

clude that close contacts are responsible for reducing the amount of free space in the unit cells and, thus, for the high crystal densities.

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### *N*-(*p*-Aminobenzoyl)-L-glutamic Acid Hydrochloride

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**Abstract.**  $C_{12}H_{15}N_2O_5^+ \cdot Cl^-$ ,  $M_r = 302.70$ , monoclinic,  $P2_1$ ,  $a = 11.819$  (3),  $b = 4.924$  (1),  $c = 12.085$  (1) Å,  $\beta = 102.4$  (1)°,  $V = 686.82$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.46$ ,  $D_m$  (floatation in benzene/bromoform) = 1.47 Mg m<sup>-3</sup>,  $\mu$ (Cu  $K\alpha$ ) = 2.704 mm<sup>-1</sup>. The structure was solved by direct methods and refined to a final  $R$  value of 0.065 for 1022 reflections. The *p*-aminobenzoic acid part of the molecule is linked to the glutamic acid through a peptide-like linkage with an amide C–N distance of 1.340 (9) Å. C <sup>$\delta$</sup>  in the glutamic acid side chain is *gauche* to C <sup>$\alpha$</sup>  ( $\chi^2 = 74^\circ$ ), and the  $\alpha$ -carboxyl C atom is *trans* to C <sup>$\gamma$</sup> , the torsion angle C–C <sup>$\alpha$</sup> –C <sup>$\beta$</sup> –C <sup>$\gamma$</sup>  being  $-177^\circ$ . The  $\alpha$ -N deviates from the plane of the  $\alpha$ -carboxyl group, and  $\psi^1$  has a value of 22.5°.

**Introduction.** *p*-Aminobenzoyl-L-glutamic acid (PABG) is a sulfanilamide antagonist and is a major portion of folic acid. It is well known that the antibacterial action of sulfonamide is related to its ability to block bacterial synthesis of folic acid. The presence of *p*-aminobenzoic acid (PABA) and glutamic acid moieties in the folic acid molecule is a feature of special biological interest (Herbert, 1966). These are linked through a peptide-like linkage to form PABG.

The present study was undertaken to provide structural data to help interpret the biological function of the molecule.

Thin, spear-shaped crystals were obtained by the slow cooling of a solution of a stoichiometric amount of PABG in 0.1 *M* hydrochloric acid. Preliminary oscillation and Weissenberg photographs using Cu  $K\alpha$  radiation showed the crystals to be monoclinic, space group  $P2_1$ . Refined cell parameters and their standard deviations were derived from a least-squares fit to the setting angles of 25 reflections centered on an Enraf–Nonius CAD-4 diffractometer.

A crystal of dimensions 0.28 × 0.13 × 0.03 mm was used for collecting intensity data on the diffractometer with graphite-monochromatized Cu  $K\alpha$  radiation. All reflections in the range  $2 < \theta < 60^\circ$  were measured with an  $\omega/2\theta$  scan, a variable scan speed and a scan range of  $(0.7 + 0.14 \tan \theta)^\circ$ . Three standard reflections were monitored after each group of 50 measurements; they did not show any noticeable change in intensity. Of the 1169 reflections measured, 1023 were considered observed with  $F_o > 2\sigma(F)$ , where  $\sigma(F)$  is the e.s.d. based on counting statistics, and these were corrected for Lorentz and polarization effects, but no correction was made for absorption.

The structure was solved by direct methods using the program *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), employing the 230 *E* values with *E* > 1.21. 17 non-hydrogen atoms were located from an *E* map calculated with a phase set having the highest combined figure of merit and the remaining three on a subsequent Fourier map. Full-matrix isotropic least-squares refinement and block-diagonal anisotropic refinement converged to a conventional *R* index ( $R = \sum ||F_o| - |F_c|| / \sum |F_o|$ ) of 0.142 and a weighted *R<sub>w</sub>* index ( $\{R_w = [\sum w(|F_o| - |F_c|)^2 / \sum wF_o^2]^{1/2}\}$ ) of 0.127. The quantity minimized in the least-squares refinement was  $w(|F_o| - |F_c|)^2$  with  $w = 1/\sigma^2(F)$ . The scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Five H atoms were located on a difference Fourier map and the rest fixed by geometry were placed at calculated positions (C-H = 1.0 Å). All the H atoms were assigned the isotropic temperature factors of the

heavier atoms to which they are bonded and their inclusion as fixed parameters gave *R* = 0.138 and *R<sub>w</sub>* = 0.116. During the next cycles of refinement, the weighting function was re-estimated (Seal & Ray, 1981) to keep  $\sum w(\Delta F)^2$  uniform over the ranges of  $(\sin \theta)/\lambda$  and  $|F_o|$ , and the final *R* value was 0.065 (*R<sub>w</sub>* = 0.086). One strong reflection (30 $\bar{1}$ ) showing extinction was excluded from the final stages of the calculations. Atomic parameters are given in Table 1.\*

