Table 3. *Shortest interionic distances* (Å) *in* TrisH$^+$.X^-

	TrisH$^+$.Cl$^-$	TrisH$^+$.Br$^-$	TrisH$^+$.I$^-$
N···O*	2·818 (3)	2·812 (3)	2·886 (15)
X···O*	3·031 (3)	3·217 (2)	3·489 (13)
X···H(O)*	2·68 (4)	2·62 (4)	—
X···H(1)	3·28 (4)	3·28 (3)	3·39
X···H(2)	2·88 (3)	3·15 (3)	3·39
X···C(C)†	3·977 (5)	4·120 (4)	4·557 (8)
X···N†	5·447 (5)	5·618 (4)	4·015 (7)
X···X	5·154 (1)	5·217 (1)	4·922 (1)

* Hydrogen-bonded distance: for TrisH$^+$.Br$^-$, O—H(O) = 0·61 (4) Å, ∠O—H(O)···Br = 169 (5)°; N—H(N) = 0·91 (4), H(N)···O = 1·93 (4) Å, ∠N—H(N)···O = 164 (3)°; for TrisH$^+$.Cl$^-$, O—H(O) = 0·73 (5), ∠O—H(O)···Cl = 112 (4)°; N—H(N) = 0·69 (3), H(N)···O = 2·14 (3) Å, ∠N—H(N)···O = 167 (4)°.

† Along threefold axis.

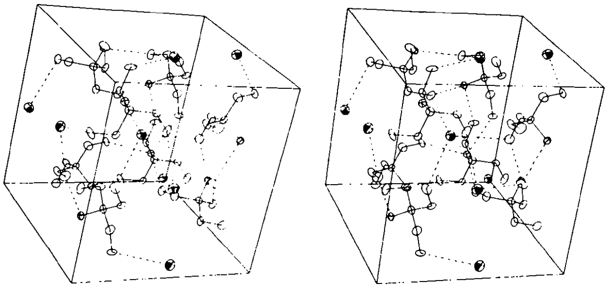

Fig. 3. Stereoview of the TrisH$^+$.I$^-$ unit cell. The I$^-$ ions are shown with shaded ellipses, while the N atoms have unshaded ellipses. The H bonding between N···O as well as the closest I···O and I···N distances are indicated by dashed lines. Focus on the I$^-$ ion closest to the center of the unit cell to observe the threefold symmetry and the iodine environment.

packing found in TrisH$^+$.I$^-$ *vs* TrisH$^+$.Br$^-$, is that the closest X···X distance is significantly shorter in TrisH$^+$.I$^-$ than in either TrisH$^+$.Br$^-$ or TrisH$^+$.Cl$^-$ (Table 3). Each I$^-$ ion lies at the center of a distorted tetrahedron formed by three adjacent I$^-$ ions [at 4·922 (1) Å] and the N atom, with three of the tetrahedral angles equal to 101·4 (4) (1)° and the other three equal to 116·7 (1)°. The sevenfold coordination about the I$^-$ ion is completed by the three nearest-neighbor O atoms (Fig. 1*b*). The structure is stabilized by the N···O hydrogen bonds.

Hydrogen bonding. The TrisH$^+$.X^- compounds are all extensively hydrogen bonded. In TrisH$^+$.I$^-$, a three-dimensional network results (Fig. 3), while in the others a layered compound is formed (Fig. 2). The Y···O distances agree with those reported by Kuleshova & Zorkii (1981): 3·12 Å for Y = Cl, 3·28 Å for Y = Br, and 2·79 Å for Y = N. However, the I···O distance is just equal to the sum of the van der Waals radii for I and O (3·48 Å, Bondi, 1968), so that in the absence of H-atom parameters, the significance of this hydrogen bond cannot be determined.

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DL-Glutamic Acid Monohydrate, $C_5H_9NO_4.H_2O$

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Abstract. M_r = 165·15, orthorhombic, *Pbca*, a = 9·119 (3), b = 15·480 (7), c = 10·627 (3) Å, V = 1500·1 Å^3, Z = 8, D_m = 1·46 (1) (by flotation), D_x = 1·46 Mg m^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 0·14 mm^{-1}, $F(000)$ = 704, room temperature. R = 0·049 for 1258 nonzero independent amplitudes. A short intermolecular H-bond with O···O = 2·574 (2) occurs between the α- and γ-carboxyl groups, as is the case in

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the α and β modifications of L-glutamic acid (L-Glu). The conformation of DL-glutamic acid, however, differs from that of the α and β modifications of L-Glu.

