SHORT STRUCTURAL PAPERS

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N-Acetyl-L-norvaline

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Abstract. $C_7H_{13}NO_3$, $M=159{\cdot}15$, crystallizes from a mixture of ethyl acetate and water in the form of orthorhombic bisphenoids: space group $P2_12_12_1$, $a=5{\cdot}805$ (1), $b=9{\cdot}957$ (4), $c=16{\cdot}180$ (6) Å (from oscillation, Weissenberg and precession photographs), $Z=4$, $D_c=1{\cdot}134$, $D_x=1{\cdot}134$ g cm^{-3} (by flotation). The molecules are linked by infinite chains of hydrogen bonds.

Introduction. Equi-inclination multiple-film Weissenberg photographs were taken with Cu $K\bar{\alpha}$ ($\bar{\lambda}=1{\cdot}5418$ Å) radiation for the layers $h0l \rightarrow h5l$ and $0kl \rightarrow 3kl$. The intensities were estimated visually from standard scales. After data reduction and interlayer scaling, 789 independent $|F_o|$ values were obtained of which 195 were too weak for observation. No absorption correction was applied. The phase problem for 165 reflexions with $E \geq 1{\cdot}25$ was solved by *MULTAN* (Main, Woolfson & Germain, 1970). The E map computed for the set with the best ABSFOM = 1·09 gave the position of all non-hydrogen atoms. A structure-factor calculation, based on the fractional coordinates obtained from the E map with an isotropic temperature factor $B=3{\cdot}82$ Å^2 applied to all atoms resulted in $R=\sum||F_o|-|F_c||/\sum|F_o|=0{\cdot}28$. Block-diagonal refinement of the fractional atomic coordinates of the non-hydrogen atoms with anisotropic vibrational parameters and weights: $w=(0{\cdot}39+F_o+0{\cdot}014F_o^2)^{-1}$ gave a final R of 0·113 for 581 observed reflexions. Inclusion of 13 reflexions affected by extinction and the 195 unobserved gave an R of 0·152. The positions of the hydrogen atoms were calculated and checked by the maxima found in the difference map. No hydrogen parameters were refined. The hydrogen atoms were assigned the isotropic vibrational parameters of the heavy atoms to which they are bound. All calculations were performed on a CDC 3300 computer with scattering factors taken from *International Tables for X-ray Crystallography* (1962). The final atomic parameters are given in Table 1.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30627 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

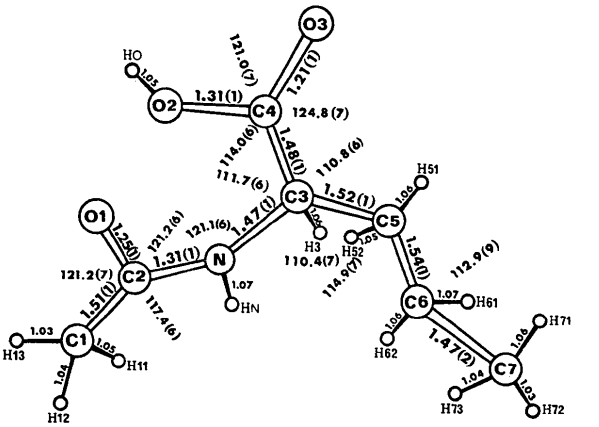

Fig. 1. Bond distances and angles with their e.s.d.'s.

Table 1. *Final atomic positional* ($\times 10^4$ *for non-hydrogen atoms*, $\times 10^3$ *for hydrogen atoms*) *and vibrational parameters*

The estimated standard deviations (except for hydrogen atoms) are in parentheses. The b_{ij} are defined by $T=\exp[-10^4(b_{11}h^2+b_{22}k^2+b_{33}l^2+b_{12}hk+b_{13}hl+b_{23}kl)]$.

	x/a	y/b	z/c	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
N	1686 (9)	5284 (6)	3652 (4)	193 (15)	85 (5)	41 (2)	5 (20)	−12 (10)	7 (7)
O(1)	3881 (10)	6542 (6)	2824 (3)	291 (15)	143 (5)	57 (2)	−80 (23)	−12 (11)	95 (6)
O(2)	4215 (9)	3209 (6)	3156 (4)	242 (15)	155 (6)	67 (2)	−81 (22)	−33 (11)	−101 (7)
O(3)	7288 (8)	3846 (6)	3848 (4)	193 (12)	128 (6)	64 (2)	−17 (22)	−46 (10)	−18 (7)
C(1)	−136 (15)	6319 (11)	2473 (6)	263 (19)	212 (13)	54 (3)	199 (35)	−26 (14)	47 (12)
C(2)	1964 (12)	6033 (7)	2994 (4)	226 (19)	94 (6)	42 (2)	25 (30)	−3 (12)	15 (8)
C(3)	3669 (14)	4876 (7)	4163 (4)	238 (18)	93 (6)	35 (2)	−87 (30)	−35 (12)	16 (8)
C(4)	5247 (12)	3969 (8)	3706 (4)	226 (18)	120 (7)	43 (2)	−94 (30)	−12 (13)	9 (10)
C(5)	2852 (16)	4223 (9)	4958 (5)	417 (29)	124 (8)	39 (2)	−26 (39)	53 (16)	15 (10)
C(6)	1365 (23)	5129 (13)	5516 (6)	569 (38)	249 (17)	52 (3)	262 (62)	77 (22)	−22 (15)
C(7)	2564 (28)	6358 (16)	5777 (9)	877 (69)	297 (20)	98 (6)	403 (88)	−69 (44)	−141 (20)

