are the two C–C bonds involving the phenyl-substituted C(1) [C(1)–C(2): 1·545 (6); C(1)–C(5): 1·549 (5) Å]. However, the third C–C bond in the furan ring, C(2)–C(3), is considerably shorter than this at 1·497 (6) Å. The angles within the ring range from a low of 96·2 (4)° at C(1) to a high of 108·1 (4)° at the oxygen.

These values are fairly typical of substituted furans, where disubstitution of any moiety at a carbon center tends to decrease the angle at that carbon. In α-2-ethyl-5-methyl-3,3-diphenyltetrahydrofuran (Singh & Ahmed, 1969), the angle at the phenyl-disubstituted carbon is slightly larger than that reported here, 99·4 (2)°, while the angle at the oxygen is also larger, 110·8 (2)°. However, the two C–O bonds in that structure, while equivalent, are considerably longer than those found in (I), averaging 1·445 (4) Å. The C–C bond not involving the phenyl-substituted carbon in that compound is also significantly shorter than the others in the furan ring.

The small value of the bond angle inside the furan ring at C(1) is consistent with other disubstituted carbon atoms in furan rings, and is not limited to those with bulky substituents. The angles at the substituted carbons in 3,3,4,4-tetrahydrofurantetrol (Mighell & Jacobson, 1964) are 6° smaller than those at the other two carbon centers in the ring, and 10° smaller than the angle at the O atom. These angles, at 100·3 (3)°, are slightly larger than in phenyl-substituted furans.

The angle at the oxygen atom tends to be larger than the four internal carbon angles in similar compounds. In 2,3,3,4,4,5-hexamethoxytetrahydrofuran (Iten, Weber & Eugster, 1976) the C–O–C angle is 112·6°, 7° larger than any of the O–C–C or C–C–C angles. Similarly, in (2*S*,4*R*,5*R*)-2-carboxymethyl-5-carboxy-2,4,5-trimethyl-2,3,4,5-tetrahydrofuran (Kirfel, Will, Wiedenfeld & Roeder, 1980), the C–O–C angle is 5·3° larger than all other internal ring angles.

There are no unusually close non-bonded contacts between molecules of (I).

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References

ARNOLD, D. R., FAHIE, B. J., LAMONT, L. J., WIERZCHOWSKI, J. & YOUNG, K. M. (1987). In the press.
CAMERON, T. S. & CORDES, R. E. (1979). *Acta Cryst.* B**35**, 748–750.
ITEN, P. X., WEBER, H. P. & EUGSTER, C. H. (1977). *Helv. Chim. Acta*, **60**, 410–416.
KARLE, J. & HAUPTMAN, H. (1958). *Acta Cryst.* **11**, 264–266.
KIRFEL, A., WILL, G., WIEDENFELD, H. & ROEDER, E. (1980). *Cryst. Struct. Commun.* **9**, 363–369.
MIGHELL, A. D. & JACOBSON, R. A. (1964). *Acta Cryst.* **17**, 1554–1560.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
SINGH, P. & AHMED, F. R. (1969). *Acta Cryst.* B**25**, 2401–2408.

Acta Cryst. (1988). C**44**, 281–285

Structure of the Dipeptide L-Prolyl-L-lysine Acetate. A New Conformation of the Lysine Side Chain

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Abstract. $C_{11}H_{22}N_3O_3.C_2H_3O_2$, $M_r = 303{\cdot}36$, monoclinic, $P2_1$, $a = 15{\cdot}712$ (3), $b = 8{\cdot}927$ (2), $c = 5{\cdot}484$ (2) Å, $\beta = 94{\cdot}31$ (2)°, $V = 767$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}31$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}109$ mm^{-1}, $F(000) = 328{\cdot}0$, room temperature. The crystal structure was solved by direct methods and refined to an R value of 0·043 using the full-matrix least-squares method for 907 observed reflections. The molecule exists as a zwitterion with the N atom of the lysine side chain protonated. The pyrrolidine ring has an envelope conformation (N1, C*A*1, C*B*1 and C*D*1 atoms in the same plane and C*G*1 in position *exo* to

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C1). The $CG1$ atom shows the largest temperature factor (6·32 Å^2) indicating considerable motion of this atom. Furthermore, some electron density was found on the other side of the ring plane which indicates a minor *endo* position for $CG1$. The peptide backbone is folded. The side chain of the lysine residue shows a conformation (g^-,*t*,*t*,g^-) which has not been previously observed.

