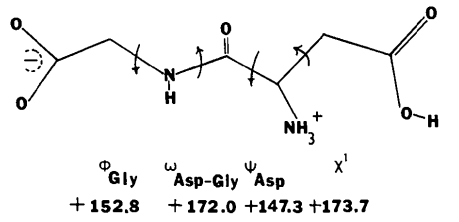

Fig. 2. Schematic showing the torsional angles (°) in the peptide backbone as defined by the IUPAC–IUB Commission on Biochemical Nomenclature (1970).

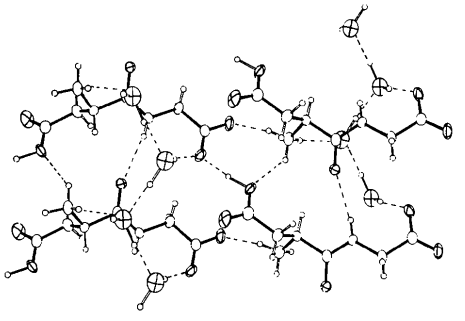

Fig. 3. The hydrogen-bonding network in crystals of α-L-aspartylglycine as viewed down **c** with **b** horizontal. O atoms are shown with principal ellipses.

donor to the peptide carboxyl O_1 along the **a** direction with $N_2\cdots O_1$ and $H_2\cdots O_1$ distances and associated N_2–$H_2\cdots O_1$ angle of 2·977 (2) Å, 2·20 (2) Å and 166(2)°, respectively. The ionized carboxyl is hydrogen bonded through O′ to the protonated amino terminus N_1 with $N_1\cdots O'$ and $H_1^1\cdots O'$ distances and N_1–$H_1^1\cdots O'$ angle of 2·715 (2) Å, 1·79 (2) Å, and 168(2)°, respectively, and through O″ to the un-ionized side chain $O_1^{\delta 2}$ of a screw-related molecule along **b** with $O_1^{\delta 2}\cdots O''$ and $H_1^{\delta}\cdots O''$ distances and $O_1^{\delta 2}$–$H_1^{\delta}\cdots O''$ angle of 2·558 (2) Å, 1·49 (3) Å, and 171 (2)°, respectively. The ionized carboxyl group is also hydrogen bonded to an adjacent water molecule with $OW_1\cdots O''$ and $HW_1\cdots O''$ distances and OW_1–$HW_1\cdots O''$ angle of 2·823 (2) Å, 2·04 (3) Å, and 173 (2)°, respectively. The water molecules form channels through the structure along the **a** direction, each water molecule acting as a donor and an acceptor towards two adjacent water molecules along the chain with an $OW_1\cdots OW_1$ distance of 2·874 (2) Å. The amino terminus also acts as a donor to an associated water molecule with an $N_1\cdots OW_1$ distance of 2·904 (2) Å, and (relatively weakly) to the Asp carboxyl group with $N_1\cdots O_1^{\delta 2}$ and $H_1^1\cdots O_1^{\delta 2}$ distances, and N_1–$H_1^1\cdots O_1^{\delta 2}$ angle of 2·932 (2) Å, 2·16 (2) Å, and 139 (2)°, respectively.

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α-L-Glutamylglycine

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Abstract. $C_7H_{12}N_2O_5$, orthorhombic, $P2_12_12_1$, a = 5·525(5), b = 12·565(4), c = 13·211(6) Å, Z = 4, D_c = 1·48, D_m (flotation in chloroform/methylene chloride) = 1·48(1) Mg m^{-3}; R_1 = 0·039, R_2 = 0·040 for 1172 observations. The dipeptide crystallizes as a zwitterion with the main-chain carboxyl ionized and the amino terminus protonated. The conformation of the peptide group is *trans*; the glutamyl side chain is extended, but

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the carboxy terminus is held by hydrogen bonding in a non-extended conformation with a torsional angle $\varphi_{Gly} = -74{\cdot}1°$.