**Discussion.** The atom numbering scheme is shown in Fig. 1. The bond lengths and bond angles are listed in Table 2. Least-squares planes have been calculated

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36764 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional parameters ( $\times 10^4$ , for H  $\times 10^3$ ) with *e.s.d.*'s in parentheses and equivalent isotropic thermal parameters ( $\text{Å}^2$ )

$$B_{eq} = \frac{1}{3}(\beta_{11} a^2 + \beta_{22} b^2 + \beta_{33} c^2 + \beta_{12} ab \cos \gamma + \beta_{13} ab \cos \beta + \beta_{23} bc \cos \alpha).$$

	x	y	z	<i>B<sub>eq</sub></i>
Cl	-1348 (2)	2500	-5769 (1)	3.5
N(1)	266 (5)	2378 (18)	-3239 (4)	3.5
C(1)	853 (6)	2007 (16)	-2047 (5)	2.6
C(2)	460 (6)	-6 (18)	-1424 (5)	3.2
C(3)	1034 (6)	-400 (16)	-309 (5)	2.8
C(4)	1974 (5)	1189 (14)	177 (5)	2.2
C(5)	2348 (6)	3178 (15)	-466 (5)	2.7
C(6)	1777 (6)	3574 (18)	-1594 (5)	3.1
C(7)	2543 (6)	689 (15)	1391 (5)	2.3
O(1)	2491 (5)	-1558 (11)	1821 (4)	3.7
N(2)	3095 (4)	2813 (12)	1957 (4)	2.1
C(8)	3614 (5)	2629 (18)	3166 (4)	2.4
C(9)	2705 (6)	3646 (18)	3823 (5)	3.1
O(2)	1707 (4)	3655 (17)	3426 (4)	5.0
O(3)	3177 (5)	4446 (20)	4846 (4)	6.1
C(10)	4751 (6)	4115 (17)	3477 (5)	2.8
C(11)	5606 (6)	3207 (18)	2773 (5)	3.2
C(12)	5388 (6)	4157 (18)	1570 (5)	3.0
O(4)	5049 (5)	6705 (12)	1448 (4)	3.8
O(5)	5555 (5)	2764 (14)	803 (4)	4.3
H(N1)	-50	122	-343	
H'(N1)	30	420	-360	
H''(N1)	62	100	-365	
H(C2)	-28	-119	-177	
H(C3)	77	-190	18	
H(O3)	275	560	510	
H(O4)	472	710	75	
H(N2)	300	467	172	
H(C8)	381	63	343	
H(C5)	310	427	-10	
H(C6)	216	477	-214	
H(C10)	505	396	429	
H'(C10)	455	603	326	
H(C11)	571	112	287	
H'(C11)	643	389	326	

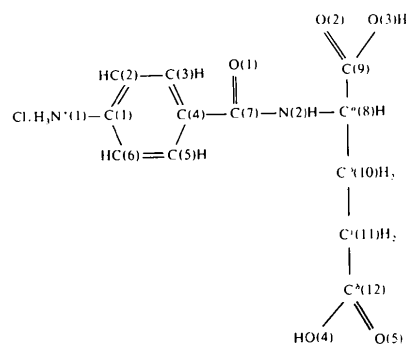


Fig. 1. The atomic numbering scheme with conformational notation of the atoms in the glutamic acid part of the molecule.

Table 2. Intramolecular bond distances and angles (°) with standard deviations in parentheses

Average O-H = 0.87 and average N-H = 1.00 Å.

