metrically the C(2)–C(8)–C(14) = 117·3 (3)° angle. S(11) is eclipsed with C(8). The short C(10)–N(12) bond length [1·325 (3) Å] indicates that the N lone pair of electrons is delocalized on this group. The S^{11}–$C(sp^3)$ = 1·830 (2) and S^{11}–$C(sp^2)$ = 1·767 (2) Å bond lengths maintained by S(9) – in accordance with the different hybridization of the C atoms – exhibit significant difference (Δ = 0·063 Å), underscoring the observations of Argay, Kálmán, Lazar, Ribár & Tóth (1977, and references therein). The length [1·658 (2) Å] of the polarized S^{11}=C double bond agrees with data found in the literature, cf. for example N-vinyl-2-thiopyrrolidone [1·659 (3) Å] reported by Kálmán, Argay & Cser (1976). The centre-of-symmetry-related enantiomers are bound together by hydrogen-bond pairs having the parameters

	$D\cdots A$ (Å)	$H\cdots A$ (Å)	$DH\cdots A$ (°)
N(12)–H(12)$\cdots$O(13)(1–x, –y, –z)	2·898 (2)	1·99 (2)	160 (1).

References

ARGAY, GY., KÁLMÁN, A., LAZAR, D., RIBÁR, B. & TÓTH, G. (1977). Acta Cryst. B33, 99–105.
ARGAY, GY., KÁLMÁN, A., PERJÉSI, P. & SZABÓ, D. (1987). Acta Cryst. C43, 324–326.
CAHN, R. S., INGOLD, L. K. & PRELOG, V. (1956). Experientia, 12, 81–94.
CREMER, D. & POPLE, J. A. (1975). J. Am. Chem. Soc. 97, 1354–1358.
Enraf–Nonius (1983). Structure Determination Package. Enraf–Nonius, Delft, The Netherlands.
KÁLMÁN, A., ARGAY, GY. & CSER, F. (1976). Acta Cryst. B32, 2672–2674.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). MULTAN82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data. Univs. of York, England, and Louvain, Belgium.
PERJÉSI, P., FÖLDESI, A., SZABÓ, D., ZSCHUNKE, A. & MÁK, M. (1987). Chem. Ber. 120, 1449–1450.
PERJÉSI, P., SZABÓ, D., BATTA, GY. & FÖLDESI, A. (1987). Tetrahedron Lett. pp. 571–572.
WALKER, N. & STUART, D. (1983). Acta Cryst. A39, 158–166.

Structure of L-Tyrosyl-L-leucine Monohydrate

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Abstract. $C_{15}H_{22}N_2O_4.H_2O$, M_r = 312·37, monoclinic, $P2_1$, a = 5·577 (2), b = 8·686 (2), c = 16·228 (2) Å, β = 92·63 (2)°, V = 785 (1) Å^3, Z = 2, D_m = 1·34, D_x = 1·32 Mg m^{-3}, Cu $K\alpha$, λ = 1·54184 Å, μ = 0·78 mm^{-1}, F(000) = 320, T = 293 K. The final R value for 1607 observed reflections $[I_o \geq 3\sigma(I_o)]$ is 0·039. The terminal N1 is protonated and the dipeptide exists as a zwitterion. The crystal structure is stabilized by extensive hydrogen-bonding interactions involving N and O atoms, with N$\cdots$O in the range 2·65 (1)–2·95 (1) Å and O$\cdots$O in the range 2·60 (1)–2·78 (1) Å.

Introduction. We report here the structure of the title compound as part of our investigation on peptides which are relevant to the problem of nucleotide–peptide interactions.

