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Structures of Two Model Peptides: *N*-Acetyl-D,L-valine and *N*-Acetyl-L-valyl-L-leucine

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Abstract. $C_7H_{13}NO_3$, $M_r = 159{\cdot}19$, monoclinic, $P2_1/c$, $a = 6{\cdot}626\ (2)$, $b = 13{\cdot}002\ (5)$, $c = 10{\cdot}031\ (2)$ Å, $\beta = 105{\cdot}37\ (3)°$, $V = 833{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}269$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}93$ cm^{-1}, $F(000) = 344$, $T = 297$ K, final $R = 0{\cdot}058$ for 1429 unique observed reflections. $C_{13}H_{24}N_2O_4$, $M_r = 272{\cdot}35$, monoclinic, $P2_1$, $a = 9{\cdot}458\ (1)$, $b = 9{\cdot}523\ (1)$, $c = 9{\cdot}409\ (1)$ Å, $\beta = 114{\cdot}55\ (1)°$, $V = 770{\cdot}9$ Å^3, $Z = 2$, $D_x = 1{\cdot}173$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}81$ cm^{-1}, $F(000) = 296$, $T = 297$ K, final $R = 0{\cdot}034$ for 1333 unique observed reflections. *N*-Acetyl-D,L-valine and *N*-acetyl-L-valyl-L-leucine have been used as model peptides in solid-state NMR spectroscopy. The X-ray crystal structure determinations were undertaken to provide opportunities for direct comparisons between solid-state NMR spectroscopy and X-ray diffraction.

Introduction. *N*-Acetyl-D,L-valine is a popular model peptide for solid-state NMR spectroscopy (Stark, Haberkorn & Griffin, 1978; Stark, Jelinski, Ruben, Torchia & Griffin, 1983; Tycko, Stewart & Opella, 1986; Ramanathan & Opella, 1988) because it has one peptide linkage per molecule and only two magnetically unique molecules per unit cell. *N*-Acetyl-L-valyl-L-leucine has two peptide linkages per molecule and two unique molecules in the unit cell and can serve as a model system with two adjoining peptide planes for solid-state NMR spectroscopy (Stewart, Tycko & Opella, 1988).

Experimental. *N*-Acetyl-D,L-valine and L-valyl-L-leucine were obtained from Sigma Chemical Company. *N*-Acetyl-L-valyl-L-leucine was prepared by acetylation of L-valyl-L-leucine. Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Table 1 lists data collection parameters for the title compounds. Lattice parameters were refined from 25 reflections in the range $6{\cdot}5 < \theta < 15{\cdot}7°$. Intensities of standard reflections measured every 3000 s of X-ray exposure showed no significant decay. Data corrected for Lorentz and polarization effects and for secondary extinction but not for absorption. Structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H atoms found

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Table 1. *Data collection parameters*

	N-Acetyl-D,L-valine	*N*-Acetyl-L-valyl-L-leucine
Crystal size (mm)	0·34 × 0·18 × 0·08	0·45 × 0·22 × 0·12
No. of reflections measured	2165	2885
hkl range	+8, −18, ±13	±11, +11, ±11
Scan method	ω–2θ	ω–2θ
Scan speed (° min^{-1})	1·3–5	1·7–10
Radiation, λ (Å)	Mo $K\alpha$ 0·71073	Mo $K\alpha$ 0·71073
2θ range (°)	2–55	4–50
No. of observed reflections [$F_o^2 > 3\sigma(F_o^2)$]	1429	1333
R_{int}	0·059	0·011
No. of variables	139	243
Weighting scheme	$w = 1/\sigma^2(F) + 0{\cdot}04F^2 + 3$	$w = 1/\sigma^2(F)$
Max. LS-shift-to-e.s.d. ratio	0·06	0·02
Residual electron density (e Å^{-3})	0·54	0·30
Extinction coefficient	5·6 × 10^{-6}	5·3 × 10^{-6}
R_1	0·058	0·034
R_2	0·069	0·047
S	1·35	1·65

from subsequent difference Fourier synthesis. Refinement by full-matrix least squares to minimize $\sum w(|F_o| - |F_c|)^2$. Non-H atoms refined anisotropically; H-atom positions were refined; H-atom thermal parameters were fixed at 6·0 Å^2. Atomic scattering factors from Cromer & Waber (1974); anomalous-dispersion terms from Ibers & Hamilton (1964). All computer programs from Enraf–Nonius *SDP* (Frenz, 1978).

