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DL-Leucine

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Abstract. $C_6H_{13}NO_2$, triclinic $P\bar{1}$, $a=14{\cdot}12$, $b=5{\cdot}39$, $c=5{\cdot}19$ Å, $\alpha=111{\cdot}1$, $\beta=97{\cdot}0$, $\gamma=86{\cdot}4°$, $Z=2$. $D_{XR}=1{\cdot}285$, D_{exp} (flotation) $=1{\cdot}29$ g cm^{-3}. The structure has been solved on the basis of clear relationships with other amino acids of known structure to an R value of 0·058. The packing of the molecules fits the scheme found for analogous compounds.

Introduction. In a recent paper (Benedetti, Pedone & Sirigu, 1973) significant relationships between the crystal packing of some amino-acid molecules in the racemic and optically active forms have been pointed out. It was shown that the substantial identity of the packing pattern for most α-amino acids in the L form (Torii & Iitaka, 1971) could be extended to the racemic form where layers of isoconfigurational molecules were preserved. To confirm further the validity of the above point, we report here the crystal structure of DL-leucine.

Crystals suitable for the X-ray work were grown from aqueous solution at isoelectric point.

For the intensity data collection, 1071 non-zero independent reflexions were measured by an on-line single-crystal Siemens diffractometer. The unit-cell dimensions were in agreement with the usual packing of α-amino acids and a comparison with that of L-leucine (Torii & Iitaka, 1971) suggests a close relationship between the two structures. The crystal-structure study of L-leucine was announced by the cited authors to be in progress but, to our knowledge, no full report has yet been published. The crystal structure could be derived from that of DL-isoleucine (Benedetti, Pedone & Sirigu, 1973). The coordinates of the COO–C(C)–N group of DL-isoleucine were transferred to the unit cell of DL-leucine and used as starting coordinates. A structure-factor calculation ($R=0{\cdot}38$) and a sequential Fourier synthesis showed the remaining three carbon atoms. The structure was refined by least-squares methods, and a difference Fourier synthesis revealed the positions of all hydrogen atoms not far from the expected stereochemical positions. The light atoms were included in the refinement

Table 1. *Positional and thermal parameters*

(*a*) Final atomic coordinates ($\times 10^4$) and thermal parameters ($\times 10^3$) for the heavy atoms

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O(1)	−1128 (1)	1154 (3)	4147 (3)	5085 (90)	2489 (69)	2860 (73)	143 (61)	370 (62)	1159 (57)
O(2)	−871 (2)	−3291 (4)	2673 (3)	6360 (107)	3041 (76)	2027 (67)	1100 (71)	777 (64)	911 (59)
N	−710 (2)	−3179 (4)	7663 (4)	4043 (94)	2690 (81)	2027 (74)	470 (68)	443 (64)	908 (64)
C(1)	−1081 (2)	−1089 (5)	4448 (4)	3303 (96)	2453 (90)	2156 (86)	−62 (74)	92 (71)	737 (73)
C(2)	−1324 (2)	−1257 (5)	7203 (4)	3941 (108)	2325 (89)	1998 (85)	313 (76)	393 (73)	608 (72)
C(3)	−2368 (2)	−2379 (6)	7416 (5)	4139 (118)	3196 (107)	3220 (107)	−114 (88)	805 (87)	1002 (88)
C(4)	−3091 (2)	−391 (7)	7518 (6)	4126 (128)	5653 (158)	4121 (133)	857 (115)	768 (102)	2071 (121)
C(5)	−4082 (3)	−2013 (12)	6827 (11)	4186 (169)	10144 (323)	8378 (279)	812 (182)	−568 (168)	278 (230)
C(6)	−3126 (3)	1902 (8)	236 (8)	5646 (171)	4752 (167)	6420 (201)	1306 (136)	1185 (142)	704 (146)