Introduction. Crystals of α-L-glutamylglycine were grown from aqueous ethanol at pH 6–7. A crystal of dimensions 0·275 × 0·475 × 0·800 mm was used in the analysis. Preliminary cell constants were obtained on an Enraf–Nonius CAD-4 diffractometer using Mo $K\alpha$ radiation and a graphite monochromator. The crystals were assigned to the orthorhombic system, space group $P2_12_12_1$ (systematic absences: $h00$, h odd; $0k0$, k odd; $00l$, l odd). Final cell constants were determined after careful centering of 24 reflections with $35° \geq 2\theta \geq 30°$. Intensity data were collected in a θ–2θ scan mode out to $2\theta = 55°$. Intensity standards monitored at regular intervals showed no sign of crystal deterioration. The data were corrected for Lorentz–polarization effects, but not for absorption ($\mu = 0{\cdot}136$ mm $^{-1}$). Excluding 71 data with $I < 0{\cdot}01\sigma(I)$, 1172 reflections were measured. The programs used throughout the analysis were those provided by Enraf–Nonius with the CAD-4–SDP system.

The data were converted to E values and the structure was determined by *MULTAN* (Main, Woolfson & Germain, 1971) using 141 reflections for which $E \geq 1{\cdot}50$. An E map calculated from that set of phases having the highest absolute figure of merit revealed all the non-hydrogen atoms. These positions, together with their isotropic temperature factors, were refined by two cycles of least squares. In all least-squares calculations the function minimized was $\sum w(|F_o| - |F_c|)^2$ where the weights were initially assigned as unity but were eventually assigned (see below) as $4F_o^2/\sigma^2(I)$.

Following the inclusion of anisotropic thermal parameters for the non-hydrogen atoms and two cycles of least-squares refinement, a difference Fourier map was calculated; it revealed the positions of all H atoms. Three final cycles of full-matrix least-squares refinement using the weights defined above [with $\sigma(I)$ as defined by Corfield, Doedens & Ibers (1967) with $p = 0{\cdot}01$] converged to final values of the standard agreement factors $R_1 = 0{\cdot}039$ and $R_2 = 0{\cdot}040$ based on all of the observed reflections. The error on an observation of unit weight was 2·976. The atom positions along with their standard deviations, as estimated from the inverse matrix, are listed in Table 1.* A final difference Fourier map was featureless except for small peaks intermediate between covalently linked atoms which may be attributable to bonding electron density.

Table 1. *Positional parameters and thermal parameters for α-L-glutamylglycine*

	x	y	z	U_{eq}*/B† (Å^2)
N_1	0·9852 (3)	−0·0358 (1)	0·3279 (1)	0·0331
N_2	0·8654 (3)	0·2455 (1)	0·3712 (1)	0·0324
$O_1^{\varepsilon 1}$	1·2468 (3)	0·0780 (1)	0·0078 (1)	0·0511
$O_1^{\varepsilon 2}$	0·8951 (3)	0·1496 (1)	−0·0406 (1)	0·0458
O_1	0·6294 (3)	0·1006 (1)	0·3593 (1)	0·0462
O′	0·6053 (3)	0·3420 (1)	0·2181 (1)	0·0396
O″	0·2924 (3)	0·3660 (1)	0·3205 (1)	0·0361
C_1^{α}	1·0502 (4)	0·0792 (2)	0·3203 (1)	0·0361
C_1^{β}	1·1375 (4)	0·1068 (2)	0·2133 (2)	0·0376
C_1^{γ}	0·9401 (4)	0·1088 (2)	0·1325 (2)	0·0376
C_1^{δ}	1·0471 (4)	0·1109 (2)	0·0274 (1)	0·0376
C_1'	0·8247 (4)	0·1427 (2)	0·3523 (1)	0·0317
C_2^{α}	0·6640 (4)	0·3142 (2)	0·3948 (2)	0·0365
C_2'	0·5078 (4)	0·3423 (1)	0·3036 (2)	0·0313
H_1^1	0·948 (4)	−0·052 (1)	0·390 (2)	2·8 (5)
H_1^2	0·869 (4)	−0·064 (2)	0·282 (2)	4·7 (6)
H_1^3	1·120 (4)	−0·078 (2)	0·315 (2)	4·7 (6)
H_1^{α}	1·178 (4)	0·094 (1)	0·368 (2)	2·6 (4)
$H_1^{\beta 1}$	1·226 (4)	0·177 (2)	0·218 (2)	3·3 (5)
$H_1^{\beta 2}$	1·262 (4)	0·058 (1)	0·198 (1)	2·7 (4)
$H_1^{\gamma 1}$	0·811 (4)	0·169 (2)	0·141 (2)	3·6 (5)
$H_1^{\gamma 2}$	0·832 (5)	0·048 (2)	0·134 (2)	4·8 (6)
H_1^{ε}	0·979 (5)	0·150 (2)	−0·099 (2)	5·9 (7)
H_2	1·001 (4)	0·278 (2)	0·363 (2)	3·3 (5)
$H_2^{\alpha 1}$	0·556 (3)	0·277 (1)	0·444 (1)	2·4 (4)
$H_2^{\alpha 2}$	0·725 (4)	0·376 (1)	0·420 (1)	2·6 (5)

