

## L-Leucine

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**Abstract.**  $C_6H_{13}NO_2$ ,  $P2_1$ ,  $a=9.61$  (8),  $b=5.31$  (8),  $c=14.72$  (8) Å,  $\beta=86.2^\circ$ ,  $Z=4$ ,  $D_m=1.14$  g cm $^{-3}$ ,  $D_c=1.16$  g cm $^{-3}$ . The structure, determined from 771 X-ray diffraction data, gave  $R=0.13$ . The conformations of the two leucine molecules are similar but not identical to each other and to that in DL-leucine. The carboxyl and amino groups are hydrogen bonded in a double layer very like that in other non-polar L-amino acids.

**Introduction.** We have been examining the structures of a number of amino acids in L- and DL-crystals, to gather information on variations of conformation which cannot be due to differences in chemical structure but must be the result of interactions with neighbouring molecules in the crystal. We have determined the structures of L- and DL-leucine. DL-Leucine has been described by Di Blasio, Pedone & Sirigu (1975); our results are less accurate but agree with theirs.\*

Very soft thin platy crystals of L-leucine were obtained by the isothermal distillation of ethanol into an aqueous solution of L-leucine. Diffraction data were recorded on Weissenberg photographs (Cu  $K\alpha$  radiation) of the layers  $h0l$  to  $h4l$  and  $0kl$  to  $6kl$ , and intensities were estimated visually. 771 independent reflexions were thus measured and 189 recorded as too weak to observe.

The structure was solved by direct methods using

\* There appears to be a mistake in their tabulation of atom parameters; the columns for  $y$  and  $z$  should be interchanged. (We would also give negative signs to the second, fourth and sixth dihedral angles quoted.)

the program *MULTAN* (Main, Woolfson & Germain, 1971). Positional and anisotropic thermal parameters for the carbon, nitrogen and oxygen atoms were refined using the program *CRYLSQ* in the X-RAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972), giving  $R=0.13$  eventually.\* A difference electron density series was calculated and showed peaks near all the stereochemically expected hydrogen atom positions and no other significant features.

**Discussion.** The very soft and sometimes slightly bent crystals gave poor photographs and the atom positions have not been as accurately determined as we would wish. Bond lengths and angles have large standard deviations (0.03–0.05 Å,  $2^\circ$ ) and do not differ significantly from expected values. The conformations of the two molecules in the asymmetric unit are similar to each other and to DL-leucine (Table 2). The differences of 10–15° in dihedral angles are quite significant, and are a typical example of the minor adjustment of torsion angles about single bonds to allow the molecules to pack better in different crystals. Such adjustments 'cost' the isolated molecule very little in energy terms (<2 kJ mole $^{-1}$ ) and if they allow the molecule to make more satisfactory van der Waals contacts or hydrogen bonds they will be worth while.

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31407 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Positional parameters ( $\times 10^3$ ) and thermal parameters ( $\times 10^3$ , in Å $^2$ ) and their estimated standard deviations

	$x$	$y$	$z$	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{31}$	$U_{12}$
O(1)	140 (1)	-24 (3)	415 (1)	51 (8)	57 (11)	21 (6)	2 (7)	-6 (6)	-9 (5)
O(2)	229 (1)	347 (4)	364 (1)	77 (11)	50 (12)	41 (8)	-9 (7)	-5 (7)	-9 (6)
N(1)	389 (1)	-235 (4)	428 (1)	46 (10)	44 (9)	19 (7)	-6 (7)	5 (6)	0 (6)
C(1)	235 (2)	119 (4)	382 (2)	41 (13)	34 (17)	47 (12)	4 (9)	18 (10)	-8 (8)
C(2)	379 (2)	-13 (4)	364 (2)	66 (14)	35 (13)	39 (10)	8 (10)	8 (10)	-3 (8)
C(3)	396 (2)	-113 (5)	269 (2)	26 (11)	49 (13)	59 (12)	15 (9)	9 (9)	-9 (9)
C(4)	407 (2)	96 (5)	197 (2)	114 (23)	75 (19)	51 (13)	-20 (16)	30 (14)	-30 (12)
C(5)	403 (3)	-46 (6)	96 (3)	67 (18)	135 (29)	113 (22)	2 (19)	14 (16)	32 (20)
C(6)	531 (3)	245 (7)	194 (3)	101 (20)	80 (20)	90 (18)	-20 (17)	3 (16)	13 (16)
O(11)	653 (1)	542 (3)	414 (1)	52 (8)	41 (8)	26 (6)	7 (6)	2 (6)	5 (5)
O(12)	735 (1)	929 (5)	382 (1)	88 (11)	32 (12)	52 (9)	-1 (7)	-4 (8)	-6 (6)
N(11)	910 (1)	353 (4)	406 (1)	67 (11)	68 (14)	27 (8)	-13 (9)	8 (8)	-12 (8)
C(11)	743 (2)	700 (5)	388 (2)	34 (12)	44 (16)	36 (11)	0 (9)	-6 (8)	-8 (8)
C(12)	889 (2)	578 (5)	349 (2)	94 (19)	36 (14)	40 (12)	-3 (11)	19 (12)	10 (8)
C(13)	886 (2)	499 (5)	256 (2)	32 (12)	70 (16)	74 (14)	24 (11)	15 (11)	7 (11)
C(14)	891 (3)	716 (7)	188 (3)	91 (20)	101 (22)	76 (16)	13 (18)	30 (15)	-5 (16)
C(15)	853 (3)	626 (7)	86 (3)	48 (17)	241 (52)	139 (26)	-21 (23)	7 (17)	87 (29)
C(16)	1021 (3)	872 (9)	184 (3)	96 (21)	109 (26)	122 (23)	-31 (19)	-26 (18)	32 (20)

