C(22)—C(23)—C(24)	119.7 (11)	120.2 (11)
C(23)—C(24)—C(25)	121.3 (12)	121.0 (11)
C(24)—C(25)—C(26)	118.1 (12)	119.1 (11)
C(1)—C(2)—C(3)—C(4)	−4.6 (7)	−0.8 (8)
C(1)—C(2)—C(11)—C(12)	−39.4 (9)	46.3 (9)
C(1)—C(2)—C(11)—C(16)	138.8 (12)	−132.8 (12)
C(1)—C(2)—C(3)—C(21)	172.2 (13)	−179.3 (13)
C(2)—C(3)—C(21)—C(22)	−43.3 (10)	34.0 (10)
C(2)—C(3)—C(21)—C(26)	136.2 (14)	−146.7 (14)
C(3)—C(2)—C(11)—C(12)	148.4 (13)	−140.1 (14)
C(3)—C(2)—C(11)—C(16)	−33.4 (9)	40.8 (10)
C(4)—C(3)—C(2)—C(11)	168.4 (12)	−174.8 (13)
C(4)—C(3)—C(21)—C(22)	133.1 (12)	−144.5 (13)
C(11)—C(2)—C(3)—O(2)	171.1 (10)	−174.8 (11)
C(11)—C(2)—C(3)—C(1)	173.0 (12)	−174.2 (14)
C(11)—C(2)—C(3)—C(21)	−14.8 (8)	6.5 (9)

Intensity data were corrected for Lorentz and polarization effects. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and refined by full-matrix least squares using *SHELX76* (Sheldrick, 1976), with anisotropic displacement factors for all the non-H atoms. All the H atoms were located from difference Fourier maps and were refined isotropically in the final cycles.

All computations were performed using a MicroVAX 3400 computer at the Chungnam National University. Data collection: *CAD-4 Software* (Enraf–Nonius, 1985). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Molecular graphics: *ORTEP*II (Johnson, 1971).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data, and a stereo packing diagram have been deposited with the IUCr (Reference: TA1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Anhydrous DL-Glutamic Acid

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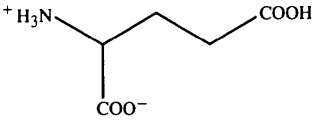
Abstract

The conformation of the glutamic acid molecule, $C_5H_9NO_4$, in the crystalline racemate differs from that observed previously in the two known chiral modifications. All three forms have a short O···O hydrogen bond between the side-chain carboxylic acid and the ionized carboxylate of another molecule.

Comment

There seems to be a persistent legend that DL-glutamic acid crystallizes only as a conglomerate of D- and L-crystals (for example, see Prelog, 1991). This may appear to be supported by the fact that the anhydrous crystalline racemate has not so far been described whereas crystal structures are known for two chiral forms: α-form, $P2_12_12_1$, a = 10.282, b = 8.779, c = 7.068 Å (Lehmann & Nunes, 1980); β-form, $P2_12_12_1$, a = 5.159, b = 17.30, c = 6.948 Å (Lehmann, Koetzle & Hamilton, 1972). According to Dunn & Stoddart (1937), crystallization of aqueous solutions of DL-glutamic acid can yield several anhydrous forms as well as a monohydrate, often crystallizing together out of the same solution. Some of these forms may not have been definitely identified but they include the known chiral forms (Sakata, Susuki & Takenouchi, 1962). In the course of our study of the stability of racemic crystals compared with their chiral counterparts (Brock, Schweizer & Dunitz, 1991) we became interested in the question of whether DL-glutamic acid was indeed obtainable or not. A commercial sample of DL-glutamic acid was examined and found to contain a mixture of small needles and plates corresponding probably to the monohydrate and the anhydrous form described by Dunn & Stoddart (1937). Recrystallization from ethanol gave plate-formed crystals that were found to correspond to the long-sought racemate, the structure of which is described here.



