LIDE, D. R. (1962). *Tetrahedron*, **17**, 125–134.
LUGER, P. & BÜLOW, R. (1983). *J. Appl. Cryst.* **16**, 431–432.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. de York, Angleterre, et Louvain, Belgique.
MARKHAM, K. R., VILAIN, C., WOLLENWEBER, E., DIETZ, V. H. & SCHILLING, G. (1985). *Z. Naturforsch. Teil C*, **40**, 317–320.
NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. C. (1968). *Acta Cryst.* A**24**, 351–359.
SBIT, M., DUPONT, L., DIDEBERG, O. & VILAIN, C. (1987). *Acta Cryst.* C**43**. A publier.
SHELDRICK, G. M. (1976). *SHELX*76. Programme pour la détermination des structures cristallines. Univ. de Cambridge, Angleterre.
VILAIN, C., HUBERT, A. J., DUPONT, L., MARKHAM, K. R. & WOLLENWEBER, E. (1986). En préparation.

Acta Cryst. (1987). C**43**, 728–729

Nicotinoylglycine, a Metabolic Product

BY S. KRISHNASWAMY AND VASANTHA PATTABHI*

Department of Crystallography and Biophysics,† University of Madras, Guindy Campus, Madras 600 025, *India*

AND T. N. GURU ROW

Physical Chemistry Division, National Chemical Laboratory, Pune 411 000, *India*

(*Received* 25 *March* 1986; *accepted* 3 *December* 1986)

Abstract. Glycine-*N*-(3-pyridinylcarbonyl), $C_8H_8N_2O_3$, $M_r = 180{\cdot}16$, triclinic, $P\bar{1}$, $a = 4{\cdot}934$ (1), $b = 8{\cdot}052$ (1), $c = 10{\cdot}518$ (2) Å, $\alpha = 101{\cdot}11$ (1), $\beta = 102{\cdot}71$ (2), $\gamma = 95{\cdot}05$ (2)°, $V = 396{\cdot}3$ (1) Å^3, $Z = 2$, D_m(flotation) = 1·49 (1), $D_x = 1{\cdot}510$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}075$ mm^{-1}, $F(000) = 188$, $T = \sim 298$ K, $R = 0{\cdot}028$ for 777 observed reflections. The pyridine ring is not protonated and is planar. The peptide torsion $\omega = -177{\cdot}1$ (2)°. The packing of the molecules is stabilized by one O—H···N, one N—H···O and three C—H···O hydrogen bonds.

Introduction. Nicotinoylglycine is a metabolic product of the vitamin B complex component niacin. It is formed by the condensation of glycine with niacin. A wide variety of pharmacological properties have been associated with this compound. The primary amongst these is the lowering of serum cholesterol levels (Burger, 1970). The structure determination of this molecule forms part of a programme of work on metabolites and drugs.

Experimental. Sample from Sigma Chemicals Ltd, USA. Crystals from slow evaporation of an aqueous solution. Crystal size 0·15 × 0·10 × 0·50 mm. Unit-cell refinement using θ values of 40 reflections with $20 \le \theta \le 24°$. Data collected on an Enraf–Nonius CAD-4 diffractometer, $2\theta_{max} = 48°$, $-5 \le h \le 5$, $-8 \le k \le 7$, $0 \le l \le 11$. Three standard reflections monitored every 2000 s of radiation time showed no appreciable variation in intensity. Data corrected for Lorentz and polarization effects. No absorption correction. 777 reflections with $F \ge 3\sigma(F)$ observed, out of 1244 reflections. Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), in the space group $P1$ after repeated trials. Origin shifted to centre of symmetry before refinement. Full-matrix refinement on F with *SHELX*76 (Sheldrick, 1976). H atoms located by difference synthesis. Refinement with H atoms isotropic and non-H atoms anisotropic. $R = 0{\cdot}028$, $wR = 0{\cdot}031$, $S = 1{\cdot}27$ for 150 parameters. $w = [\sigma^2(F_o) + 0{\cdot}0002F_o^2]^{-1}$. $(\Delta/\sigma)_{max} = 0{\cdot}005$, final difference map peak $< 0{\cdot}15$ e Å^{-3}. R with unobserved reflections = 0·089. Atomic scattering factors those of *SHELX*76 (Sheldrick, 1976).

Discussion. The coordinates and equivalent thermal factors of non-H atoms are listed in Table 1‡ and a view of the molecule is shown in Fig. 1. The average e.s.d.'s in bond lengths and bond angles are 0·002 Å and 0·2° respectively. The bond lengths and bond angles are

* To whom correspondence should be addressed.

† Contribution No. 696 from the Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Madras 600 025, India.

‡ Lists of anisotropic thermal factors for non-H atoms, coordinates and isotropic thermal factors of H atoms, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43579 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/87/040728-02$01.50

normal. The pyridine ring is planar ($\chi^2 = 10{\cdot}30$) and not protonated, in agreement with the fact that the bond angle at N(1) (Fig. 2) is narrower than 125°, a value usually observed when N is protonated (Takusagawa, Hirotsu & Shimada, 1973). The peptide group is non-planar [$\omega = -177{\cdot}1$ (2)°]. The angle between the pyridine ring and the peptide plane is 28·0 (2)°. This value is similar to that found in *para*-substituted pyridine structures: nicotinamide 24° (Wright & King, 1954), indole acetic acid–nicotinamide complex 30·6° (Inoue, Sakakai, Fujiwara & Tomita, 1978) and *N*-methyl benzyl nicotinamide 34° (Little & Morimoto, 1981). The torsion angles corresponding to the φ, ψ angles of the amino acids are C(6)–N(6)–C(7)–C(8) 154·3 (2)° and N(6)–C(7)–C(8)–O(9) 177·6 (2)°.