Table 2. *Dihedral angles*

	L-Leucine molecule 1 ( $n=0$ )	L-Leucine molecule 2 ( $n=10$ )	L-Leucine in DL-leucine*
$O(n+1)-C(n+1)-C(n+2)-C(n+3)$	92	82	84
$O(n+1)-C(n+1)-C(n+2)-N(n+1)$	-26	-36	-36
$C(n+1)-C(n+2)-C(n+3)-C(n+4)$	68	74	71
$C(n+2)-C(n+3)-C(n+4)-C(n+5)$	-172	-167	-162
$C(n+2)-C(n+3)-C(n+4)-C(n+6)$	69	63	76

\* Values calculated from atom coordinates of Di Blasio *et al.* (1975) after interchanging their O(1) and O(2).

Hydrogen bonds hold the carboxyl and amino groups together in double layers while the non-polar groups lie alongside each other. The whole structure is very similar to L-isoleucine (Torii & Iitaka, 1971). In fact the structures of many amino-acids with non-polar side chains have this same arrangement of a double layer of carboxyl and amino groups; for L-valine (Torii & Iitaka, 1970), L-methionine (Torii & Iitaka, 1973), L-cysteine (Harding & Long, 1968) and L-leucine and L-isoleucine, the atom positions within the double layer are the same, within  $\sim 0.25$  Å; there are two molecules per asymmetric unit, in space group  $P2_1$ , with  $a=9.6 \pm 0.1$  Å and  $b=5.2 \pm 0.1$  Å;  $c$  and  $\beta$  vary according to the size and packing of the side chains, and are tabulated by Torii & Iitaka (1971). Here we have used the unconventional choice,  $\beta < 90^\circ$ , for L-leucine because this makes the atom coordinates directly comparable with those in the other structures. (In L-cysteine the published  $a$  and  $c$  must be interchanged.)

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### Tetraamminepalladium(II) Chloride Monohydrate: The Location of the Water Molecule

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**Abstract.** Tetragonal, apparently  $P4/mbm$ ,  $a=10.337$  (1),  $c=4.2707$  (6) Å,  $\text{Pd}(\text{NH}_3)_4\text{Cl}_2 \cdot \text{H}_2\text{O}$ ,  $Z=2$ ,  $D_c=1.917$  g cm $^{-3}$ . The structure is confirmed as previously described, except that the water molecule appears to be disordered over positions in the vicinity of  $(0, \frac{1}{2}, \pm \frac{1}{4})$ .

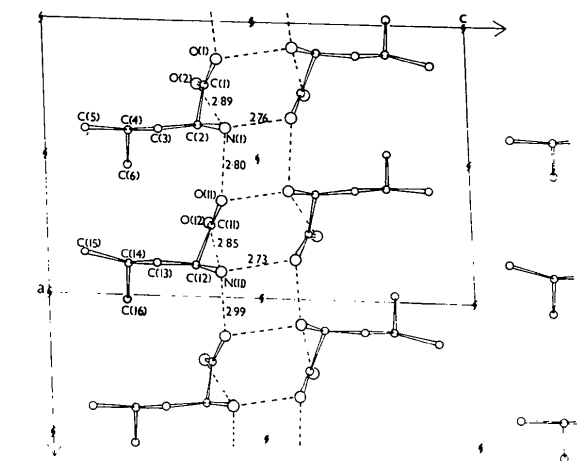


Fig. 1. Projection of the structure looking down the  $b$  axis. Comparison of this with Fig. 3 of Torii & Iitaka (1971) shows the very close similarity to L-isoleucine.

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**Introduction.** Crystals were grown as colourless tetragonal needles, which quickly discolour on exposure and continue to darken until they have obviously decomposed. They could be stabilized by sealing, either in a capillary or by spraying with varnish. During data collection monitor reflexions showed no change in