Discussion. Final positional and equivalent isotropic thermal parameters are listed in Tables 2 and 3.* *ORTEP* drawings (Johnson, 1965) of the molecules appear in Figs. 1 and 2 and *ORTEP* drawings of the unit cells in Figs. 3 and 4. Important backbone torsion angles in the two compounds are as follows.

In *N*-acetyl-D,L-valine, ω_1 the C(1)—C(2)—N—C(3) torsion angle is 174·8 (2), φ_1 C(2)—N—C(3)—C(7) is −136·5 (2), ψ_1 N—C(3)—C(7)—O(3) is 178·2 (2)°.

In *N*-acetyl-L-valyl-L-leucine, ω_1 the C(1)—C(2)—N(1)—C(3) torsion angle is 175·4 (2), φ_1 C(2)—N(1)—C(3)—C(7) is −118·3 (2), ψ_1 N(1)—C(3)—C(7)—N(2) is 131·7 (2); ω_2 the C(3)—C(7)—N(2)—C(8) torsion angle is −175·2 (2), φ_2 C(7)—N(2)—C(8)—C(13) is −63·1 (3)°.

The ω torsion angle, which describes the planarity of a peptide unit, is within ± 5° of 180° for all three peptide planes (one in *N*-acetyl-D,L-valine and two in *N*-acetyl-L-valyl-L-leucine). The φ and ψ torsion angles, which describe the relative orientations of two adjacent peptide planes, indicate a β-sheet structure for *N*-acetyl-L-valyl-L-leucine. The hydrogen-

* Tables of anisotropic thermal parameters, bond distances, bond angles and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52144 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Refined positional parameters for N-acetyl-D,L-valine*

$B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab\cos\gamma + \beta_{13}ac\cos\beta + \beta_{23}bc\cos\alpha)$.

	x	*y*	*z*	B_{eq}(Å^2)
C(1)	0·9579 (4)	0·1341 (2)	0·3350 (3)	4·11 (6)
C(2)	0·8030 (3)	0·2004 (2)	0·3797 (3)	3·06 (5)
C(3)	0·4965 (3)	0·3134 (2)	0·3028 (2)	2·91 (4)
C(4)	0·5435 (4)	0·4300 (2)	0·3136 (3)	3·40 (5)
C(5)	0·7373 (5)	0·4532 (3)	0·4289 (3)	5·22 (7)
C(6)	0·5621 (5)	0·4750 (2)	0·1769 (3)	4·62 (6)
C(7)	0·3042 (3)	0·2911 (2)	0·1857 (2)	3·10 (5)
N	0·6679 (3)	0·2521 (2)	0·2805 (2)	3·01 (4)
O(1)	0·8000 (3)	0·2056 (2)	0·5027 (2)	3·76 (4)
O(2)	0·3039 (3)	0·2343 (2)	0·0904 (2)	4·48 (4)
O(3)	0·1370 (2)	0·3417 (2)	0·1992 (2)	3·89 (4)
H(1)	0·946 (5)	0·139 (3)	0·231 (3)	6·0*
H(1′)	0·923 (5)	0·066 (3)	0·351 (3)	6·0*
H(1″)	1·094 (5)	0·153 (2)	0·385 (3)	6·0*
H(3)	0·469 (5)	0·289 (3)	0·389 (3)	6·0*
H(4)	0·419 (5)	0·463 (2)	0·338 (3)	6·0*
H(5)	0·730 (5)	0·421 (3)	0·512 (3)	6·0*
H(5′)	0·761 (5)	0·532 (3)	0·444 (3)	6·0*
H(5″)	0·853 (5)	0·420 (3)	0·413 (3)	6·0*
H(6)	0·677 (5)	0·442 (3)	0·156 (3)	6·0*
H(6′)	0·589 (5)	0·552 (3)	0·185 (3)	6·0*
H(6″)	0·432 (5)	0·462 (2)	0·103 (3)	6·0*
H(N)	0·675 (5)	0·244 (3)	0·187 (3)	6·0*
H(03)	0·026 (5)	0·322 (3)	0·118 (3)	6·0*

* Thermal parameters for starred atoms were not refined.