Experimental. The dipeptide was purchased from Sigma Chemicals and used without further purification. Platey crystals were obtained by evaporation of an aqueous solution containing 10 mM peptide and 5% Me$_2$SO at room temperature. The size of the crystal used for X-ray data collection was 0·10 x 0·2 x 0·5 mm. The cell parameters were initially obtained from Weissenberg photographs and were later refined on a CAD-4 diffractometer using 25 high-angle (10 $\leq \theta \leq$ 38°) reflections. Three-dimensional intensity X-ray data were collected up to θ = 70° using ω–2θ scans. The crystal was found to be stable to X-rays; standard reflections monitored periodically during data collection showed no significant intensity decrease. The ranges of the indices are h = –6 to 6, k = 0 to 10 and l = 0 to 19. A total of 1607 reflections were considered observed $[I \geq 3\sigma(I)]$ out of 1737 collected. Lorentz and polarization corrections were applied but no absorption correction was made.

The structure was solved using MULTAN11/82 (Germain, Main & Woolfson, 1971). Non-hydrogen atoms were refined anisotropically using full-matrix methods. All hydrogen atoms were located from difference Fourier maps and refined isotropically. The final R = 0·039 and wR = 0·042, where the w is $1/[\sigma(F_o)]^2$. The function minimized was $\sum w(|F_o| - |F_c|)^2$. $(\Delta/\sigma)_{max}$ = 0·13; $\Delta\rho$ variations in a final dif-

Table 1. *Positional parameters and their estimated standard deviations*

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	$B_{eq}(\text{Å}^2)$
O1	0.9581 (4)	0.360	1.2357 (2)	2.93 (5)
O2	0.9759 (5)	−0.0895 (4)	1.2462 (2)	3.67 (5)
O3	1.3243 (4)	−0.0095 (3)	1.3028 (2)	3.74 (6)
O4	1.0165 (5)	0.1967 (3)	0.8653 (2)	3.85 (6)
OW	0.4145 (5)	0.2314 (4)	0.7124 (2)	4.24 (6)
N1	0.5731 (5)	0.5037 (4)	1.1791 (2)	2.94 (6)
N2	0.7588 (4)	0.1768 (3)	1.3042 (2)	2.27 (5)
C1	0.5331 (6)	0.3457 (4)	1.2115 (2)	2.24 (6)
C2	0.7715 (5)	0.2938 (4)	1.2521 (2)	1.95 (5)
C3	0.9725 (5)	0.1135 (4)	1.3464 (2)	2.14 (6)
C4	1.1013 (6)	−0.0035 (4)	1.2935 (2)	2.34 (6)
C5	0.4385 (6)	0.2372 (5)	1.1431 (2)	2.64 (6)
C6	0.5934 (6)	0.2284 (4)	1.0695 (2)	2.33 (6)
C7	0.5338 (6)	0.3085 (5)	0.9976 (2)	2.75 (7)
C8	0.6706 (6)	0.3017 (4)	0.9293 (2)	2.73 (6)
C9	0.8764 (6)	0.2102 (4)	0.9317 (2)	2.49 (6)
C10	0.9375 (6)	0.1289 (5)	1.0021 (2)	2.76 (7)
C11	0.8007 (6)	0.1370 (4)	1.0705 (2)	2.47 (6)
C12	0.9143 (6)	0.0378 (4)	1.4277 (2)	2.50 (6)
C13	0.8543 (7)	0.1445 (5)	1.4984 (2)	3.07 (7)
C14	0.886 (1)	0.0621 (7)	1.5800 (3)	5.6 (1)
C15	0.6087 (8)	0.2170 (7)	1.4886 (3)	5.8 (1)

Fig. 1. Molecular structure and atomic numbering.

Table 2. *Molecular geometry of* L-*tyrosyl-*L-*leucine*

Numbers in parentheses are estimated standard deviations.

(*a*) Bond distances (Å)

O1—C2	1.232 (4)	C5—C6	1.508 (5)
O2—C4	1.260 (4)	C6—C7	1.386 (5)
O3—C4	1.247 (4)	C6—C11	1.402 (5)
O4—C9	1.365 (4)	C7—C8	1.375 (5)
N1—C1	1.490 (5)	C8—C9	1.395 (5)
N2—C2	1.325 (4)	C9—C10	1.374 (5)
N2—C3	1.455 (4)	C10—C11	1.376 (5)
C1—C2	1.525 (4)	C12—C13	1.524 (5)
C1—C5	1.531 (5)	C13—C14	1.510 (6)
C3—C4	1.530 (5)	C13—C15	1.509 (6)
C3—C12	1.523 (5)		

