

Refinement of the Structure of *N*-Acetyl-L-tyrosine Ethyl Ester Monohydrate\*

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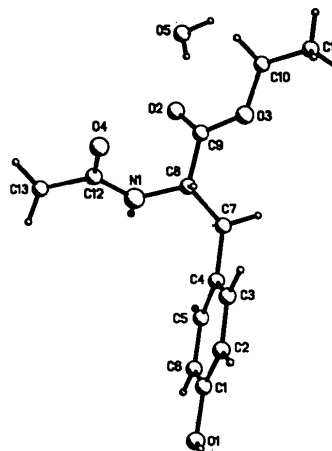
(Received 21 January 1992; accepted 27 April 1992)

**Abstract.**  $C_{13}H_{17}NO_4 \cdot H_2O$ ,  $M_r = 269.3$ , orthorhombic,  $P2_12_12_1$ ,  $a = 7.383$  (1),  $b = 13.134$  (5),  $c = 14.510$  (4) Å,  $V = 1407$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.27$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 0.778$  mm<sup>-1</sup>,  $F(000) = 576$ ,  $T = 293$  K,  $R = 0.052$  for 1046 observed reflections. The crystal structure determined from Weissenberg data ( $R = 0.107$  for 1036 visual estimations) by Pieret, Durant, Germain & Koch [*Cryst. Struct. Commun.* (1972), **1**, 75–77] is properly refined and the hydrogen-bonding scheme is clarified. The conformational angles  $\varphi$ ,  $\psi$ ,  $\chi^1$ ,  $\chi^2$  for this structure [IUPAC–IUB Commission on Biochemical Nomenclature (1970). *Biochemistry*, **9**, 3471–3479; Klyne & Prelog (1960). *Experientia*, **16**, 521–523] and the previous determination are  $-74.8$  (5),  $164.0$  (4),  $-61.7$  (5) and  $-63.2$  (5)°, and  $75.2$ ,  $-164.4$ ,  $63.0$  and  $62.5$ °, respectively. The crystal structure is stabilized by a three-dimensional network of O—H...O and N—H...O hydrogen bonds.

**Experimental.** The compound, purchased from Sigma Chemical Company, was recrystallized from aqueous methanol and gave colourless crystals. A crystal of size  $0.10 \times 0.22 \times 0.28$  mm was used for data collection on a Nicolet P3F four-circle diffractometer with Ni-filtered Cu  $K\alpha$  radiation. Lattice parameters were determined from 25 machine-centred reflections with  $12.2 < 2\theta < 27.4$ °. Of 1143 reflections with  $3 < 2\theta < 115$ ° for one octant, 1046 were independent with  $I > 2.8\sigma(I)$ . Reflections were measured over the index range  $h 0 \rightarrow 7$ ,  $k 0 \rightarrow 14$ ,  $l 0 \rightarrow 15$ , using the  $\omega$ -scan mode with variable scan speed. Two standard reflections ( $00\bar{2}$  and  $\bar{1}2\bar{1}$ ), monitored every 50 measurements, showed no significant variation. Intensities were corrected for Lorentz–polarization effects but not for absorption. Data were adjusted to an approximately absolute scale and an overall  $U$  value of  $0.047$  Å<sup>2</sup>. Blocked-cascade least-squares refinement used *SHELXTL* (Sheldrick, 1985) with all non-H atoms treated anisotropically; H atoms of CH, CH<sub>2</sub> and CH<sub>3</sub> were allowed to ride on their bonded C atoms with a fixed isotropic  $U = 0.06$  Å<sup>2</sup>. The H atoms bonded to N and O atoms

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors (Å<sup>2</sup>  $\times 10^3$ )

	$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$ .			
	x	y	z	$U_{eq}$
N(1)	2730 (5)	9809 (3)	8387 (2)	44 (1)
O(1)	-289 (6)	14403 (3)	8228 (2)	66 (1)
O(2)	2151 (7)	7969 (2)	9254 (2)	73 (1)
O(3)	1893 (5)	8726 (2)	10611 (2)	60 (1)
O(4)	5654 (4)	9654 (3)	8800 (2)	60 (1)
O(5)	1449 (5)	5733 (2)	9340 (2)	65 (1)
C(1)	-140 (7)	13416 (3)	8542 (3)	49 (1)
C(2)	876 (7)	13146 (3)	9298 (3)	51 (1)
C(3)	975 (7)	12139 (3)	9561 (3)	53 (1)
C(4)	57 (6)	11374 (3)	9088 (3)	43 (1)
C(5)	-987 (7)	11666 (3)	8345 (3)	58 (2)
C(6)	-1089 (7)	12669 (3)	8062 (3)	60 (2)
C(7)	170 (6)	10259 (3)	9386 (3)	49 (1)
C(8)	2055 (6)	9804 (3)	9328 (3)	41 (1)
C(9)	2053 (6)	8728 (3)	9709 (3)	45 (1)
C(10)	1854 (11)	7742 (4)	11083 (3)	74 (2)
C(11)	1665 (17)	7861 (5)	12026 (4)	127 (4)
C(12)	4491 (6)	9697 (3)	8198 (3)	50 (1)
C(13)	5000 (9)	9619 (4)	7195 (3)	70 (2)

Fig. 1. The molecular structure of *N*-acetyl-L-tyrosine ethyl ester monohydrate, showing atom numbering.

were located on a difference Fourier map at an advanced stage of anisotropic refinement and their coordinates refined.  $\sum w(\Delta F)^2$  was minimized, where  $w = [\sigma^2(F_o) + 0.00264(F_o)^2]^{-1}$ , in which  $\sigma$  is the standard deviation of observed amplitudes, based on counting statistics. Isotropic extinction parameter  $\chi = 0.012$ . In the last cycle:  $(\Delta/\sigma)_{max} = 0.019$ ;  $\Delta\rho = -0.25$ – $0.23$  e Å<sup>-3</sup>;  $S = 1.32$ ; final  $R = 0.052$  and  $wR$

\* Contribution No. 1136 of the Instituto de Química, UNAM.

