974) in which it is likely that C—H...O interactions facilitate edge-to-edge packing of molecular tapes to give a planar sheet structure.

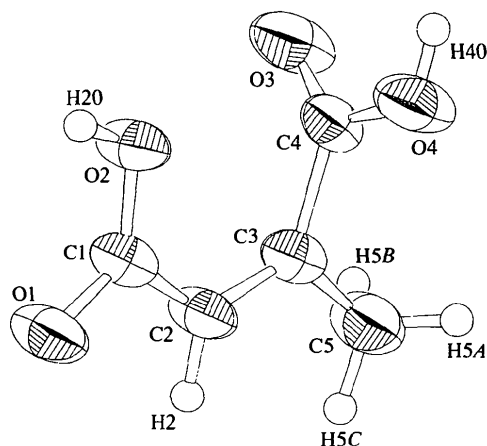


Fig. 1. The structure of (I) showing 50% probability displacement ellipsoids.

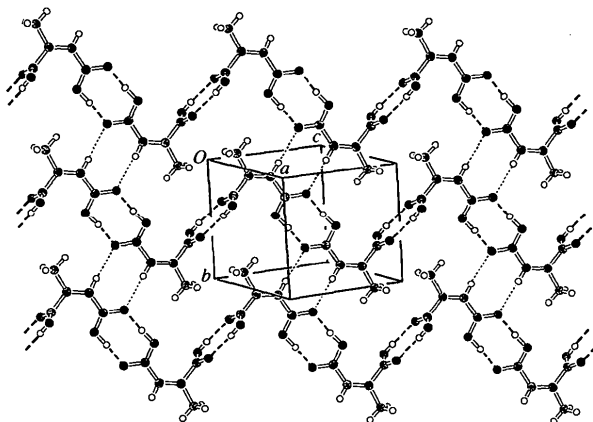


Fig. 2. View of the $(\bar{1}01)$ plane. Hydrogen-bonded cyclic motifs lead to an extended α network, which in turn is joined together by C—H...O interactions forming the sheet as shown.

Experimental

The sample of (I) was obtained from the Sigma-Aldrich Company Ltd. Single crystals were grown by slow evaporation from a solution of ethyl acetate.

Crystal data

$C_5H_6O_4$
 $M_r = 130.10$
 Triclinic
 $P\bar{1}$
 $a = 5.4239(5)$ Å
 $b = 7.1863(9)$ Å
 $c = 8.1732(8)$ Å
 $\alpha = 88.860(9)^\circ$
 $\beta = 71.254(8)^\circ$
 $\gamma = 81.261(9)^\circ$
 $V = 298.03(5)$ Å³
 $Z = 2$
 $D_x = 1.450$ Mg m⁻³
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 25 reflections
 $\theta = 15-20^\circ$
 $\mu = 0.128$ mm⁻¹
 $T = 295(2)$ K
 Rod
 $0.30 \times 0.10 \times 0.05$ mm
 Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: none
 1134 measured reflections
 1054 independent reflections
 685 reflections with $I > 2\sigma(I)$

$R_{int} = 0.039$
 $\theta_{max} = 24.95^\circ$
 $h = -6 \rightarrow 6$
 $k = -8 \rightarrow 8$
 $l = 0 \rightarrow 9$
 3 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.157$
 $S = 0.973$
 1054 reflections
 93 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0241P)^2 + 0.6395P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.011$
 $\Delta\rho_{max} = 0.219$ e Å⁻³
 $\Delta\rho_{min} = -0.211$ e Å⁻³
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C1	1.260(3)	C1—C2	1.460(4)
O2—C1	1.270(3)	C2—C3	1.323(4)
O3—C4	1.205(4)	C3—C5	1.493(5)
O4—C4	1.293(4)	C3—C4	1.503(4)
O1—C1—C2—C3	-173.8(3)	C2—C3—C4—O3	-98.3(4)
O2—C1—C2—C3	5.3(5)	C2—C3—C4—O4	85.9(4)

The structure was solved by direct methods and refined using full-matrix least-squares refinement on F^2 . The olefinic and methyl H atoms were refined using a riding model with the H atoms positioned geometrically with respect to the parent atom. The acidic H atoms were found by inspection of the difference Fourier map. Isotropic displacement parameters were used in the refinement of all H atoms.