N(1)-C(1)	1.470 (8)	N(2)-C(8)	1.461 (7)
C(1)-C(2)	1.384 (10)	C(8)-C(9)	1.549 (9)
C(2)-C(3)	1.386 (9)	C(9)-O(2)	1.175 (9)
C(3)-C(4)	1.382 (10)	C(9)-O(3)	1.303 (8)
C(4)-C(5)	1.381 (10)	C(8)-C(10)	1.505 (10)
C(5)-C(6)	1.399 (9)	C(10)-C(11)	1.522 (10)
C(6)-C(1)	1.353 (10)	C(11)-C(12)	1.497 (9)
C(4)-C(7)	1.497 (8)	C(12)-O(4)	1.316 (10)
C(7)-O(1)	1.229 (9)	C(12)-O(5)	1.202 (9)
C(7)-N(2)	1.340 (9)		
N(1)-C(1)-C(2)	118.6 (6)	C(7)-N(2)-C(8)	121.1 (5)
N(1)-C(1)-C(6)	119.8 (6)	N(2)-C(8)-C(9)	107.7 (5)
C(6)-C(1)-C(2)	121.6 (7)	N(2)-C(8)-C(10)	112.3 (6)
C(1)-C(2)-C(3)	118.6 (7)	C(8)-C(9)-O(2)	122.6 (7)
C(2)-C(3)-C(4)	121.0 (7)	C(8)-C(9)-O(3)	112.4 (7)
C(3)-C(4)-C(5)	119.2 (6)	O(2)-C(9)-O(3)	125.0 (8)
C(4)-C(5)-C(6)	120.1 (6)	C(9)-C(8)-C(10)	113.3 (6)
C(5)-C(6)-C(1)	119.6 (7)	C(8)-C(10)-C(11)	112.5 (6)
C(3)-C(4)-C(7)	118.3 (6)	C(10)-C(11)-C(12)	117.1 (6)
C(5)-C(4)-C(7)	122.5 (6)	C(11)-C(12)-O(4)	113.1 (6)
C(4)-C(7)-O(1)	120.6 (6)	C(11)-C(12)-O(5)	123.2 (7)
C(4)-C(7)-N(2)	116.0 (6)	O(4)-C(12)-O(5)	123.7 (7)
O(1)-C(7)-N(2)	123.4 (6)		

Table 3. Least-squares planes with displacements of atoms from the planes (Å) and e.s.d.'s in parentheses

The plane equations are in the form  $AX + BY + CZ = D$ , where  $X$ ,  $Y$  and  $Z$  are expressed in Å, referred to orthogonal axes. Atoms included in the calculation of the least-squares planes are denoted by asterisks.

## Plane P1: phenyl ring

$$0.6910X - 0.6587Y - 0.2977Z - 1.1345 = 0$$

N(1)	0.030 (7)	*C(4)	-0.002 (7)
*C(1)	-0.002 (7)	*C(5)	-0.000 (7)
*C(2)	-0.001 (8)	*C(6)	0.004 (8)
*C(3)	0.004 (8)	C(7)	-0.020 (7)

Plane P2:  $\alpha$ -carboxyl group

$$0.1562X + 0.9263Y - 0.3430Z - 0.4569 = 0$$

N(2)	0.526 (6)	*O(2)	-0.001 (8)
*C(8)	-0.001 (8)	*O(3)	-0.001 (10)
*C(9)	0.003 (8)	C(10)	0.748 (8)

Plane P3:  $\delta$ -carboxyl group

$$-0.9387X - 0.3090Y - 0.1530Z + 6.5280 = 0$$

C(10)	0.850 (7)	*O(4)	-0.003 (6)
*C(11)	-0.005 (8)	*O(5)	-0.004 (6)
*C(12)	0.017 (7)		

through different regions of the molecule and are given in Table 3.

The PABA part of the molecule is connected to glutamic acid by a  $-\text{CO}-\text{NH}-$  bond with  $\text{C}(7)-\text{N}(2)$  1.340 (9) Å. The  $\text{N}(2)-\text{C}(8)$  distance of 1.461 (7) Å also compares well with values obtained in peptides.  $\text{C}(4)-\text{C}(7) = 1.497$  (8),  $\text{C}(7)-\text{O}(1) = 1.229$  (9),  $\text{C}(7)-\text{N}(2) = 1.340$  (9) and  $\text{N}(2)-\text{C}(8) = 1.461$  (7) Å are comparable to the corresponding values [1.496 (6), 1.238 (5), 1.336 (6) and 1.454 (6) Å] found in hippuric acid (Ringertz, 1971). The C-C bond distances in the glutamic acid side chain are normal.

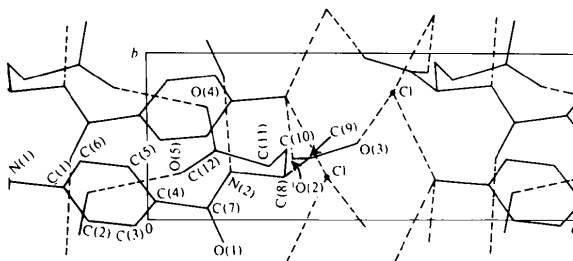
The conformation of the glutamic acid part is described in terms of the torsion angles given in Table 4. The side chain is buckled with  $\text{C}^\delta$  *gauche* to  $\text{C}^\alpha$  with respect to  $\text{C}^\beta-\text{C}^\gamma$  ( $\chi^2 = 74.2^\circ$ ). As a result there is a close contact of 2.923 (9) Å between C(12) and the amide nitrogen N(2). The  $\alpha$ -carboxyl carbon C(9) is *trans* to  $\text{C}^\gamma$ , as indicated by the torsion angle  $\text{C}(9)-\text{C}(8)-\text{C}(10)-\text{C}(11) = -176.5^\circ$ . N(2) deviates considerably from the  $\alpha$ -carboxyl plane [Table 3, 0.526 (6) Å] and  $\psi^1 = 22.5^\circ$ , which is close to the value obtained in L-glutamic acid hydrochloride (Sequeira, Rajagopal & Chidambaram, 1972), but opposite in sign.