Table 1. *Fractional coordinates* (× 10⁴) *and equivalent isotropic thermal factors* (× 10⁴) *for non*-H *atoms in nicotinoylglycine, with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}(Å²)
N(1)	−4880 (3)	−2468 (2)	−2366 (1)	410 (7)
C(1)	−6165 (4)	−3003 (3)	−3649 (2)	352 (7)
C(2)	−5203 (3)	−2493 (2)	−4667 (2)	280 (6)
C(3)	−2777 (4)	−1361 (2)	−4317 (2)	341 (7)
C(4)	−1432 (4)	−786 (3)	−2989 (2)	418 (8)
C(5)	−2558 (4)	−1368 (3)	−2052 (2)	433 (8)
C(6)	−6940 (4)	−3082 (2)	−6058 (2)	291 (6)
O(6)	−9492 (2)	−3439 (2)	−6257 (1)	442 (6)
N(6)	−5654 (4)	−3147 (2)	−7047 (1)	340 (6)
C(7)	−7211 (4)	−3628 (3)	−8416 (2)	367 (7)
C(8)	−5768 (4)	−2918 (2)	−9336 (2)	334 (7)
O(8)	−3581 (3)	−2002 (2)	−8986 (1)	484 (5)
O(9)	−7187 (3)	−3430 (2)	−10574 (1)	531 (6)

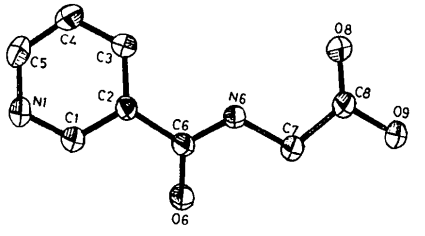

Fig. 1. *ORTEP* drawing (Johnson, 1965) showing thermal ellipsoids at 50% probability level for nicotinoylglycine.

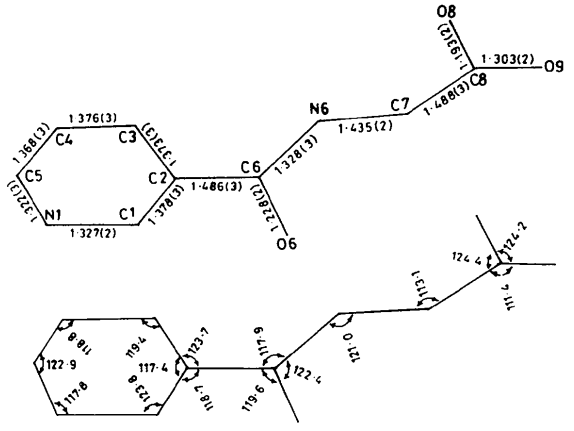

Fig. 2. Bond lengths (Å) and bond angles (°) for nicotinoylglycine: e.s.d.'s for bond lengths are given in parentheses, average e.s.d. for bond angles is 0·2°.

Table 2. *Hydrogen-bond lengths* (Å) *and angles* (°) *for nicotinoylglycine*

D–H···*A*	*D*···*A*	H···*A*	*D*–H···*A*	Equivalent position of *A*
O(9)–H(O9)···N(1)	2·608 (2)	1·60 (3)	170 (3)	*x*, *y*, *z*−1
N(6)–H(N6)···O(6)	3·013 (2)	2·28 (2)	163 (2)	*x*+1, *y*, *z*
C(4)–H(C4)···O(8)	3·231 (3)	2·35 (2)	151 (2)	−*x*, −*y*, −*z*−1
C(7)–H(C7)···O(8)	3·486 (3)	2·51 (2)	170 (2)	*x*−1, *y*, *z*
C(1)–H(C1)···O(6)	3·395 (3)	2·53 (2)	153 (2)	−*x*−2, −*y*−1, −*z*−1

An intermolecular O–H···N hydrogen bond is formed between the hydroxyl group and the pyridine N atom (Table 2). This is typical of pyridinecarboxylic acids, where O–H···N(pyridine) hydrogen bonds are preferred over O–H···O bonds (Takusagawa & Shimada, 1976). A weaker intermolecular N–H···O hydrogen bond is formed between the N and O atoms of the peptide. The presence of C–H···O hydrogen bonds could be the reason why a zwitterion is not formed. It has been reported that the formation of C–H···O hydrogen bonds involving the carbonyl of the carboxyl group is energetically preferred to zwitterion formation and is consequently competitive with protonation of the pyridine N atom (Takusagawa & Shimada, 1976).

SK thanks the University Grants Commission for financial assistance and Professor K. Venkatesan, Organic Chemistry, Indian Institute of Science, Bangalore for helpful discussions.

References

BURGER, A. (1970). *Medicinal Chemistry*, Part II. New York: Wiley–Interscience.

INOUE, M., SAKAKAI, T., FUJIWARA, T. & TOMITA, K. (1978). *Bull. Chem. Soc. Jpn*, **51**, 1118–1122.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

LITTLE, R. G. & MORIMOTO, C. (1981). *Acta Cryst.* B**37**, 1637–1639.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

TAKUSAGAWA, F., HIROTSU, T. & SHIMADA, A. (1973). *Bull. Chem. Soc. Jpn*, **46**, 2292–2299.

TAKUSAGAWA, F. & SHIMADA, A. (1976). *Acta Cryst.* B**32**, 1925–1927.

WRIGHT, W. B. & KING, G. S. D. (1954). *Acta Cryst.* **7**, 283–288.