Introduction. The structure determination of L-prolyl-L-lysine acetate is part of a general study of oligopeptides containing basic amino acid residues, related to histones and other basic proteins that interact with DNA. The L-Pro-L-Lys sequence is repeated about ten times (depending on species and tissues) in the *C*-terminal region of histone H1 (Elgin & Weintraub, 1975).

Experimental. The title compound was supplied by Bachem AG and used without further purification. It was crystallized by vapour diffusion of 2-propanol into a 2-propanol aqueous solution of the dipeptide. Plate-like crystals appeared after 1 d. One crystal 1·15 × 1·0 × 0·65 mm was mounted in a capillary. Data recorded on a Philips PW1100 diffractometer, Mo $K\alpha$ radiation, graphite monochromator. Cell parameters from 25 reflections ($4 < \theta < 9°$). ω-scan technique, three reflections measured every 2 h as intensity control, no significant differences. Lp corrections applied but no absorption correction. 2498 independent reflections ($\theta < 25°$), 907 with $I > 2{\cdot}5\sigma(I)$ (index range: h −17 to 17, k 0 to 10, l 0 to 16). Structure determined by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Anisotropic full-matrix least-squares refinement on F with *SHELX*76 (Sheldrick, 1976). f, f', f'' for all atoms from *International Tables for X-ray Crystallography* (1974). All H atoms visible in difference Fourier maps were included at calculated positions and refined with geometrical constraints and overall isotropic temperature factor. Final $R = 0{\cdot}043$, $wR = 0{\cdot}044$, weighting scheme $[\sigma^2(F) + 0{\cdot}000467|F|^2]^{-1}$, max. shift/e.s.d. = 0·59, max. and min. heights in final difference Fourier synthesis not considering the disordered $CG1$ position: 0·209 and −0·21 e Å^{-3}. A VAX 750 computer was used.

Discussion. The molecular structure with the atomic numbering is shown in Fig. 1 and the final positional and thermal parameters are listed in Table 1.* Bond lengths and bond angles are listed in Table 2 and conformation angles (IUPAC–IUB Commission on Biochemical Nomenclature, 1970) in Table 3.

(a) Backbone conformation

According to Suresh & Vijayan (1985), the backbone conformation of dipeptides can be described just by the φ_2 angle because in general $\psi_1 \sim 180$, $\omega \sim 180$, $\psi_2' \sim 0$ and $\psi_2'' \sim 180°$. However, in our case the L-Pro-L-Lys acetate structure shows a considerable departure of ψ_2' from 0°, although it agrees with the values found in the terminal carboxylate of other dipeptide structures (Suresh & Vijayan, 1985). The φ_2 value indicates a partially folded conformation of the backbone in L-Pro-L-Lys acetate and it is in the most favourable range taking into account the steric factors (Suresh & Vijayan, 1985). Such a conformation may also be influenced by packing forces on the side chains and terminal groups, which determine hydrophobic and hydrophilic zones as described below.

The ω torsion angle indicates the conformation around the peptidic bond. A considerable deviation from planarity is found ($\omega = 167°$) which is a characteristic of peptide bonds surrounding a proline residue owing to the steric hindrance between the peptidic backbone and the pyrrolidine ring. The ω value agrees with the literature data, in particular with that of the L-Pro-L-Val.H_2O structure (Narasimhan, Chacko & Swaminathan, 1982).