A projection of the structure along *a* is shown in Fig. 2, in which the hydrogen bonds are indicated by broken lines. All six H atoms available for hydrogen-bond formation participate in hydrogen bonding and the structure is stabilized by a three-dimensional network of hydrogen bonds. The geometry of the hydrogen

Table 4. Selected torsion angles ( $^\circ$ )

The nomenclature followed is that recommended by the IUPAC-IUB Commission on Biochemical Nomenclature (1970). The mean standard deviation is 0.8 $^\circ$ .

$\text{N}(2)-\text{C}(8)-\text{C}(9)-\text{O}(2)$	( $\psi^1$ )	22.5
$\text{N}(2)-\text{C}(8)-\text{C}(9)-\text{O}(3)$	( $\psi^2$ )	-158.0
$\text{N}(2)-\text{C}(8)-\text{C}(10)-\text{C}(11)$	( $\chi^1$ )	-54.3
$\text{C}(8)-\text{C}(10)-\text{C}(11)-\text{C}(12)$	( $\chi^2$ )	74.2
$\text{C}(10)-\text{C}(11)-\text{C}(12)-\text{O}(5)$	( $\chi^{3,4,1}$ )	-142.0
$\text{C}(10)-\text{C}(11)-\text{C}(12)-\text{O}(4)$	( $\chi^{3,4,2}$ )	41.0
$\text{C}(3)-\text{C}(4)-\text{C}(7)-\text{O}(1)$		26.2

Fig. 2. Projection of the crystal structure down *a*. H atoms are omitted.

bonding is given in Table 5. It should be noted, however, that N(1) has four short contacts which are of a suitable distance for hydrogen-bond formation (Table 5).  $\text{H}'(\text{N}1)$  and  $\text{H}''(\text{N}1)$  form two hydrogen bonds with Cl atoms at  $(-x, y - \frac{1}{2}, -1 - z)$  and  $(-x, y + \frac{1}{2}, -1 - z)$  respectively.  $\text{H}(\text{N}1)$  is involved in two short contacts between N(1) and two acceptor atoms  $\text{O}(2)^a$  at  $(-x, y - \frac{1}{2}, -z)$  and Cl at  $(x, y, z)$ . The interaction  $\text{H}(\text{N}1) \cdots \text{Cl}$  (2.86 Å) is weaker than the  $\text{H}(\text{N}1) \cdots \text{O}(2)^a$  interaction (1.91 Å) with angles  $\text{N}(1)-\text{H}(\text{N}1) \cdots \text{Cl} = 102^\circ$  and  $\text{N}(1)-\text{H}(\text{N}1) \cdots \text{O}(2)^a = 165^\circ$ . Following the convention of Jeffrey & Maluszynska (1982), the term 'three-center' hydrogen bond instead of 'bifurcated' hydrogen bond is more appropriately used to describe

Table 5. Hydrogen-bond distances (Å) and angles ( $^\circ$ )

$D-\text{H} \cdots A$	$D \cdots A$	$\text{H} \cdots A$	$\angle D-\text{H} \cdots A$
$\text{N}(1)-\text{H}(\text{N}1) \cdots \text{O}(2)^a$	2.936 (10)	1.91	165
* $\text{N}(1)-\text{H}(\text{N}1) \cdots \text{Cl}$	3.238 (5)	2.86	102
$\text{N}(1)-\text{H}'(\text{N}1) \cdots \text{Cl}^c$	3.177 (8)	2.27	150
$\text{N}(1)-\text{H}''(\text{N}1) \cdots \text{Cl}^d$	3.083 (8)	2.11	168
$\text{N}(2)-\text{H}(\text{N}2) \cdots \text{O}(1)^b$	2.858 (8)	1.96	154
* $\text{N}(2)-\text{H}(\text{N}2) \cdots \text{C}(12)$	2.923 (9)	2.88	83
$\text{O}(3)-\text{H}(\text{O}3) \cdots \text{Cl}^e$	3.036 (7)	2.20	163
$\text{O}(4)-\text{H}(\text{O}4) \cdots \text{O}(5)^f$	2.711 (7)	1.86	164

Symmetry code: none  $x, y, z$ ; (a)  $-x, y - \frac{1}{2}, -z$ ; (b)  $x, y + 1, z$ ; (c)  $-x, y + \frac{1}{2}, -1 - z$ ; (d)  $-x, y - \frac{1}{2}, -1 - z$ ; (e)  $-x, y + \frac{1}{2}, -z$ ; (f)  $1 - x, y + \frac{1}{2}, -z$ .