Introduction. L-Glutamic acid is found in two modifications: α (Bernal, 1931) and β (Hirokawa, 1955). In the α modification (Lehmann & Nunes, 1980) C^{γ} is *trans* to the N atom and C^{δ} is *gauche* ($+\chi^2$) to C^{α}; in the β modification (Hirokawa, 1955; Lehmann, Koetzle & Hamilton, 1972) C^{γ} is *trans* to the C atom and C^{δ} is *gauche* ($-\chi^2$) to C^{α}. In the crystals of L-glutamic acid hydrochloride (Sequeira, Rajagopal & Chidambaram, 1972) C^{γ} is *trans* to the C atom and C^{δ} is *trans* to C^{α}.

The α and β modifications of L-Glu show a short intermolecular H-bond between the two carboxyl groups in the molecule with O···O lengths of 2·581 (1) and 2·519 (2) Å respectively. We expected to find a different conformation of the carbon skeleton but similar H-bonds in DL-glutamic acid (DL-Glu) crystals.

Experimental. Colourless, transparent crystals from aqueous solution; 0·4 × 0·4 × 0·2 mm, density measured in benzene/1,1,2,2-tetrachloroethane, preliminary crystal data from oscillation and Weissenberg photographs, Syntex $P2_1$ computer-controlled four-circle diffractometer, scintillation counter, graphite monochromator, cell parameters by least squares from setting angles of 15 reflections, 1290 independent reflections, $2\theta \leq 55°$, variable θ–2θ scans, scan rate 2·0–29·3° min^{-1} depending on intensity, two standards every 50 reflections with average intensities and e.s.d.'s of 101396(5688) and 344475(16551); corrections for Lorentz–polarization, but not for absorption; 1258 reflections with $I > 1{\cdot}96\sigma(I)$ used for structure determination (index range h 0–11, k 0–20, l 0–13); calculations performed with the Syntex (1976) *XTL* system, neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); direct methods [*MULTAN* (Germain, Main & Woolfson, 1971)], full-matrix least-squares refinement [$\sum w(\Delta F)^2$ minimized]; H atoms from difference synthesis; non-H atoms with anisotropic and H atoms with isotropic temperature factors were refined (144 variables); $(\Delta/\sigma)_{\max} \leq 0{\cdot}1$, $(\Delta\rho)_{\max}$ in final difference map $\leq 0{\cdot}3$ e Å^{-3}; $R = 0{\cdot}049$, $R_w = 0{\cdot}042$, $S = 2{\cdot}87$, $w = 1/\sigma^2(F)$.

Discussion. Atomic parameters are in Table 1* and interatomic distances and angles in Table 2 [the

Table 1. *Positional and thermal parameters with e.s.d.'s in parentheses*

For non-H atoms, $B_{\text{eq}} = \frac{1}{3}\sum B_{ii}$ (Å²). For H atoms B_{iso} (Å²) is given.

	x	y	z	$B_{\text{eq}}/B_{\text{iso}}$
O(1)	0·3464 (2)	0·2718 (1)	0·1375 (1)	2·8 (1)
O(2)	0·1189 (2)	0·3226 (1)	0·1311 (2)	2·5 (1)
$O^{\epsilon}(1)$	0·1870 (2)	0·5044 (1)	0·5606 (2)	4·4 (2)
$O^{\epsilon}(2)$	0·3602 (2)	0·5686 (1)	0·4489 (2)	2·8 (1)
O(*W*)	0·5254 (3)	0·5819 (2)	0·2083 (2)	4·5 (2)
N	0·3478 (2)	0·2367 (1)	0·3780 (2)	1·9 (1)
C	0·2287 (2)	0·2911 (1)	0·1868 (2)	1·8 (1)
C^{α}	0·2113 (2)	0·2767 (1)	0·3283 (2)	1·8 (1)
C^{β}	0·1748 (3)	0·3591 (2)	0·4008 (2)	2·1 (2)
C^{γ}	0·2827 (3)	0·4318 (2)	0·3787 (2)	2·4 (2)
C^{δ}	0·2699 (3)	0·5039 (2)	0·4725 (2)	2·3 (2)
H^{α}	0·134 (2)	0·237 (1)	0·339 (2)	2·0 (5)
H(1)	0·340 (3)	0·227 (2)	0·463 (2)	2·4 (5)
H(2)	0·364 (3)	0·189 (2)	0·343 (3)	3·3 (7)
H(3)	0·438 (3)	0·273 (2)	0·362 (3)	4·5 (7)
$H^{\beta}(1)$	0·078 (3)	0·377 (1)	0·377 (2)	2·4 (5)
$H^{\beta}(2)$	0·171 (3)	0·342 (2)	0·488 (2)	2·5 (5)
$H^{\gamma}(1)$	0·383 (3)	0·414 (2)	0·382 (2)	2·8 (6)
$H^{\gamma}(2)$	0·272 (3)	0·456 (2)	0·296 (2)	3·1 (6)
$H^{\epsilon}(2)$	0·361 (3)	0·609 (2)	0·513 (3)	5·9 (9)
H(10)	0·465 (4)	0·558 (2)	0·163 (3)	5·4 (9)
H(11)	0·492 (4)	0·583 (2)	0·283 (4)	7·3 (11)