(b) The proline ring

Bond lengths and angles of the pyrrolidine group in the present structure fall between the values of

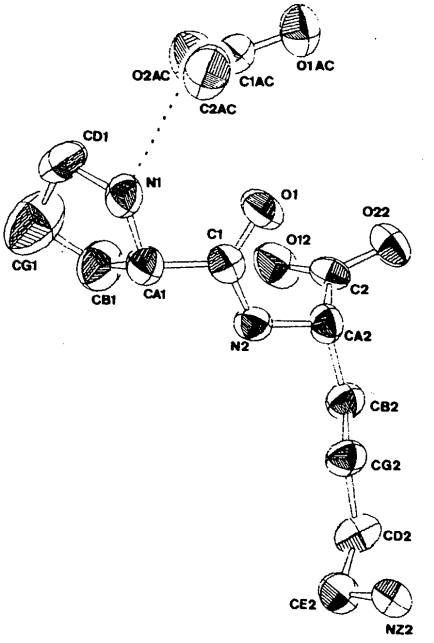

Fig. 1. Molecular structure and atom numbering. Thermal ellipsoids of 90% probability are shown.

* 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 44406 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* (Å²)

	x	*y*	*z*	B_{eq}*
C1*AC*	0·1199 (4)	0·8980	0·6319 (11)	3·10
C2*AC*	0·1190 (4)	0·7565 (13)	0·4759 (13)	4·57
O1*AC*	0·1521 (3)	0·8852 (10)	0·8511 (7)	3·64
O2*AC*	0·0885 (3)	0·0152 (10)	0·5444 (9)	4·92
N1	0·1422 (3)	0·1444 (10)	0·0978 (8)	2·86
C*A*1	0·2150 (3)	0·2441 (11)	0·0333 (10)	2·69
C*B*1	0·1722 (4)	0·3936 (12)	0·9561 (12)	3·92
C*G*1	0·0843 (5)	0·3506 (13)	0·8622 (16)	6·32
C*D*1	0·0605 (4)	0·2254 (12)	0·0336 (12)	4·12
C1	0·2782 (4)	0·2598 (11)	0·2579 (10)	2·49
O1	0·2621 (3)	0·2105 (10)	0·4571 (7)	3·86
N2	0·3490 (3)	0·3350 (10)	0·2195 (8)	2·35
C*A*2	0·4055 (3)	0·3863 (11)	0·4282 (8)	2·38
C*B*2	0·4849 (3)	0·4627 (11)	0·3345 (10)	2·44
C*G*2	0·5388 (3)	0·3620 (11)	0·1844 (9)	2·56
C*D*2	0·6104 (3)	0·4489 (11)	0·0771 (11)	3·19
C*E*2	0·6514 (4)	0·3659 (11)	0·8743 (10)	2·86
N*Z*2	0·7037 (3)	0·2336 (10)	0·9657 (7)	2·42
C2	0·3600 (3)	0·4995 (11)	0·5829 (10)	2·26
O12	0·3059 (3)	0·5852 (10)	0·4814 (7)	3·60
O22	0·3839 (3)	0·5047 (10)	0·8052 (6)	2·94

* $B_{eq} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

N1–C*A*1	1·511 (7)	C*B*1–C*G*1	1·487 (9)
C*A*1–C*B*1	1·539 (9)	C*G*1–C*D*1	1·525 (10)
C*A*1–C1	1·530 (7)	C*D*1–N1	1·492 (8)
C1–O1	1·222 (6)	C*B*2–C*G*2	1·520 (7)
C1–N2	1·328 (7)	C*G*2–C*D*2	1·521 (7)
N2–C*A*2	1·469 (6)	C*D*2–C*E*2	1·519 (8)
C*A*2–C*B*2	1·543 (7)	C*E*2–C*Z*2	1·503 (7)
C*A*2–C2	1·530 (8)	C2*AC*–C1*AC*	1·525 (10)
C2–O12	1·244 (7)	O1*AC*–C1*AC*	1·273 (7)
C2–O22	1·249 (6)	O2*AC*–C1*AC*	1·238 (8)