\* These are the weaker components of bifurcated interactions and are not shown in Fig. 2.

the type of interaction in which one of the components is weaker than the other. Another criterion observed is that the H atom lies in or close to the plane of the donor and the two acceptor atoms. The planarity of N(1), H(N1), Cl and O(2)<sup>a</sup> for the present molecule is indicated by the sum of the three angles at H(N1), which is 360°.

The short intramolecular distance of 2.923 (9) Å between N(2) and C(12) in the molecule mentioned earlier also appears to be due to a three-center hydrogen bond involving the atoms N(2), O(1)<sup>b</sup> (x, y + 1, z) and C(12), the weaker component being N(2)–H(N2)···C(12) (83°). The relevant distances are H(N2)···C(12) = 2.88 and H(N2)···O(1)<sup>b</sup> = 1.96 Å. The angles N(2)–H(N2)···C(12) (83°), N(2)–H(N2)···O(1) (154°) and C(12)···H(N2)···O(1) (114°) at H(N2) sum to 351°.

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### N-Methyl-10-azabicyclo[4.3.1]decane-8-spiro-5'-hydantoin\*†

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**Abstract.** C<sub>12</sub>H<sub>19</sub>N<sub>3</sub>O<sub>2</sub>, *M<sub>r</sub>* = 237.303, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 6.853 (1), *b* = 11.995 (1), *c* = 14.835 (1) Å, β = 90.58 (3)°, *V* = 1217.40 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.293 g cm<sup>-3</sup>, μ (Mo *K*α) = 0.8396 cm<sup>-1</sup>. The structure was solved by direct methods and refined to an *R* of 0.042 for 2621 observed reflections. The bicyclo[4.3.1]decane system has a chair–boat conformation. The CH<sub>3</sub> group is attached in an axial position.

**Introduction.** The title compound was synthesized by Dr E. Martínez Muñoz of the Departamento de Química Orgánica de la Facultad de Farmacia de la Universidad Complutense de Madrid. The chemical structure could not be unequivocally established by conventional spectroscopic techniques.

The crystal used for all X-ray measurements was a transparent colourless parallelepiped of approximate dimensions 0.33 × 0.27 × 0.21 mm. An accurate determination of the cell parameters was made by a least-squares fit of the settings of 25 reflections on an automatic Enraf–Nonius CAD-4 diffractometer.

\* The Conformation of Heterocyclic Spiro Compounds. XI. Part X: Vilches, Florencio, Smith-Verdier & García-Blanco (1981).

† Hydantoin is 2,4-imidazolidinedione.

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The space group *P*2<sub>1</sub>/*n* was assigned uniquely from the systematic absences. The intensities of reflections up to 2θ = 60° were collected on the diffractometer using graphite-monochromatized Mo *K*α radiation. 3486 independent reflections were recorded with the θ–2θ scan mode, 2621 of which were considered as observed [*I* ≥ 2σ(*I*) where σ(*I*) was calculated from counting statistics]. The intensities were not corrected for absorption.

The structure was solved by *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The solution was based on phases determined for 250 reflections with *E* ≥ 1.90. The *E* map with the best figure of merit revealed the positions of all the non-hydrogen atoms.

After full-matrix least-squares refinement with anisotropic temperature factors (*R* = 0.090), the H atoms were located in a difference map. Final refinement, in which the positional parameters and isotropic temperature factors of the H atoms were allowed to vary, converged at *R* = 0.042 and *R<sub>w</sub>* = [∑ w(|*F<sub>o</sub>*| – |*F<sub>c</sub>*|)<sup>2</sup>/∑ w|*F<sub>o</sub>*|<sup>2</sup>]<sup>1/2</sup> = 0.047. The function minimized was ∑ w(|*F<sub>o</sub>*| – |*F<sub>c</sub>*|)<sup>2</sup> with *w* = 1.00. The final difference map contained no peaks >0.3 e Å<sup>-3</sup>. The scattering factors were taken from *International Tables for*