(*b*) Final parameters of the hydrogen atoms

	x	y	z	B	Bonded to
H(1)	−1157 (17)	543 (51)	8464 (50)	0·16 (47)	C(2)
H(2)	−749 (21)	−3153 (62)	9487 (61)	1·68 (60)	N
H(3)	−86 (20)	−2696 (57)	7401 (55)	1·81 (53)	N
H(4)	−899 (23)	−4936 (68)	6626 (66)	2·04 (68)	N
H(5)	−2503 (23)	−4201 (66)	5873 (64)	2·31 (64)	C(3)
H(6)	−2459 (22)	−2818 (65)	9109 (66)	1·41 (66)	C(3)
H(7)	−2865 (24)	454 (69)	6123 (69)	3·41 (74)	C(4)
H(8)	−4600 (39)	−671 (113)	6996 (113)	6·95 (1·42)	C(5)
H(9)	−4303 (36)	−2994 (113)	8727 (110)	7·33 (1·38)	C(5)
H(10)	−4109 (45)	−3191 (138)	5273 (138)	5·66 (1·90)	C(5)
H(11)	−3575 (30)	3163 (90)	156 (78)	4·93 (1·06)	C(6)
H(12)	−2543 (35)	2773 (101)	730 (105)	7·84 (1·27)	C(6)
H(13)	−3353 (32)	933 (91)	1755 (93)	4·55 (1·04)	C(6)

Table 2. *Bond distances* (Å) *and angles* (°)

				Dihedral angles*	
O(1)–C(1)	1·241 (3)	O(1)–C(1)–O(2)	125·8 (1)	C(1)–C(2)–C(3)–C(4)	71·3
O(2)–C(1)	1·253 (3)	O(1)–C(1)–C(2)	118·2 (1)	N——C(2)–C(3)–C(4)	169·0
C(1)–C(2)	1·533 (4)	O(2)–C(1)–C(2)	115·9 (1)	O(1)–C(1)–C(2)–N	145·9
C(2)–N	1·491 (3)	C(1)–C(2)–N	108·9 (1)	O(1)–C(1)–C(2)–C(3)	94·8
C(2)–C(3)	1·530 (4)	N——C(2)–C(3)	108·1 (1)	O(2)–C(1)–C(2)–C(3)	83·6
C(3)–C(4)	1·522 (4)	C(2)–C(3)–C(4)	114·9 (1)	C(2)–C(3)–C(4)–C(5)	162·9
C(4)–C(5)	1·536 (6)	C(3)–C(4)–C(6)	111·6 (2)	C(2)–C(3)–C(4)–C(6)	75·8
C(4)–C(6)	1·522 (6)	C(3)–C(4)–C(5)	109·6 (2)		
		C(6)–C(4)–C(5)	110·1 (2)		
		C(1)–C(2)–C(3)	111·5 (1)		

* *trans* = 180°.

and the final R value was 0·058.* The weighting scheme adopted throughout the refinement corresponds to that suggested by Cruickshank & Pilling (1961). In Table 1 a list of the final atomic coordinates and thermal factors is given. A list of molecular parameters is reported in Table 2 (see also Fig. 1). In Fig. 2 the molecular packing DL-leucine orthogonal to the *ac* plane is shown.

Discussion. Isoconfigurational molecules are packed in layers according to a pattern which has been already discussed (Benedetti, Pedone & Sirigu, 1973).

The crystal packing of DL-leucine is made up by double layers of molecules of opposite configuration and is strictly related to the crystal packing of DL-isoleucine, *i.e.* the a axis of DL-leucine corresponds to the $a+c$ axis of DL-isoleucine. From these observations and the examination of the cell parameters of L-leucine and L-isoleucine, one can safely presume that the same structural relationships existing between the packing of DL- and L-isoleucine hold for DL- and L-leucine.

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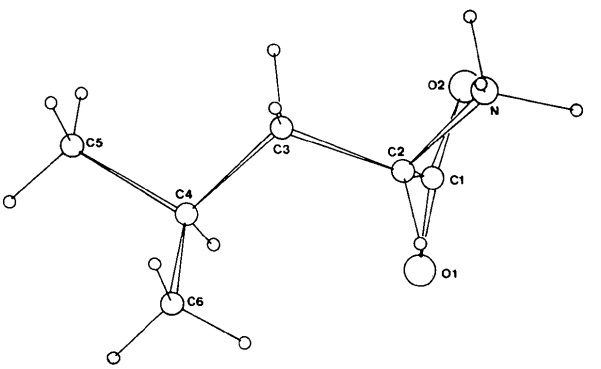

Fig. 1. Molecular model of DL-leucine.

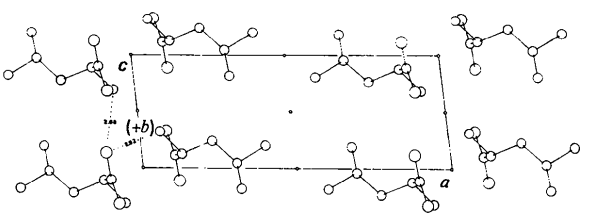

Fig. 2. Packing of DL-leucine orthogonal to the *ac* plane.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30739 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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