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Acta Cryst. (1995). C51, 2718–2720

L-Glycyl-L-Glutamine Monohydrate†

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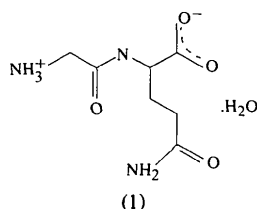
(Received 2 February 1995; accepted 6 July 1995)

Abstract

The crystal structure of C₇H₁₃N₃O₄·H₂O is stabilized by a three-dimensional network of N—H···O and O—H···O hydrogen bonds. The peptide unit exists in a *trans* conformation.

Comment

The investigation of the title compound (1) was undertaken as part of a project to determine the structures of peptides and other compounds containing the peptide linkage, to determine the conformation and crystal packing, and confirm the stereochemistry.



A view of the molecule with our numbering scheme is shown Fig. 1. The dimensions of the peptide group are in good agreement with the average values of peptide dimensions (Ramanadham & Chidambaram, 1978). The molecule exists as a zwitterion with the N atom (N1) of glycine protonated as NH₃⁺ and the carboxyl group of the glutamine residue as ionized COO⁻. The peptide unit is in a *trans* [$\omega = -179.6(2)^\circ$] conformation and the peptide unit is planar within 0.002(1) Å. The carboxyl group is also planar within 0.004(1) Å and makes a dihedral angle of 57.5(1)° with the adjacent peptide unit. The backbone torsion angles (Table 2) indicate the

molecule is in a folded conformation. The side-chain torsion angles χ_1 , χ_2 , χ_{31} and χ_{32} of the glutamine residue are 168.3(2), 70.9(3), $-3.0(3)$ and 177.2(2)°, respectively. These values correspond to the lowest energy conformation of the glutamine side chain (Edsall *et al.*, 1966).

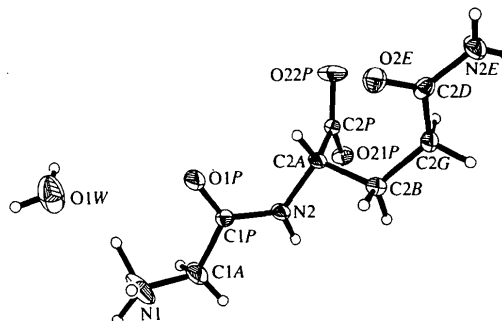


Fig. 1. The molecular structure of the title compound with the atom-labelling scheme and 50% probability displacement ellipsoids.

The molecular packing viewed down the *a* axis is shown in Fig. 2. The hydrogen-bonding scheme is given in Table 3. The NH₃⁺ group of the glycyl residue is hydrogen bonded to O1P, symmetry-related O1P, O21P and water oxygen (O1W). One of the H atoms (H1) is involved in bifurcated hydrogen bonds. The peptide N atom (N2) forms an N—H···O hydrogen bond of 2.943(3) Å with a translated (*a* axis) O22P atom. The water molecule (O1W) forms O—H···O hydrogen bonds with symmetry-related O2E and

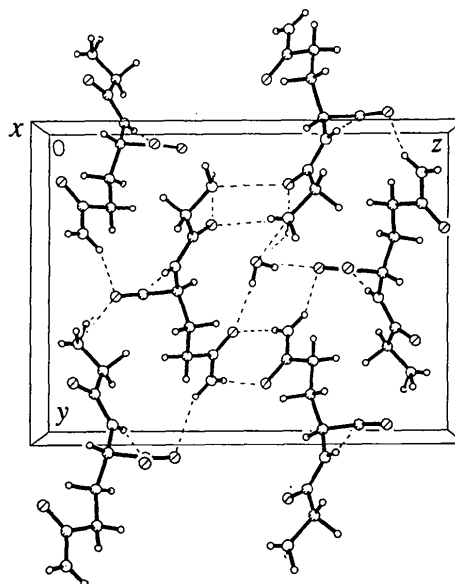


Fig. 2. A perspective drawing of the packing arrangement (011) with dashed lines indicating the hydrogen-bonding scheme.

† Contribution No. 1325 of the Instituto de Química, UNAM.

O21P atoms at distances of 2.861 (3) and 2.772 (3) Å, respectively (Allen, Kennard & Taylor, 1983). An interesting point is that this peptide structure forms a head-to-tail arrangement through N—H...O hydrogen bonds along the direction of the *b* axis (Fig. 2).

Experimental

The title compound was recrystallized from water at room temperature.