Table 2. *Molecular dimensions*

(*a*) Bond lengths (Å)

C–O(1)	1·231 (3)	C^{β}–C^{γ}	1·513 (3)
C–O(2)	1·261 (3)	C^{γ}–C^{δ}	1·502 (3)
C–C^{α}	1·528 (3)	C^{δ}–$O^{\epsilon}(1)$	1·203 (3)
C^{α}–N	1·487 (3)	C^{δ}–$O^{\epsilon}(2)$	1·321 (3)
C^{α}–C^{β}	1·527 (3)		

(*b*) Bond angles (°)

O(1)–C–O(2)	125·9 (2)	C^{α}–C^{β}–C^{γ}	113·6 (2)
O(1)–C–C^{α}	118·3 (2)	C^{β}–C^{γ}–C^{δ}	113·6 (2)
O(2)–C–C^{α}	115·8 (2)	C^{γ}–C^{δ}–$O^{\epsilon}(1)$	124·7 (2)
C–C^{α}–C^{β}	113·4 (1)	C^{γ}–C^{δ}–$O^{\epsilon}(2)$	112·9 (2)
C–C^{α}–N	108·9 (2)	$O^{\epsilon}(1)$–C^{δ}–$O^{\epsilon}(2)$	122·4 (2)
N–C^{α}–C^{β}	110·6 (2)		

(*c*) Torsion angles (°) (IUPAC–IUB Commission on Biochemical Nomenclature, 1970)

	DL-Glu	L-Glu	
		α form	β form
O(1)–C–C^{α}–N	−2·2 (3)	−50·2 (1)	−42·3 (2)
O(2)–C–C^{α}–N	178·1 (3)	130·3 (1)	141·2 (2)
O(1)–C–C^{α}–C^{β}	121·3 (3)		
O(2)–C–C^{α}–C^{β}	−58·4 (3)		
C–C^{α}–C^{β}–C^{γ}	−54·6 (3)		
N–C^{α}–C^{β}–C^{γ}	68·0 (3)	178·2 (1)	−51·8 (1)
C^{α}–C^{β}–C^{γ}–C^{δ}	−165·5 (3)	68·3 (1)	−73·1 (2)
C^{β}–C^{γ}–C^{δ}–$O^{\epsilon}(1)$	2·7 (4)	74·2 (1)	18·8 (2)
C^{β}–C^{γ}–C^{δ}–$O^{\epsilon}(2)$	−177·2 (3)	−104·6 (1)	−160·7 (1)

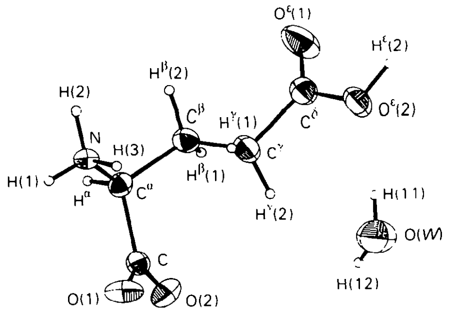

Fig. 1. View along **b** of the asymmetric unit of DL-Glu.H$_2$O. Thermal ellipsoids are drawn at the 50% probability level; H atoms are shown as spheres of arbitrary size.