Table 3. *Refined positional parameters for N-acetyl-L-valyl-L-leucine*

	x	*y*	*z*	B_{eq}(Å^2)
C(1)	0·2680 (4)	0·500	0·3798 (4)	5·27 (8)
C(2)	0·3237 (2)	0·3521 (3)	0·4258 (2)	3·32 (5)
C(3)	0·2503 (2)	0·1026 (3)	0·3725 (3)	2·92 (5)
C(4)	0·1467 (3)	0·0310 (3)	0·4416 (3)	3·98 (6)
C(5)	0·1619 (4)	−0·1287 (4)	0·4414 (4)	5·95 (8)
C(6)	0·1870 (4)	0·0870 (5)	0·6055 (3)	6·23 (8)
C(7)	0·2213 (2)	0·0462 (2)	0·2108 (2)	2·64 (4)
C(8)	0·3136 (2)	−0·0839 (3)	0·0480 (2)	2·85 (4)
C(9)	0·4572 (2)	−0·1606 (3)	0·0533 (3)	3·53 (5)
C(10)	0·5885 (2)	−0·0659 (3)	0·0532 (3)	3·86 (6)
C(11)	0·7040 (3)	−0·1542 (5)	0·0155 (4)	6·71 (9)
C(12)	0·6717 (3)	0·0124 (4)	0·2062 (4)	5·11 (7)
C(13)	0·1706 (2)	−0·1748 (3)	−0·0346 (2)	3·00 (4)
N(1)	0·2200 (2)	0·2524 (2)	0·3553 (2)	3·05 (4)
N(2)	0·3295 (2)	−0·0365 (2)	0·2003 (2)	2·75 (4)
O(1)	0·4560 (2)	0·3261 (2)	0·5229 (2)	4·83 (5)
O(2)	0·1049 (2)	0·0788 (2)	0·0937 (2)	3·61 (4)
O(3)	0·1007 (2)	−0·1732 (3)	−0·1737 (2)	4·57 (4)
O(4)	0·1368 (2)	−0·2583 (2)	0·0573 (2)	4·17 (4)
H(1)	0·168 (4)	0·511 (6)	0·315 (4)	6·0*
H(1′)	0·296 (4)	0·514 (6)	0·274 (4)	6·0*
H(1″)	0·327 (4)	0·553 (5)	0·451 (4)	6·0*
H(3)	0·365 (4)	0·118 (6)	0·440 (4)	6·0*
H(4)	0·028 (4)	0·060 (5)	0·373 (5)	6·0*
H(5)	0·133 (4)	−0·190 (6)	0·337 (4)	6·0*
H(5′)	0·274 (4)	−0·146 (6)	0·510 (4)	6·0*
H(5″)	0·105 (4)	−0·156 (6)	0·481 (4)	6·0*
H(6)	0·305 (4)	0·040 (6)	0·664 (5)	6·0*
H(6′)	0·128 (4)	0·028 (5)	0·643 (5)	6·0*
H(6″)	0·165 (4)	0·188 (6)	0·592 (4)	6·0*
H(8)	0·294 (4)	0·003 (5)	−0·005 (5)	6·0*
H(9)	0·492 (4)	−0·222 (5)	0·138 (5)	6·0*
H(9′)	0·419 (4)	−0·217 (6)	−0·043 (4)	6·0*
H(10)	0·542 (4)	−0·001 (6)	−0·042 (4)	6·0*
H(11)	0·772 (4)	−0·097 (6)	0·000 (4)	6·0*
H(11′)	0·623 (4)	−0·151 (6)	−0·106 (4)	6·0*
H(11″)	0·749 (4)	−0·232 (5)	0·118 (5)	6·0*
H(12)	0·598 (4)	0·074 (5)	0·244 (5)	6·0*
H(12′)	0·764 (4)	0·076 (6)	0·200 (4)	6·0*
H(12″)	0·718 (4)	−0·066 (6)	0·299 (4)	6·0*
H(N1)	0·124 (4)	0·305 (5)	0·299 (4)	6·0*
H(N2)	0·404 (4)	−0·073 (6)	0·288 (4)	6·0*
H(O4)	0·057 (4)	−0·307 (6)	0·000 (5)	6·0*

* Thermal parameters for starred atoms were not refined.

bonding schemes shown in the unit-cell drawings (Figs. 3 and 4) describe a β-sheet structure for both molecules:

In *N*-acetyl-D,L-valine, H(N) and H(O3) participate in intermolecular hydrogen bonding to O(1) atoms of acetyl groups in neighboring molecules: H(N)···O(1) 2·32 (4), N···O(1) 3·182 (3), H(O3)···O(1) 1·67 (3), O(3)···O(1) 2·630 (2), H(N)—N 0·95 (3), H(O3)—O(3) 0·97 (3) Å. The H(N)—N···O(1) angle is 21 (2), H(O3)—O(3)···O(1) is 9 (2)°.