Data collection: *CAD-4 Manual* (Enraf-Nonius, 1988). Cell refinement: *SETANG* in *CAD-4 Manual*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Pro-

gram(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1205). Services for accessing these data are described at the back of the journal.

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Cyclothreonine at 122 K

UFFE ANTHONI,^a TINE MUXOLL FATUM^a AND CLAUDIUS FLENSBURG^b

^aDepartment of Chemistry, University of Copenhagen, Universitetsparken 5, DK-2100 Copenhagen, Denmark, and ^bCentre for Crystallographic Studies, Department of Chemistry, University of Copenhagen, Universitetsparken 5, DK-2100 Copenhagen, Denmark. E-mail: claus@laue.ki.ku.dk

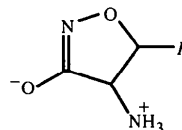
(Received 24 April 1997; accepted 17 October 1997)

Abstract

Spectroscopic and crystallographic data for the title compound (4*S*,5*R*)-4-amino-5-methyl-3-isoxazolidinone are reported. The title compound crystallizes with one half equivalent of water in a zwitterionic structure, C₄H₈N₂O₂·0.5H₂O, stabilized by intermolecular hydrogen bonds.

Comment

Cyclothreonine has been characterized as part of a study of the siderophore pseudomonine, isolated from iron-deficient cultures of *Pseudomonas fluorescens* (Anthoni *et al.*, 1995). The stereochemistry of cyclothreonine determined from ¹H NMR spectra (D₂O) was discussed by Turchin (1971), who stated that the coupling constant between the two H atoms connected to the five-membered ring (H4 and H5) would be 7.0 Hz in a *cis* conformation and 8.7 Hz in a *trans* conformation. However, this has not been confirmed by independent methods, so an investigation using X-ray diffraction analysis was initiated. The structure of an analogous compound, cycloserine (4-amino-3-isoxazolidinone), has been examined by IR spectroscopy (Preti & Tosi, 1979), derivatization and potentiometric studies (Hidy *et al.*, 1955). It was deduced that cycloserine exists as a zwitterion in aqueous solution and in the solid state. An early study of the crystal structure of cycloserine hydrochloride has been reported (Turley & Pepinsky, 1957).



R = H, cycloserine
R = CH₃, cyclothreonine

From Fig. 1 and the geometric parameters in Table 2, it can be seen that the methyl and amino groups are *cis* with respect to each other confirming the suggestions of Turchin (1971). The amino group is protonated giving rise to a zwitterionic structure, as in cycloserine. The isoxazolidinone ring adopts an envelope conformation, the O1, N2, C3, O6 and C4 atoms being coplanar [r.m.s. deviation of the least-squares plane is 0.01 Å and

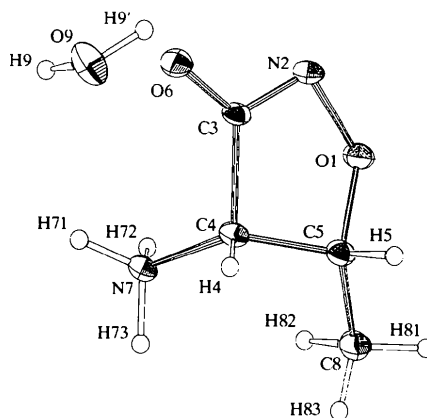


Fig. 1. View of cyclothreonine showing the labelling of the atoms. Displacement ellipsoids are shown at the 50% probability level. H atoms are drawn as spheres of fixed radii.