* Calculated from the principal r.m.s. amplitudes in Å, with $U_{eq}^3 = (U_1 U_2 U_3)$; e.s.d.'s are 0·0007 Å^2.
† For H atoms.

Discussion. As part of our research on the structures of peptides containing acidic amino acids (Valente, Hiskey & Hodgson, 1979; Eggleston, Valente & Hodgson, 1981) we are examining the crystal structures of peptides containing L-glutamyl residues. To our knowledge only the structures of the totally blocked dipeptide Z-(γ-ethyl)-L-glutamyl-(γ-ethyl)-L-glutamic acid ethyl ester (Benedetti, DiBlasio, Pavone, Pedone, Germain & Goodman, 1979) and of glutathione (Wright, 1958), in which the glutamyl residue is bound to the peptide chain through its γ-carboxyl group, have appeared. We find no published structural information for linear peptides containing α-glutamyl residue(s) in which the γ-carboxyl group is not blocked. We here report the structure of α-L-glutamylglycine.

An *ORTEP* drawing (Johnson, 1965) of a single molecule of the dipeptide is shown in Fig. 1; the notation used in the labeling of atoms is that adopted by the IUPAC–IUB Commission on Biochemical Nomenclature (1970). Most of the backbone bond distances are similar to the average values tabulated for peptides by Marsh & Donohue (1967). Notable exceptions, however, occur for the N–C^{α} distance,

* Lists of structure factors, anisotropic thermal parameters, and bond angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35958 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

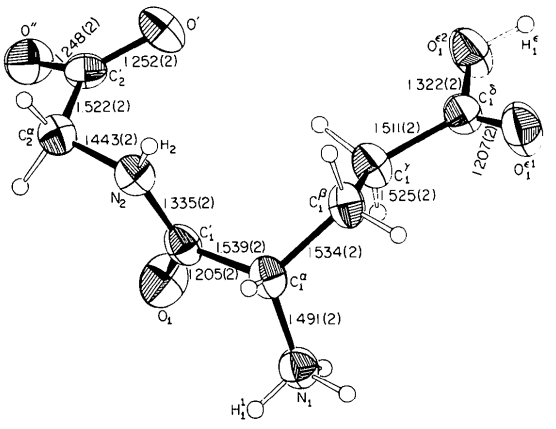

Fig. 1. View of the α-L-glutamylglycine molecule, showing the principal bond lengths (Å) with their e.s.d.'s. Thermal ellipsoids are drawn at the 50% probability level; H atoms are shown as small circles of arbitrary size.