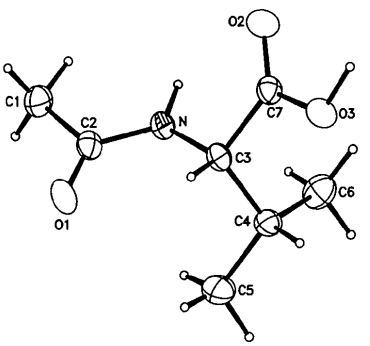

Fig. 1. *ORTEP* drawing of *N*-acetyl-D,L-valine (30% probability thermal ellipsoids).

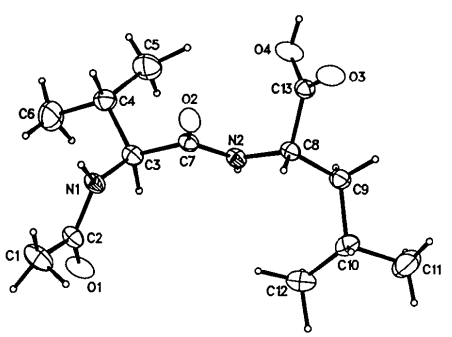

Fig. 2. *ORTEP* drawing of *N*-acetyl-L-valyl-L-leucine (30% probability thermal ellipsoids).

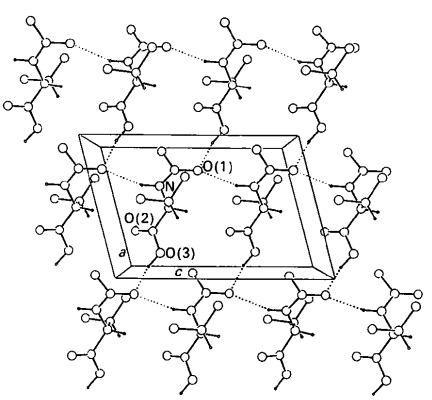

Fig. 3. *ORTEP* drawing of the unit cell of *N*-acetyl-D,L-valine. Only molecules related by the *c*-glide are shown. The packing diagram involving the other two symmetry positions is related to this figure by the crystallographic center of symmetry.

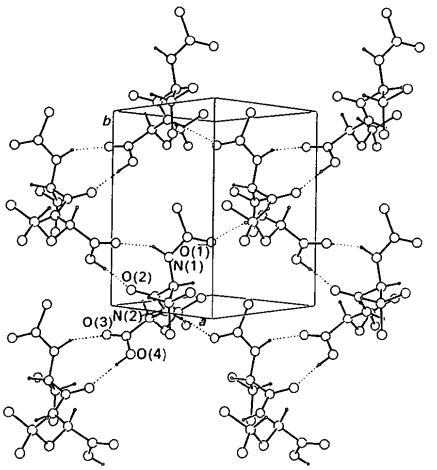

Fig. 4. *ORTEP* drawing of the unit cell of *N*-acetyl-L-valyl-L-leucine viewed perpendicular to the $\bar{1}01$ plane.

In *N*-acetyl-L-valyl-L-leucine, H(N1), H(N2) and H(O4) participate in intermolecular hydrogen bonding to O(3), O(1), and O(2) atoms in neighboring molecules: H(N1)···O(3) 1·97 (3), N(1)···O(3) 2·883 (2), H(N2)···O(1) 1·97 (4), N(2)···O(1) 2·867 (2), H(O4)···O(2) 1·78 (4), O(4)···O(2) 2·637 (2), H(N1)—N(1) 0·98 (4), H(N2)—N(2) 0·91 (3), H″(O4)—O(4) 0·86 (4) Å. The H(N1)—N(1)···O(3) angle is 17 (3), H(N2)—N(2)···O(1) is 6 (3), H(O4)—O(4)···O(2) is 5 (3)°.