(*b*) Bond angles (°)

C2—N2—C3	121.5 (3)	C5—C6—C7	121.2 (4)
N1—C1—C2	106.5 (3)	C5—C6—C11	121.4 (3)
N1—C1—C5	111.3 (3)	C7—C6—C11	117.4 (4)
C2—C1—C5	113.3 (3)	C6—C7—C8	122.4 (3)
O1—C2—N2	124.8 (3)	C7—C8—C9	119.2 (3)
O1—C2—C1	119.7 (3)	O4—C9—C8	121.8 (4)
N2—C2—C1	115.5 (3)	O4—C9—C10	118.9 (4)
N2—C3—C4	112.5 (3)	C8—C9—C10	119.2 (3)
N2—C3—C12	111.6 (3)	C9—C10—C11	121.3 (3)
C4—C3—C12	108.9 (3)	C6—C11—C10	120.4 (3)
O2—C4—O3	124.9 (3)	C3—C12—C13	116.9 (3)
O2—C4—C3	118.3 (3)	C12—C13—C14	110.6 (4)
O3—C4—C3	116.9 (3)	C12—C13—C15	113.8 (3)
C1—C5—C6	114.6 (3)	C14—C13—C15	111.0 (4)

(*c*) Conformational angles (in °; average e.s.d. 0.4°)

N1—C1—C2—N2	ψ	161.8
C1—C2—N2—C3	ω	179.0
C2—N2—C3—C4	φ_2	−83.6
N1—C2—C5—C6	χ^1	55.2
C1—C5—C6—C7	χ^{21}	−101.0
C1—C5—C6—C11	χ^{22}	80.2
N2—C3—C12—C13	χ^4	−72.7
C3—C12—C13—C14	χ^{21}	−160.3
C3—C12—C13—C15	χ^{22}	73.9

(*d*) Hydrogen-bond geometry; average e.s.d.'s in distances 0.01 Å and in angles 5°

A—H···B	A···B (Å)	A—H···B (°)	Unit-cell translation of atom B	Symmetry code
N1—H1···O4	2.95	133	2 0 2	ii
N1—H3···OW	2.65	163	1 0 2	ii
N2—H···O3	2.91	159	−1 0 0	i
O4--H···O2	2.60	151	2 0 2	ii
OW—H1···O2	2.78	161	1 0 2	ii
OW—H2···O3	2.70	157	2 0 2	ii

Symmetry code: (i) x, y, z; (ii) $-x, \frac{1}{2}+y, -z$.

ference Fourier map were within ±0.21 e Å⁻³. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All computations were performed on a PDP 11/44 computer using the structure determination package supplied by Enraf–Nonius (1979).

Discussion. The final positional and thermal parameters are given in Table 1* and molecular geometry is listed

* 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 51175 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

in Table 2. The molecular structure and atomic numbering scheme are shown in Fig. 1. The dipeptide exists as a zwitterion with the terminal N atom protonated. The peptide bond is planar with the conformational angle $\omega = 179.0$ (3)° (IUPAC–IUB Commission on Biochemical Nomenclature, 1970).

This side-chain conformation (Table 2c) of tyrosine is similar to that found in the structures of L-tyrosyl-L-glutamic acid (Pandit, Seshadri & Viswamitra, 1984) and L-tyrosyl-L-valine (Ramakrishnan, Seshadri & Viswamitra, 1984). The molecular interactions seen in the crystal structure (Table 2d and Fig. 2) are also similar to those seen in L-tyrosyl-L-valine
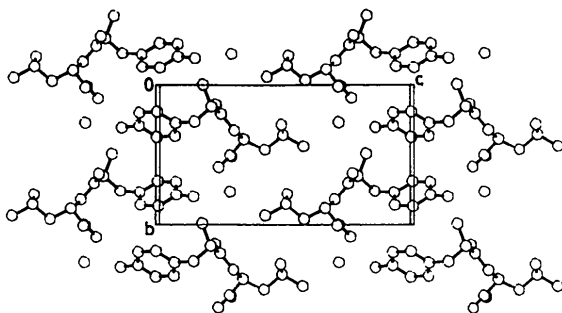
Fig. 2. View of crystal packing along a.