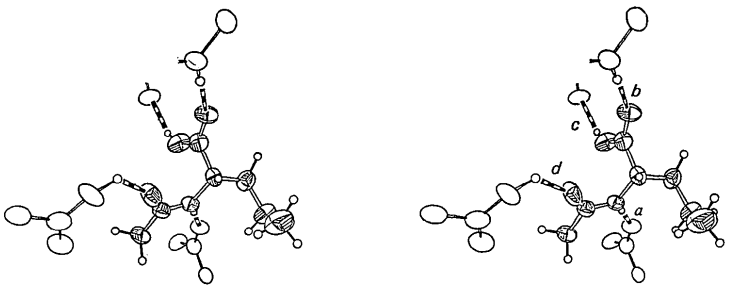

Fig. 2. Stereoscopic (*ORTEP*) diagram of the molecular packing. The non-hydrogen atoms are represented by thermal ellipsoids Keys to the hydrogen bonds are: *a* N–H[x,y,z]...O(3)[$x-1,y,z$], *b* N–H[$x+1,y,z$]...O(3)[x,y,z], *c* O(2)–H[x,y,z]...O(1)[$1-x,y-\frac{1}{2},\frac{1}{2}-z$], *d* O(2)–H[$1-x,y+\frac{1}{2},\frac{1}{2}-z$]...O(1)[$x,y,z$].

Table 1 (*cont.*)

	x/a	*y/b*	*z/c*	*B*
H(N)	15	471	372	3·0 Å²
H(O)	409	237	276	4·7
H(11)	−140	557	246	4·3
H(12)	−101	719	264	4·3
H(13)	36	654	188	4·3
H(3)	467	574	429	3·3
H(51)	427	367	519	4·5
H(52)	174	340	490	4·5
H(61)	86	464	608	6·5
H(62)	−17	532	518	6·5
H(71)	425	620	600	9·6
H(72)	155	676	624	9·6
H(73)	240	706	530	9·6

Discussion. Conformational analysis of the *N*-acetyl-L-norvaline derivatives, where the hydrogen atoms of the acetyl moiety are replaced systematically by different substituents (*e.g.* Cl–, Br–, CH_3–) is of biological interest in order to throw light on the enzymatic hydrolysis (Ötvös, Moravcsik & Mády, 1971) of these compounds. A series of the most promising models has been subjected to X-ray structure analysis. First (as the diploma work of Gy.L.) the structure analysis of the title compound was completed.

The bond lengths and angles shown in Fig. 1 are comparable with the corresponding data found in the literature. The lone pair of the nitrogen atom can interact only with the carbonyl group, thus forming a strong C(2)–N = 1·31 multiple bond. The best plane of the N, O(1), C(1), C(2) and C(3) moiety [$-0{\cdot}202X+0{\cdot}793Y+0{\cdot}572Z=7{\cdot}315$] is almost perpendicular (82·5°) to the plane of the carbonyl group [$0{\cdot}177X+0{\cdot}707Y-0{\cdot}685Z=-0{\cdot}797$]. The molecules are bound together by two infinite chains of hydrogen bonds. One of them is built from N–H···O bonds [H···O = 1·88, N···O = 2·95 Å, N–H···O = 174·7°] approximately in the direction of **a**; the other by O–H···O bonds [H···O = 1·70, O···O = 2·55 Å, O–H···O = 131·7°] which lie roughly in the molecular planes given above (Fig. 2).

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References

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

MAIN, P., WOOLFSON, M. M. & GERMAIN, G. (1970). *MULTAN. A System of Computer Programs for the Automatic Solution of Non-Centrosymmetric Crystal Structures*. Universities of York (England) and Leuven (Belgium).

ÖTVÖS, L., MORAVCSIK, E. & MÁDY, G. (1971). *Biochem. Biophys. Res. Commun.* **44**, 1056–1064.