Table 2. Bond distances (Å), bond angles (°) and geometry of the hydrogen bonds (Å, °)

N(1)—C(8)	1.453 (5)	N(1)—C(12)	1.337 (6)
O(1)—C(1)	1.378 (5)	O(2)—C(9)	1.197 (5)
O(3)—C(9)	1.314 (5)	O(3)—C(10)	1.464 (5)
O(4)—C(12)	1.226 (5)	C(1)—C(2)	1.376 (6)
C(1)—C(6)	1.392 (6)	C(2)—C(3)	1.378 (6)
C(3)—C(4)	1.393 (6)	C(4)—C(5)	1.380 (6)
C(4)—C(7)	1.528 (6)	C(5)—C(6)	1.381 (6)
C(7)—C(8)	1.517 (6)	C(8)—C(9)	1.518 (5)
C(10)—C(11)	1.384 (8)	C(12)—C(13)	1.507 (7)
C(8)—N(1)—C(12)	121.8 (4)	C(9)—O(3)—C(10)	118.0 (3)
O(1)—C(1)—C(2)	123.3 (4)	O(1)—C(1)—C(6)	117.3 (4)
C(2)—C(1)—C(6)	119.4 (4)	C(1)—C(2)—C(3)	119.8 (4)
C(2)—C(3)—C(4)	122.1 (4)	C(3)—C(4)—C(5)	117.1 (4)
C(3)—C(4)—C(7)	121.7 (4)	C(5)—C(4)—C(7)	121.2 (4)
C(4)—C(5)—C(6)	121.8 (4)	C(1)—C(6)—C(5)	119.8 (4)
C(4)—C(7)—C(8)	114.3 (4)	N(1)—C(8)—C(7)	111.5 (3)
N(1)—C(8)—C(9)	110.3 (3)	C(7)—C(8)—C(9)	110.2 (3)
O(2)—C(9)—O(3)	123.6 (4)	O(2)—C(9)—C(8)	125.0 (4)
O(3)—C(9)—C(8)	111.4 (3)	O(3)—C(10)—C(11)	111.4 (4)
N(1)—C(12)—O(4)	122.7 (4)	N(1)—C(12)—C(13)	116.7 (4)
O(4)—C(12)—C(13)	120.7 (4)		
D—H...A	D...A	H...A	D—H...A
O(5)—H(5B)...O(2)	2.985 (5)	2.31 (5)	153 (5)
N(1)—H(1)...O(1)	3.005 (5)	2.09 (5)	166 (4)
O(1)—H(1A)...O(5 <sup>b</sup> )	2.702 (5)	2.00 (6)	171 (6)
O(5)—H(5A)...O(4 <sup>m</sup> )	2.809 (5)	1.93 (6)	173 (5)

Symmetry code: (i)  $-x, -0.5 + y, 1.5 - z$ ; (ii)  $x, -1 + y, z$ ; (iii)  $-0.5 + x, 1.5 - y, 2.0 - z$ .

= 0.076. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The correct absolute molecular structure has been assigned to be *S* at C(8). All computations were performed on a Nova 4S computer and plots drawn

on a Tektronix plotter with the *SHELXTL* system of programs.

Atomic coordinates are given in Table 1.\* A perspective molecular drawing and the atom labelling are displayed in Fig. 1. Bond distances, angles and hydrogen-bond geometry are given in Table 2.

**Related literature.** The crystal structure for *N*-acetyl-L-tyrosine ethyl ester has been published (Pieret, Durant, Germain & Koch, 1972).

I thank Mr R. A. Toscano for technical assistance.

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55407 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1001]

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*Acta Cryst.* (1993). **C49**, 97–99

## Structure of 1,8-Dimethyl-4-oxo-1,2,2a,3,4,5-hexahydrocyclopenta[*de*]quinoline

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(Received 20 December 1991; accepted 13 April 1992)

**Abstract.**  $C_{13}H_{15}NO$ ,  $M_r = 201.3$ , triclinic,  $P\bar{1}$ ,  $a = 8.929$  (2),  $b = 8.990$  (2),  $c = 8.094$  (2) Å,  $\alpha = 103.79$  (2),  $\beta = 116.71$  (2),  $\gamma = 67.86$  (2)°,  $V = 535.7$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.25$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 5.83$  cm<sup>-1</sup>,  $F(000) = 216$ ,  $T = 295$  K,  $R = 0.049$ ,  $wR = 0.066$ , for 1493 unique observed reflections [ $I > 1.5\sigma(I)$ ]. The phenyl ring *B* is planar with a maximum deviation of  $-0.025$  (3) Å, and all

molecular dimensions are normal. Ring *A* forms a somewhat flattened half-chair conformation. Ring *C* has an envelope conformation. Molecules are linked by an N—H...O hydrogen bond [2.877 (2) Å, 176 (3)°].

**Experimental.** Crystals of the title compound were provided by Professor V. T. Ramakrishnan and K. Joseph Prabahar of the Department of Organic Chemistry, University of Madras. Data were collected for a colourless transparent crystal (0.25 ×

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† Contribution No. 794.