(Ramakrishnan, Seshadri & Viswamitra, 1984). Fig. 2 shows that the tyrosyl rings do not stack in the crystal lattice. The lone water molecule forms hydrogen bonds with the amino and carboxyl terminal atoms of the dipeptide molecules (Table 2d).

References

Enraf–Nonius (1979). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A27, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

IUPAC–IUB COMMISSION ON BIOCHEMICAL NOMENCLATURE (1970). *J. Mol. Biol.* 52, 1–17.

PANDIT, J., SESHADRI, T. P. & VISWAMITRA, M. A. (1984). *Acta Cryst.* C40, 169–170.

RAMAKRISHNAN, B., SESHADRI, T. P. & VISWAMITRA, M. A. (1984). *Acta Cryst.* C40, 1248–1250.

Structure of L-Arginyl-L-aspartic Acid Dihydrate

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Abstract. $C_{10}H_{19}N_5O_5.2H_2O$, $M_r = 325.32$, monoclinic, $P2_1$, $a = 12.029$ (2), $b = 4.904$ (2), $c = 13.215$ (2) Å, $\beta = 107.68$ (2)°, $V = 743$ (1) Å^3, $Z = 2$, $D_m = 1.45$, $D_x = 1.45$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1.54184$ Å, $\mu = 1.01$ mm^{-1}, $F(000) = 348$, $T = 293$ K. The final R value for 1277 observed reflections $[I_o \geq 3\sigma(I_o)]$ is 0.031. The dipeptide exists as a zwitterion. The arginyl side-chain conformation is similar to that found in arginyl-glutamic acid [Pandit, Seshadri & Viswamitra (1983). *Acta Cryst.* C39, 1669–1672]. The guanidyl group forms a pair of hydrogen bonds with oxygen atoms of the backbone carboxyl group. The crystal structure is also stabilized by H-bonding interactions involving both water molecules.

Introduction. The arginine side chain is expected to play an important role in the specific recognition process between proteins and nucleic acids. In view of this interest we have initiated a study of arginine-containing peptides (Pandit, Seshadri & Viswamitra, 1983) and their complexes with nucleotides. We report here the crystal structure of L-arginyl-L-aspartic acid as part of this study.

Experimental. The dipeptide was purchased from Sigma Chemicals and used without further purification. Thin needle crystals of the peptide alone appeared in our attempts to complex it with oligonucleotides, using 2-methyl-2,4-pentanediol (MPD) vapour-diffusion methods. The size of the crystal used for X-ray data collection was $0.05 \times 0.1 \times 0.5$ mm. The cell parameters were initially determined from Weissenberg photographs and were later refined on a CAD-4 diffractometer using 25 high-angle $(10 \leq \theta \leq 51°)$ reflections. Three-dimensional intensity data were collected up to $\theta = 70°$ using ω–2θ scans. Three standard reflections during the data collection showed no significant variation in their intensities, indicating that the crystal was stable to X-rays. The range of indices was $h = 0$ to 14, $k = 0$ to 5 and $l = -15$ to 14. A total of 1277 reflections was considered observed $|I \geq 3\sigma(I)|$ out of 1587 collected. Lorentz and polarization corrections were applied but no absorption correction was made.

The structure was solved using *MULTAN*11/82 (Germain, Main & Woolfson, 1971). Non-hydrogen atoms were refined anisotropically using full-matrix methods. All hydrogen atoms were located from difference Fourier maps and refined isotropically. The final $R = 0.031$ and $wR = 0.029$, where the weight w is $1/|\sigma(F_o)|^2$. The function minimized was $\sum w(|F_o| - |F_c|)^2$. The average $\Delta/\sigma = 0.02$; $\Delta\rho$ variations in the