N1–C*A*1–C*B*1	104·6 (4)	C*A*1–C*B*1–C*G*1	104·2 (6)
C*B*1–C*A*1–C1	112·7 (5)	C*B*1–C*G*1–C*D*1	103·9 (6)
N1–C*A*1–C1	108·8 (5)	C*G*1–C*D*1–N1	104·7 (5)
C*A*1–C1–O1	121·4 (5)	C*D*1–N1–C*A*1	108·2 (5)
C*A*1–C1–N2	114·8 (5)	C*A*2–C*B*2–C*G*2	114·6 (5)
O1 C1 N2	123·8 (5)	C*B*2–C*G*2–C*D*2	111·5 (5)
C1–N2–C*A*2	119·9 (4)	C*G*2–C*D*2–C*E*2	113·6 (5)
N2–C*A*2–C*B*2	109·6 (4)	C*D*2–C*E*2–N*Z*2	113·0 (4)
N2–C*A*2–C2	110·9 (4)	O1*AC*–C1*AC*–C2*AC*	116·2 (6)
C*B*2–C*A*2–C2	108·4 (5)	O2*AC*–C1*AC*–C2*AC*	119·7 (6)
C*A*2–C2–O12	119·3 (5)	O2*AC*–C1*AC*–O1*AC*	124·1 (7)
C*A*2–C2–O22	116·6 (5)		
O12–C2–O22	124·0 (5)		

pyrrolidine rings in L-Pro-L-Val.H_2O (Narasimhan, Chacko & Swaminathan, 1982) and L-Pro-L-Tyr-L-Ile-L-Leu (Cotrait, Geoffre, Hospital & Precigoux, 1979).

In comparison with structures which have the pyrrolidine N atom unprotonated (Ashida & Kakudo, 1974), it may be seen that the C*A*1–N1 and N1–C*D*1 bonds are longer and C*A*1–N1–C*D*1 angle is smaller when this N atom is protonated.

The proline-ring dihedral angles agree fairly well with those of proline in the tetrapeptide L-Pro-L-Tyr-L-Ile-L-Leu (Cotrait, Geoffre, Hospital & Precigoux, 1979) (see Table 3).

Table 3. *Conformational angles* (°) *of the dipeptide and comparison of conformation angles of the* L-*lysine side chain with other oligopeptide structures which contain this amino acid*

C*D*1–N1–C*A*1–C*B*1		6·25	C1–N2–C*A*2–C2	ω_2	−63·34
N1–C*A*1–C1–N2	ψ_1	174·73	N2–C*A*2–C2–O22	ψ_2'	151·23
C*A*1–C*B*1–C*G*1–C*D*1	ω	166·58	N2–C*A*2–C2–O12	ψ_2''	−32·71
N1–C*A*1–C*B*1–C*G*1	χ_1^1	−27·51	C*B*1–C*G*1–C*D*1–N1	χ_1^1	−34·12
C*A*1–C*B*1–C*G*1–C*D*1	χ_1^2	37·99	C*G*1–C*D*1–N1–C*A*1	χ_1^2	16·79

		(1)	(2)	(3)	(4)	(5)	(6)	(7)
N2–C*A*2–C*B*2–C*G*2	χ_2^1	−60·90	−67·7	−56·4	64·9	179·1	53·6	−61·27
C*A*2–C*B*2–C*G*2–C*D*2	χ_2^2	174·18	179·6	−176·0	−179·1	168·1	179·6	178·04
C*B*2–C*G*2–C*D*2–C*E*2	χ_2^3	−164·80	165·5	−171·1	−179·5	172·2	176·3	178·26
C*G*2–C*D*2–C*E*2–N*Z*2	χ_2^4	−70.25	161·2	179·2	−177·7	−73·1	−73·3	172·64

References: (1) L-Pro-L-Lys acetate, this work; (2) L-Lys-L-Asp (Bhat & Vijayan, 1976); (3) L-Lys.HCl.2H_2O (Koetzle, Lehmann, Verbist & Hamilton, 1972); (4) Ac-Gly-L-Lys-Me ester (Salunke & Vijayan, 1982); (5) L-Lys $PtCl_6$ (L'Haridon, Lang, Pastuszak & Dobrowolski, 1978); (6) L-Lys SO_4 (Capasso, Mattia, Mazzarella & Zagari, 1983); (7) L-Lys-L-Tyr (Urpi, Coll & Subirana, 1988).