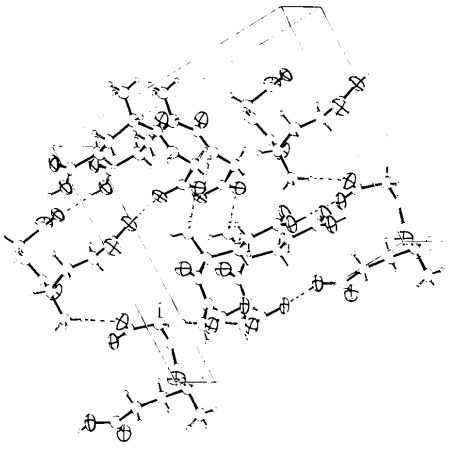

Fig. 3. The hydrogen-bonding network in crystals of α-L-glutamylglycine with the *a* axis horizontal. Oxygen atoms are shown as principal ellipses.

1·491(2) Å, of the Glu residue which is 0·04 Å longer than the tabulated average of 1·45 Å, and for the peptide carbonyl distance of 1·205(2) Å which is shorter than the reported 1·24 Å average. A progressive shortening of the C–C distances along the glutamyl side chain in proceeding to the γ-carbon atom is noted. The bond angles in the molecule have been deposited.*

The torsional angles along the peptide chain are shown in Fig. 2; the definitions of the torsional angles are those of the IUPAC–IUB Commission on Biochemical Nomenclature (1970). The values of +166·0 and +175·6° for ψ_{Glu} and $\omega_{Glu\text{-}Gly}$ respectively describe an extended conformation along the peptide backbone. The χ^1 and χ^2 angles of −47·8 and −166·7° observed here in the Glu side chain are similar to those reported in other Glu structures (Benedetti *et al.*, 1979). The φ_{Gly} angle of −74·1°, however, is very different from that expected for an extended β-peptide conformation; this non-extended structural feature is presumably due to the strong hydrogen bonding involving both O′ and O″ (see below).

* See previous footnote.

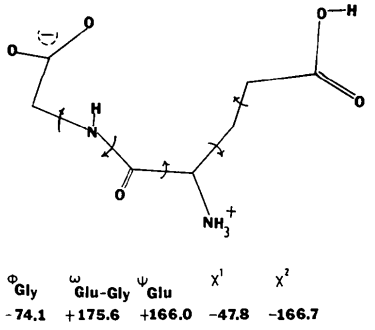

Fig. 2. Schematic showing the torsional angles (°) in the peptide backbone as defined by the IUPAC–IUB Commission on Biochemical Nomenclature (1970).

A drawing of the hydrogen-bonding network is given in Fig. 3. The molecule is held in a bent conformation with intermolecular hydrogen bonding between the ionized carboxyl O′ and the protonated $O_1^{\varepsilon 2}$ of the side-chain carboxyl of molecules related by the screw along **c** with $O' \cdots O_1^{\varepsilon 2}$ and $H_1^{\varepsilon} \cdots O'$ distances of 2·617 (1) Å and 1·73 (2) Å and $O_1^{\varepsilon 2}-H_1^{\varepsilon} \cdots O'$ angle of 172(2)°. The ionized carboxyl is also hydrogen bonded through O″ to the protonated amino group at N_1 of a screw-related molecule along **b** with $N_1 \cdots O''$ and $H_1^1 \cdots O''$ distances of 2·777 (2) Å and 1·85 (2) Å and $N_1-H_1^1 \cdots O''$ angle of 166 (2)°. A second hydrogen bond through O″ extends to the amide N_2 of a screw-related molecule along **a** with $N_2 \cdots O''$ and $H_2 \cdots O''$ distances of 2·883 (2) Å and 2·03 (2) Å and $N_2-H_2 \cdots O''$ angle of 170 (2)°. The peptide oxygen atom O_1 apparently does not participate in hydrogen bonding.