The results presented here have enabled us to make direct comparisons for structures determined by solid-state NMR spectroscopy and X-ray diffraction on a model containing a single peptide plane, *N*-acetyl-D,L-valine (Tycko *et al.*, 1986; Ramanathan & Opella, 1989) and on a model containing two peptide planes, *N*-acetyl-L-valyl-L-leucine (Stewart *et al.*, 1988).

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Structure of 9-Chloro-7-(2-fluorophenyl)-5*H*-pyrimido[5,4-*d*][2]benzazepine*

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Abstract. $C_{18}H_{11}ClFN_3$, $M_r = 323{\cdot}8$, monoclinic, $P2_1/n$, $a = 8{\cdot}651$ (2), $b = 10{\cdot}336$ (3), $c = 16{\cdot}855$ (3) Å, $\beta = 90{\cdot}39$ (2)°, $V = 1507{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}427$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}20$ cm^{-1}, $F(000) = 664$, $T = 293$ K, $R = 0{\cdot}036$ for 2425 observed reflections. The angle between the mean planes of the fluorophenyl ring and the fused benzo moiety is 71·7 (3)°. The seven-membered heterocyclic ring adopts a cycloheptatriene-like boat conformation with bow and stern angles of 59·3 (4) and 32·4 (4)°. The pyrimidine ring and the two benzene rings are each planar to within ±0·025 Å. Bond lengths and angles are normal.

Introduction. The title compound (Trybulski, Benjamin, Earley, Fryer, Gilman, Reeder, Walser, Davidson, Horst, Sepinwall, O'Brien & Dairman, 1983) is related to the classical psychoactive 5-phenyl-1,4-benzodiazepin-2-ones such as diazepam,† but differs from these in having the N atom at the 1-position of the seven-membered ring replaced by a C atom. Further, a six-membered hetero-ring is fused across the C1—C4A bond (corresponding to the N1—C2 bond of the benzodiazepine system). Such compounds have been found to have similar pharmacological profiles to the benzodiazepines and have a high affinity for the benzodiazepine receptor *in vitro*. In the present case the affinity is similar to that of diazepam. We now report the crystal structure of the title compound as part of a continuing study of structure–activity relationships for this class of compounds.

Experimental. Crystal size 0·65 × 0·5 × 0·3 mm. X-ray measurements were made on an Enraf–Nonius CAD-4 diffractometer; cell dimensions were determined from the setting angles of 24 reflections in the range $17 < \theta < 25°$; graphite-monochromated Mo $K\alpha$ radiation was used; 5969 reflections were scanned by $\omega/2\theta$ scans up to $\theta = 26°$, 2982 unique, $R_{int} = 0{\cdot}021$, 2425 [$I > 2{\cdot}5\sigma(I)$] reflections were considered observed and used in the analysis, index range h −10 to 10, k 0 to 12, l 0 to 20. Two standard reflections measured every 2 h showed no significant variation in intensity. No absorption corrections were applied. The structure was determined by direct methods and refined on F by full-matrix least-squares calculations using anisotropic thermal parameters for the non-H atoms. The H atoms were located in a subsequent difference synthesis and refined isotropically. Weights $w = 1/[\sigma^2(F) + 0{\cdot}005 \times F^2]$ were used in the least-squares refinement. The refinement converged to $R = 0{\cdot}036$, $wR = 0{\cdot}067$, with maximum shift/e.s.d. ratio of 0·3. The residual electron density in a final difference map was within ±0·3 e Å^{-3}. No correction for secondary extinction was applied.

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Computations were carried out on the University of Birmingham Honeywell DPS 8/70 computer and on the CDC 7600 at the University of Manchester Regional Computer Centre with the *SHELX*76 (Sheldrick, 1976) and *PLUTO* (Motherwell & Clegg, 1978) programs.

Discussion. Fig. 1 illustrates the atomic numbering scheme. Atomic coordinates are listed in Table 1.‡ Bond lengths, bond angles and selected torsion angles are in Table 2.

* Contribution from the Crystallography Unit, Universities of Aston and Birmingham, England.

† 7-Chloro-1,3-dihydro-1-methyl-5-phenyl-2*H*-1,4-benzodiazepin-2-one. Marketed as Valium (Roche).

‡ 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 52208 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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