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* Lists of structure factors, least-squares planes, hydrogen-bond distances and angles and anisotropic thermal parameters, and a full list of bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38615 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

atom-labelling scheme in this paper is that recommended by the IUPAC–IUB Commission on Biochemical Nomenclature (1970)]. The distances are quite similar to the values found in both the α and β forms of L-Glu (except for C—H, N—H and O—H because the structures of L-Glu were solved on the basis of neutron diffraction). As can be seen in Fig. 1 the α-carboxyl group is ionized and the proton is transferred to the amino group. The γ-carboxyl group is in the un-ionized form. Ionized and protonated carboxyl groups are quite planar [maximum atomic deviation 0·002 (2) Å].* The C—C^{α} bond length to the ionized α-carboxyl group (1·528 Å) is significantly longer than the C^{γ}—C^{δ} bond to the protonated γ-carboxyl group (1·502 Å). This trend also occurs in both modifications of L-Glu and in the structures of DL-aspartic acid, DL-Asp (Rao, 1973), and L-aspartic acid, L-Asp (Derissen, Endeman & Peerdeman, 1968). The average difference in bond length between C—C^{α} to the ionized carboxyl group and C^{β}—C^{γ} (for DL-Asp and L-Asp) or C^{γ}—C^{δ} (for DL-Glu and the α and β forms of L-Glu) to the protonated carboxyl group for all these structures is 0·030 (3) Å.

The conformation of glutamic acid in DL-Glu.$\mathrm{H_2O}$ crystals is different from those in the α and β forms of L-Glu (Table 2*c*). The α-amino part of the molecule is nearly planar. The deviation of the N atom from the C^{α}CO(1)O(2) plane is 0·050 (2) Å and the O(1)—C—C^{α}—N torsion angle (ψ^1) is—2·2°;C^{δ} is *gauche* to C and N. This implies that C^{δ} is *trans* to C^{α}, being the only possible conformation. The side chain is almost planar, and coplanar with the γ-carboxyl group.

The structure is strongly hydrogen-bonded.* Each molecule of glutamic acid is bound to adjacent molecules of glutamic acid and water through nine H-bonds. O(1), $\mathrm{O}^{\varepsilon}(1)$ and $\mathrm{O}^{\varepsilon}(2)$ each accepts one H bond and O(2) accepts two H-bonds. The donors are $\mathrm{O}^{\varepsilon}(2)$ and N, involved in one and three H-bonds respectively. The water molecule is the acceptor of one and the donor of two weak H-bonds. $\mathrm{O}^{\varepsilon}(2)$—$\mathrm{H}^{\varepsilon}(2)\cdots\mathrm{O}(2^{\mathrm{iv}})$ of length 2·574 Å is stronger than the others. This intermolecular H-bond is similar to the bonds found in the α and β forms of L-Glu, more extensively described by Lehmann & Nunes (1980), L-Asp and DL-Asp.

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* See deposition footnote.

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Quinone Oligomerization. IV.* Structure of Tetraphenyleno[1,16-*bcd*:4,5-*b'c'd'*:8,9-*b''c''d''*:12,13-*b'''c'''d'''*]tetrafuran, $\mathrm{C_{24}H_8O_4}$

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(*Received* 14 *February* 1983; *accepted* 13 *May* 1983)

Abstract. $M_r = 360\cdot4$, monoclinic, $P2_1/c$, $a = 3\cdot745\,(1)$, $b = 11\cdot602\,(3)$, $c = 16\cdot597\,(3)$ Å, $\beta = 93\cdot87\,(2)°$, $V = 719\cdot5\,(5)$ Å^3, $Z = 2$, $D_x = 1\cdot664\,(1)$ Mg m^{-3}, $\mu(\mathrm{Cu}\,K\alpha) = 0\cdot61$ mm^{-1}, $F(000) = 368$. The final R value is 0·068 for 491 observed independent reflexions, collected using Cu $K\alpha$ radiation ($\lambda = 1\cdot5418$ Å) at room temperature and for which $I_{\mathrm{net}} \geq 4\sigma(I_{\mathrm{net}})$. The molecule is planar within experimental errors. The packing is dense with a packing coefficient of 0·75.

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* Part III: Karlsson, Pilotti & Söderholm (1980).

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