This investigation was supported by a Grant-in-Aid from the American Heart Association and with funds contributed in part by the North Carolina Heart Association. Purchase of the diffractometer was made possible by Grant CHE78-03064 from the National Science Foundation.

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Diglycidyl Ether of Bisphenol A (DGEBA)*

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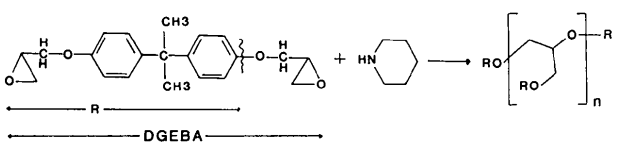
Abstract. $C_{21}H_{24}O_4$, monoclinic, $P2_1/c$, $a = 8{\cdot}260$ (4), $b = 11{\cdot}659$ (4), $c = 20{\cdot}228$ (6) Å, $\beta = 105{\cdot}6$ (1)°, $Z = 4$, $d_c = 1{\cdot}205$ (1) Mg m^{-3}. The structure was solved by the symbolic addition procedure and refined, using 2453 reflections, to an R_w of 0·055. The epoxide ring on one end of the molecule is disordered.

Introduction. DGEBA is the monomer of one of the most commonly used epoxy resins. It can be cured with piperidine. One curing mechanism, as determined by NMR is



(Sojka & Moniz, 1976). Crystals used in the X-ray analysis were provided by A. N. Garroway of the Chemistry Division of the Naval Research Laboratory. The crystals were prepared by sublimation from a melt of DER 332 (Dow Chemical). The melting point was determined to be 315–315·5 K. The crystals were extremely large (2–10 mm dimensions) and quite soft. Data were collected on an irregularly shaped fragment which had been chipped off the corner of one of the parent crystals. Cell dimensions were determined from a least-squares refinement of 18 independently measured reflections. Data were collected on a Nicolet P3F diffractometer using Cu $K\alpha$ radiation with a graphite monochromator on the incident beam. The θ–2θ scan technique was used to measure the intensities of 2453 independent reflections out to $2\theta_{max} = 112°$.

The structure was solved by routine application of the symbolic addition procedure (Karle & Karle, 1966). Atomic scattering factors used for the least-squares refinement were those listed in *International Tables for X-ray Crystallography* (1962) and the function minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = 1{\cdot}0$. All data were used in the full-matrix least-squares refinement (Busing *et al.*, 1975). It became obvious after only a few cycles of refinement that the structure was not complete. A difference map computed at this point indicated a disorder at one end of the molecule.

It should be mentioned at this point that the disorder was suspect when it first appeared and the possibility of an ordered structure in space group $P2_1$ was explored. The disorder appeared in the $P2_1$ cell as well and the refinement was thus continued in the original $P2_1/c$ cell.

The disorder consists of two alternative orientations for the epoxide ring connected to C(22). However, both alternatives have one atomic position in common, labelled A(24). One epoxide alternative consists of C(23), A(24) and O(25). The other alternative, approximately equally favored, contains C(23′), A(24) and C(25′). Occupancies for the four split atomic sites were set at 0·5 and not varied. The parameters of only one alternative were refined in any particular cycle; atom A(24) remained unresolved in difference Fourier maps and was treated as if it were a single atom during refinement. All atoms of the disordered epoxide rings were considered to be C atoms until the refinement was nearly complete. H atoms on the ordered portion of the molecule were found in a difference map computed after some anisotropic refinement had been performed. H coordinates were varied in the ensuing cycles but their thermal parameters were set equal to the final isotropic values for the atoms to which they were bonded and held constant. Under these conditions the refinement converged at $R_w = 0{\cdot}081$ where $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$. At this point atom 25 was converted to an O because it had an intermolecular approach to a neighboring C atom of 3·18 (1) Å. This change reduced R_w to 0·069. The

* 2,2′-[4,4′-Isopropylidenebis(phenoxymethyl)]bisoxiran.

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