The conformation of the pyrrolidine ring is C_s—Cγ-*exo* [notation of Ashida & Kakudo (1974)] with Cγ (C*G*1) 0·553 Å below the plane defined by the four remaining atoms of the ring whereas C1 is 1·146 Å above it. The C*G*1 atom was refined in this position with a site occupation factor (s.o.f.) of 1, but a residual electron density of 0·35 e Å^{-3} was found in the *endo* position (*cis* with respect to C1). Better convergence was obtained by refining the C*G*1 atom fully localized in the *exo* position rather than with a s.o.f. partitioned between *exo* and *endo*. Less than 15% occupation was estimated for the *endo* site. Besides this feature of an additional *endo* position, the C*G*1 atom has the highest temperature factor of the structure and the largest standard deviations for its coordinates (see Table 1).

As found in this structure, in most proline rings in the solid state the Cγ atom exhibits the largest temperature factors, and the ellipsoids of thermal motion show that the Cγ atom moves perpendicular to the ring plane (Arnoux, Prangé & Pascard, 1977; Ashida & Kakudo, 1974). Sometimes the Cγ atom appears in such a state of disorder that Fourier maps show two peaks for Cγ with variable s.o.f. (Narasimhan, Chacko & Swaminathan, 1982; Narasimhan & Chacko, 1982; Yadava & Padmanabhan, 1981; Marsh, 1980).

Balasubramanian, Lakshminarayanan, Sabesan, Tegoni, Venkatesan & Ramachandran (1971) classified pyrrolidine rings into two conformations without taking into account whether the ring conformation was an envelope or a half boat:

conformation *A*: χ_1 negative and Cγ *exo*,

conformation *B*: χ_1 positive and Cγ *endo*.

Ashida & Kakudo (1974) found a relation between the dihedral angle ψ of proline with the conformation (*A* or *B*) of the proline ring:

ψ of collagen type occurs with ring conformation *B*,

ψ of α-helix type appears with ring conformation *A*.

These correlations are due to steric hindrance between the pyrrolidine and the peptide backbone. But the structure L-Pro-L-Lys acetate does not obey such a correlation, it has a ψ_1 value which corresponds to the collagen type (see Table 3) but the pyrrolidine has the *A* conformation. This may be due to the greater importance of intermolecular forces in small peptides in comparison with intramolecular ones, which is not the case for larger peptides. In fact, the *N*-methyl 1-acetyl-L-prolinamide structure has ψ of α-helix type and Cγ is *endo* (conformation *B*) (Matsuzaki & Iitaka, 1971).

(c) The lysine side chain

The lysine side-chain torsion angles χ_2^1, χ_2^2 and χ_2^3 are in the minimum-energy conformation where the chain is fully extended as is found in this molecule. These results agree with those found in previously determined lysine structures (see Table 3) and theoretical minimum-energy studies (Benedetti, Morelli, Némethy & Scheraga, 1983).

The χ_2^1 angle is *gauche* (–). This conformation is favoured because the C*G*2 atom is at a maximum distance from C2 (actually this angle is also *trans* but is defined *gauche* for nomenclature reasons) and occupies the hole between the N2 and HC*A*1 atoms.

The last side-chain torsion angle (χ_2^4) has a *gauche* (–) conformation, whereas in most lysine structures the *trans* conformation is found (see Table 3). It should be noted that the global conformation of the L-lysine side chain (g^-, *t*, *t*, g^-) had not been found earlier (see Table 3).

(d) Packing

It can be seen in Fig. 2 that H bonds predominate in certain zones of the crystal where there are many H-bond donor and acceptor groups (hydrophilic zones). These zones alternate with regions without H bonds (hydrophobic zones) formed by lysine side chains arranged in an antiparallel way (Fig. 2).

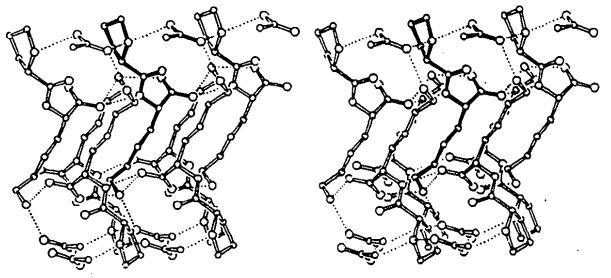

Fig. 2. Stereoview of the crystal structure of L-Pro-L-Lys acetate. The empty bonds represent all the dipeptide molecules which are around the molecule with solid black bonds and form hydrogen bonds with it. The broken lines indicate hydrogen bonds.