DL-Glutamic acid

It turns out that the conformation of the side chain is different in the three crystal structures (α, β and racemate; Table 3) which do, however, share the common feature that the OH group of the side-chain carboxylic acid forms a short hydrogen bond to the carboxylate anion of a neighbouring molecule [O(4)···O(2) 2.525, O(4)—H···O(2) 1.54 Å, 174° in the racemate; 2.581, 1.57 Å, 169°, respectively, in the α-form; 2.519, 1.48 Å, 172°, respectively, in the β-form]. A detailed discussion of this type of interaction has been given by Lehmann & Nunes (1980). The shortest intermolecular contacts of the other O atoms in the racemate are O(1)···N(1) 2.829 Å [O(1)···H(1)—N(1) 1.83 Å, 167°] and O(3)···N(1) 2.953 Å [O(3)···H(2)—N(1) 2.38 Å, 117°].

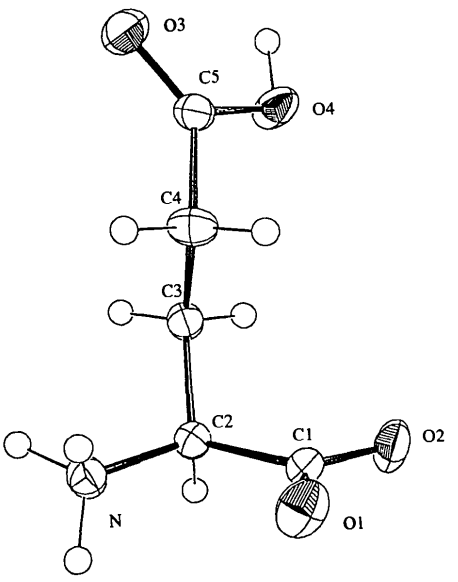


Fig. 1. The glutamic acid molecule in the crystalline racemate, showing 50% probability displacement ellipsoids (*ORTEP*; Johnson, 1965).

Experimental

A sample of DL-glutamic acid (Sigma Chemical Co.) containing a mixture of anhydrous and monohydrate crystals was warmed in ethanol; the solution was separated from the undissolved material and seeded with a few plate-shaped crystals of the original sample. By slow evaporation of the ethanol, the plate-shaped crystals were obtained.

Crystal data

$C_5H_9NO_4$
$M_r = 147.13$
Monoclinic
$P2_1/n$
$a = 5.743\ (1)$ Å
$b = 13.036\ (2)$ Å
$c = 8.431\ (3)$ Å
$\beta = 103.66\ (2)°$
$V = 613.3$ Å^3
$Z = 4$
$D_x = 1.593$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 18 reflections
$\theta = 8.9$–$14.7°$
$\mu = 0.139$ mm^{-1}
$T = 293\ (2)$ K
Plate
0.15 × 0.15 × 0.07 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1475 measured reflections
1475 independent reflections
853 observed reflections [$I > 3\sigma(I)$]
$\theta_{max} = 27.93°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 17$
$l = -11 \rightarrow 10$
2 standard reflections
frequency: 10 000 s
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 3\sigma(F^2)] = 0.0380$
$wR(F^2) = 0.1132$
$S = 1.195$
853 reflections
128 parameters
All H-atom parameters refined
$w = \{\exp[2(\sin\theta/\lambda)^2]\}/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.114$
$\Delta\rho_{max} = 0.238$ e Å^{-3}
$\Delta\rho_{min} = -0.175$ 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)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
C(1)	0.1653 (4)	−0.0412 (2)	0.7793 (3)	0.0218 (5)
C(2)	0.2642 (4)	0.0477 (2)	0.6944 (3)	0.0214 (5)
C(3)	0.1322 (4)	0.1481 (2)	0.7034 (3)	0.0224 (5)
C(4)	0.1248 (5)	0.1815 (2)	0.8759 (3)	0.0270 (6)
C(5)	−0.0259 (4)	0.2769 (2)	0.8733 (3)	0.0227 (5)
N(1)	0.5264 (4)	0.0587 (2)	0.7647 (3)	0.0262 (5)
O(1)	0.2987 (4)	−0.08649 (15)	0.8952 (2)	0.0343 (5)
O(2)	−0.0528 (3)	−0.06025 (14)	0.7177 (3)	0.0343 (5)
O(3)	0.0467 (3)	0.35544 (14)	0.9441 (2)	0.0324 (5)
O(4)	−0.2484 (3)	0.26680 (13)	0.7846 (2)	0.0323 (5)