Crystal data

C₇H₁₃N₃O₄·H₂O

M_r = 221.22

Orthorhombic

P2₁2₁2₁

a = 5.415 (2) Å

b = 11.618 (5) Å

c = 15.558 (9) Å

V = 978.8 (8) Å³

Z = 4

D_x = 1.501 Mg m⁻³

Cu Kα radiation

λ = 1.54178 Å

Cell parameters from 25 reflections

θ = 15–45°

μ = 1.094 mm⁻¹

T = 293 (2) K

Needle

0.4 × 0.2 × 0.2 mm

Colourless

Data collection

P4 diffractometer

θ/2θ scans

Absorption correction:

none

1146 measured reflections

1017 independent reflections

1011 observed reflections

[*I* > 2σ(*I*)]

*R*_{int} = 0.0192

θ_{max} = 56.72°

h = -1 → 5

k = -1 → 12

l = -1 → 16

3 standard reflections monitored every 100 reflections

intensity decay: 2%

Refinement

Refinement on *F*²

R[*F*² > 2σ(*F*²)] = 0.0340

wR(*F*²) = 0.0898

S = 1.052

1017 reflections

196 parameters

All H atoms refined isotropically

w = 1/[σ²(*F*_o²) + (0.0797*P*)² + 0.0888*P*]

where *P* = (*F*_o² + 2*F*_c²)/3
(Δ/σ)_{max} = 0.154

Δρ_{max} = 0.163 e Å⁻³

Δρ_{min} = -0.197 e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Absolute configuration:

Flack (1983) parameter = -0.1 (3)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
N1	0.5693 (6)	0.1944 (3)	0.4106 (2)	0.0522 (9)
C1A	0.5362 (5)	0.2842 (2)	0.3491 (2)	0.0300 (7)
C1P	0.2990 (4)	0.3502 (2)	0.36250 (13)	0.0174 (5)
O1P	0.1313 (3)	0.31040 (15)	0.40638 (11)	0.0321 (5)
N2	0.2894 (5)	0.4495 (2)	0.32012 (13)	0.0202 (5)
C2A	0.0792 (5)	0.5281 (2)	0.32375 (14)	0.0172 (5)
C2B	0.1780 (5)	0.6460 (2)	0.35417 (15)	0.0210 (6)
C2G	0.0034 (5)	0.7471 (2)	0.34601 (15)	0.0208 (6)
C2D	-0.2124 (4)	0.7470 (2)	0.40779 (12)	0.0198 (5)

N2E	-0.3593 (5)	0.8387 (2)	0.40291 (15)	0.0291 (6)
O2E	-0.2512 (3)	0.66808 (12)	0.45907 (9)	0.0271 (5)
C2P	-0.0449 (4)	0.5382 (2)	0.23465 (15)	0.0183 (6)
O21P	0.0969 (3)	0.54403 (13)	0.17026 (9)	0.0233 (5)
O22P	-0.2737 (4)	0.5424 (2)	0.23150 (11)	0.0356 (5)
O1W	0.1587 (5)	0.0621 (2)	0.48354 (13)	0.0501 (7)

Table 2. Selected geometric parameters (Å, °)

N1—C1A	1.428 (4)	C2B—C2G	1.514 (4)
C1A—C1P	1.510 (4)	C2G—C2D	1.513 (3)
C1P—O1P	1.227 (3)	C2D—O2E	1.234 (3)
C1P—N2	1.330 (3)	C2D—N2E	1.331 (3)
N2—C2A	1.460 (3)	C2P—O22P	1.241 (3)
C2A—C2B	1.544 (3)	C2P—O21P	1.264 (3)
C2A—C2P	1.545 (4)		
N1—C1A—C1P	112.6 (2)	C2G—C2B—C2A	116.5 (2)
O1P—C1P—N2	125.0 (2)	C2D—C2G—C2B	115.4 (2)
O1P—C1P—C1A	121.1 (2)	O2E—C2D—N2E	122.0 (2)
N2—C1P—C1A	113.9 (2)	O2E—C2D—C2G	122.9 (2)
C1P—N2—C2A	123.7 (2)	N2E—C2D—C2G	115.1 (2)
N2—C2A—C2B	107.2 (2)	O22P—C2P—O21P	125.0 (2)
N2—C2A—C2P	110.6 (2)	O22P—C2P—C2A	118.2 (2)
C2B—C2A—C2P	111.0 (2)	O21P—C2P—C2A	116.8 (2)
N1—C1A—C1P—O1P	-17.5 (4)		
N1—C1A—C1P—N2	165.2 (3)	ψ ₁	
O1P—C1P—N2—C2A	3.2 (3)		
C1A—C1P—N2—C2A	-179.6 (2)	ω ₁	
C1P—N2—C2A—C2B	123.6 (2)		
C1P—N2—C2A—C2P	-115.3 (2)	φ ₂	
N2—C2A—C2B—C2G	168.3 (2)	χ ₁	
C2P—C2A—C2B—C2G	47.4 (3)		
C2A—C2B—C2G—C2D	70.9 (3)	χ ₂	
C2B—C2G—C2D—O2E	-3.0 (3)	χ ₃₁	
C2B—C2G—C2D—N2E	177.2 (2)	χ ₃₂	
N2—C2A—C2P—O22P	140.3 (2)	ψ ₂₂	
C2B—C2A—C2P—O22P	-100.8 (3)		
N2—C2A—C2P—O21P	-41.1 (3)	ψ ₂₁	
C2B—C2A—C2P—O21P	77.8 (2)		