Table 4. *Hydrogen-bond lengths* (Å) *and angles* (°)

A—H···*B*	*A*···*B*	H···*B*	*A*—H···*B*
N1—H···O2*AC*^{i}	2·890 (7)	1·967 (7)	141·3 (2)
N1—H′···O1*AC*^{ii}	2·691 (7)	1·612 (7)	176·5 (2)
N2—H···O22^{iii}	2·819 (6)	1·857 (6)	146·1 (2)
NZ2—H···O12^{iv}	2·781 (12)	1·748 (14)	158·46 (6)
NZ2—H′···O22^{v}	2·812 (17)	1·798 (15)	154·60 (7)
NZ2—H″···O1*AC*^{vi}	2·763 (18)	1·709 (16)	163·9 (1)

Symmetry code: (i) *x*, *y*, *z*; (ii) *x*, *y*, *z*−1; (iii) −*x*−1, *y*−0·5, −*z*−2; (iv) −*x*−1, *y*−0·5, −*z*−1; (v) −*x*−1, *y*+0·5, −*z*−1.

All hydrogen bonds are given in Table 4 and in Fig. 2. The H-bonding scheme does not give rise to a head-to-tail arrangement with H bonds between N and C terminal groups as is frequently found in amino acids (Suresh & Vijayan, 1983; Coll, Subirana, Solans, Font-Altaba & Mayer, 1987) and dipeptides (Suresh & Vijayan, 1985). An important fact that disturbs the typical head-to-tail pattern is the presence of counterions and polar groups in the lysine side chain. However, there is a hydrogen bond characteristic of dipeptides, which appears between the N2 (peptide bond) and O22 (terminal carboxylate). This bond is the type S5→9 according to the notation of Suresh & Vijayan (1985).

The dipeptide molecules which are oriented in the same direction show a single H bond between the carboxylate terminal group of one molecule and the peptidic N atom of the other; furthermore they are connected through an acetate anion which acts as a bridge between the two N1 atoms. Other H bonds between dipeptide molecules are found between molecules which are oriented in opposite directions and are separated by +0·5 on the *y* axis. The ε-amino group of lysine forms a bridge between two carboxylate groups which belong to different neighbouring molecules. This kind of packing results in regions with lysine side chains arranged in an antiparallel way and other regions with proline rings alternating with acetate anions.

Each acetate anion is H-bonded to the N1 atoms of two dipeptide molecules equally oriented and to an NZ2 atom of a dipeptide molecule oppositely oriented.

The hydrogen-bonding pattern of the terminal N side chain of lysine probably determines the conformation of the χ_2^1 and χ_2^4 torsion angles. The χ_2^4 angle optimizes the hydrogen bonds with neighbouring molecules, whereas χ_2^1 minimizes the interaction with the peptide backbone. In the available structures (Table 3) it may be seen that χ_2^1 and χ_2^4 are the torsion angles of the lysine side chain which are more variable. This feature correlates fairly well with observations in arginine residues, in which the most variable torsion angle is also χ^1 (Coll, Solans, Font-Altaba & Subirana, 1984; Coll, Subirana, Solans, Font-Altaba & Mayer, 1987). In these cases the planar guanidinium group at the end of the arginine side chain does not allow the three conformations that are possible in the last torsion angle of the lysine side chain.