Table 2. *Selected geometric parameters* (Å, °)

C(1)—O(1)	1.240 (3)	C(3)—C(4)	1.528 (3)
C(1)—O(2)	1.262 (3)	C(4)—C(5)	1.512 (3)
C(1)—C(2)	1.539 (3)	C(5)—O(3)	1.209 (3)
C(2)—N(1)	1.490 (3)	C(5)—O(4)	1.326 (3)
C(2)—C(3)	1.523 (3)		
O(1)—C(1)—C(2)	126.8 (2)	C(2)—C(3)—C(4)	114.7 (2)
O(1)—C(1)—C(2)	119.9 (2)	C(5)—C(4)—C(3)	111.4 (2)
O(2)—C(1)—C(2)	113.3 (2)	O(3)—C(5)—C(4)	122.5 (2)
N(1)—C(2)—C(3)	111.5 (2)	O(3)—C(5)—C(4)	124.0 (2)
N(1)—C(2)—C(1)	109.7 (2)	O(4)—C(5)—C(4)	113.5 (2)
C(3)—C(2)—C(1)	112.4 (2)		

Table 3. *Selected torsion angles* (°) *in the three crystal structures of anhydrous glutamic acid*

	Racemate	α-Form	β-Form
N(1)—C(2)—C(3)—C(4)	69	178	−52
C(2)—C(3)—C(4)—C(5)	−175	68	−73
C(3)—C(4)—C(5)—O(4)	57	−105	−161

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Data reduction: *MolEN* (Enraf–Nonius, 1990). Program(s) used to

solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: SE1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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11-(4-Methylpiperazin-1-yl)-5*H*-pyrido-[4,3-*b*][1,5]benzodiazepine

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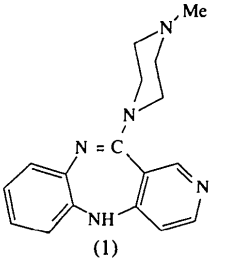
Abstract

The determination of the crystal structure of the title compound, $C_{17}H_{19}N_5$, has been undertaken as part of our studies of dopamine receptors. The diazepine ring is in a boat conformation. The dihedral angle between the two aromatic rings is 125.35 (6)°. The distances between the methylpiperazine N atom and the centres of the two aromatic rings are 5.999 (4) and 7.712 (4) Å. There is no hydrogen bond.

Comment

The title compound, (1), was prepared as part of our study of dopamine receptors. The structures of the related compounds 11-formyl-5-(4-methylpiperazin-1-yl)-11*H*-pyrido[2,3-*b*][1,5]benzodiazepine and of 6-(4-methylpiperazin-1-yl)-11-methyl-11*H*-pyrido[2,3-*b*]-[1,4]benzodiazepine (Dupont, Englebert, Dideberg, Liégeois & Delarge, 1991) have been reported previously. Other new analogous compounds are under investigation.



(1)

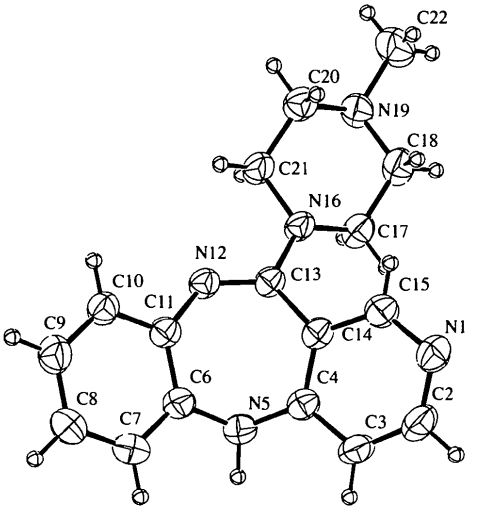


Fig. 1. The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

Experimental

Crystal data

$C_{17}H_{19}N_5$
$M_r = 293.37$

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å