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...O1P	1.16 (10)	2.29 (9)	2.729 (3)	97 (2)
N1—H1...O1W	1.16 (10)	1.98 (9)	2.931 (3)	137 (2)
N1—H2...O21P ⁱ	1.03 (6)	1.84 (5)	2.811 (3)	156 (2)
N1—H3...O1P ⁱⁱ	0.61 (4)	2.28 (4)	2.867 (3)	161 (2)
N2—H6...O22P ⁱⁱⁱ	0.79 (4)	2.15 (4)	2.943 (3)	175 (3)
N2E—N12...O21P ^{iv}	0.88 (4)	2.23 (4)	3.001 (3)	146 (2)
N2E—H13...O2E ^v	0.83 (4)	2.21 (3)	3.020 (3)	168 (2)
O1W—H14...O2E ^{vi}	0.82 (4)	2.05 (4)	2.861 (3)	168 (2)
O1W—H15...O21P ^{vi}	0.86 (5)	1.93 (4)	2.772 (3)	166 (2)

Symmetry codes: (i) 1 - *x*, *y* - ½, ½ - *z*; (ii) ½ + *x*, ½ - *y*, 1 - *z*; (iii) 1 + *x*, *y*, *z*; (iv) -*x*, ½ + *y*, ½ - *z*; (v) *x* - ½, ½ - *y*, 1 - *z*; (vi) -*x*, *y* - ½, ½ - *z*.

Data collection: P4 diffractometer software. Cell refinement: XSCANS (Siemens, 1992). Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL-Plus (Sheldrick, 1991). Software used to prepare material for publication: SHELXL93.

KP acknowledges the CONACYT (Cátedra Patrimonial Nivel II) for fellowship assistance. We thank the Instituto de Biotecnología, UNAM, for data collection.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the IUCr (Reference: FG1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 2720–2722

Xanthyletin†

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(Received 12 May 1995; accepted 7 July 1995)

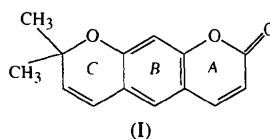
Abstract

The present X-ray diffraction study establishes the molecular structure of the title compound, 8,8-dimethyl-2*H*,8*H*-benzo[1,2-*b*:5,4-*b'*]dipyran-2-one, C₁₄H₁₂O₃. The molecule consists of three six-membered rings. Ring C has a screw-boat conformation, whereas rings A and B are planar. The crystal structure is stabilized by van der Waals forces and there are three C—H···O intermolecular interactions.

† Contribution No. 1326 of the Instituto de Química, UNAM.

Comment

Xanthyletin, (I), was isolated from the roots of *Stauranthus perforatus* (*Rutaceae*) and collected in Felipe Carrillo Puerto, Quintana Roo, México. The roots are used locally for the treatment of rheumatism. The preparation of crystals of (I) is described in the *Experimental* section.



A view of the title molecule with the atom-numbering scheme is shown in Fig. 1. The molecule consists of three six-membered rings A (C1–C4, C12, O1), B (C4–C6, C10–C12) and C (C6–C9, O2, C10). Bond lengths and angles are quite similar to those of related compounds such as amyrolin (Kato, 1970). Rings A and

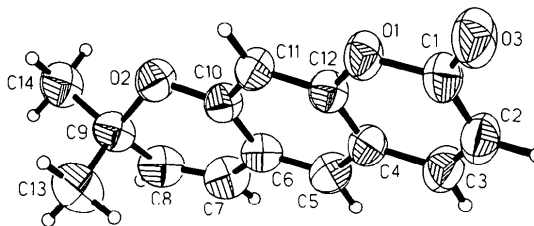


Fig. 1. The molecular structure of the title compound showing the atom-labelling scheme and 50% probability displacement ellipsoids.

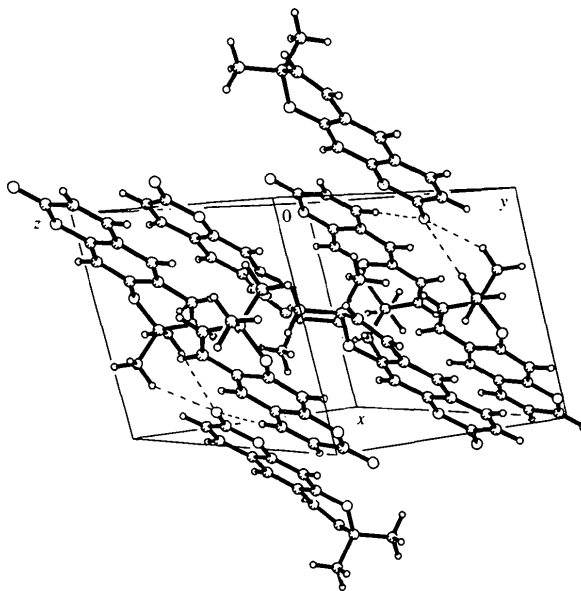


Fig. 2. A unit-cell drawing of the packing arrangement with dashed lines indicating C—H···O intermolecular interactions.