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References

ARNOUX, B., PRANGÉ, T. & PASCARD, C. (1977). *Cryst. Struct. Commun.* **6**, 29–32.
ASHIDA, T. & KAKUDO, M. (1974). *Bull. Chem. Soc. Jpn*, **47**(5), 1129–1133.
BALASUBRAMANIAN, R., LAKSHMINARAYANAN, A. V., SABESAN, M. N., TEGONI, G., VENKATESAN, K. & RAMACHANDRAN, G. N. (1971). *Int. J. Protein Res.* **3**, 25–33.
BENEDETTI, E., MORELLI, G., NEMETHY, G. & SCHERAGA, H. A. (1983). *Int. J. Pept. Protein Res.* **22**, 1–15.
BHAT, T. N. & VIJAYAN, M. (1976). *Acta Cryst.* B**32**, 891–895.
CAPASSO, S., MATTIA, C. A., MAZZARELLA, L. & ZAGARI, A. (1983). *Acta Cryst.* C**39**, 281–283.
COLL, M., SOLANS, X., FONT-ALTABA, M. & SUBIRANA, J. A. (1984). *Int. J. Pept. Protein Res.* **23**, 242–247.
COLL, M., SUBIRANA, J. A., SOLANS, X., FONT-ALTABA, M. & MAYER, R. (1987). *Int. J. Pept. Protein Res.* **29**, 708–713.
COTRAIT, M., GEOFFRE, S., HOSPITAL, M. & PRECIGOUX, G. (1979). *Acta Cryst.* B**35**, 114–118.
ELGIN, S. C. R. & WEINTRAUB, H. (1975). *Annu. Rev. Biochem.* 725–733, 749–760.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
IUPAC–IUB COMMISSION ON BIOCHEMICAL NOMENCLATURE (1970). *Biochemistry*, **9**, 3471–3479.
KOETZLE, T. F., LEHMANN, M. S., VERBIST, J. J. & HAMILTON, W. C. (1972). *Acta Cryst.* B**28**, 3207.
L'HARIDON, P., LANG, J., PASTUSZAK, R. & DOBROWOLSKI, J. (1978). *Acta Cryst.* B**34**, 2436–2439.
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.
MARSH, R. E. (1980). *Acta Cryst.* B**36**, 1265–1267.
MATSUZAKI, T. & IITAKA, Y. (1971). *Acta Cryst.* B**27**, 507–516.
NARASIMHAN, P. & CHACKO, K. K. (1982). *Cryst. Struct. Commun.* **11**, 2051–2056.
NARASIMHAN, P., CHACKO, K. K. & SWAMINATHAN, S. (1982). *Cryst. Struct. Commun.* **11**, 695–700.
SALUNKE, D. M. & VIJAYAN, M. (1982). *Acta Cryst.* B**38**, 287–289.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
SURESH, C. G. & VIJAYAN, M. (1983). *Int. J. Pept. Protein Res.* **22**, 617–621.
SURESH, C. G. & VIJAYAN, M. (1985). *Int. J. Pept. Protein Res.* **26**, 311–328.
URPÍ, L., COLL, M. & SUBIRANA, J. A. (1988). *Int. J. Biol. Macromol.* In the press.
YADAVA, V. S. & PADMANABHAN, V. M. (1981). *Acta Cryst.* A**37**, C66.

Acta Cryst. (1988). C**44**, 285–287

Conformation and Structure of 9-Propylanthracene

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Abstract. $C_{17}H_{16}$, $M_r = 220{\cdot}23$, orthorhombic, *Pbca*, $a = 7{\cdot}816$ (2), $b = 20{\cdot}455$ (4), $c = 16{\cdot}204$ (4) Å, $V = 2591$ Å^3, $Z = 8$, $D_x = 1{\cdot}13$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}6$ cm^{-1}, $F(000) = 944$, $T = 293$ K, $wR = 0{\cdot}057$ for 1167 unique observed $[F > 3\sigma(F)]$ reflections. The propyl side chain is perpendicular to the ring system and itself adopts an extended conformation. There is a slight folding of the molecule along the C(9)–C(10) axis, the angle between the two outer aromatic ring planes being 3·6 (5)°. There are no unusually close intermolecular contacts.

Introduction. The conformation about a bond between an aromatic sp^2 carbon and an sp^3 carbon (*e.g.* to a saturated carbon chain) depends on the nature of the aromatic ring. In ethylbenzene, the simplest molecule of this type, the existence of eclipsed (coplanar), skew and perpendicular conformers has been deduced from the experimental data. The energy barrier between the different conformers is very low (< 11·7 kJ mol^{-1}; Schrumpf, unpublished results). The situation is probably analogous to that in the homologues with longer straight-chain substituents. 9-Propylanthracene might be considered as an *ortho*-disubstituted benzene displaying steric interactions between the *peri* H atoms and the chain in ecliptic or skew conformations. It appeared interesting to study the conformation of this molecule in the crystal and to investigate whether the

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