

LETTERS TO THE EDITOR

CRYSTAL STRUCTURES OF GLYCYL-L-THREONINE DIHYDRATE AND DL-LEUCYL-LEUCINE

A PROGRAM of the study of the crystal structures and conformation of dipeptides has been undertaken in our laboratory. The crystal structure analyses of dipeptides glycyl-L-threonine dihydrate and DL-leucyl-DL-leucine form a part of it.

Glycyl-L-threonine dihydrate.—Colourless, rectangular prismatic crystals of glycyl-L-threonine dihydrate were grown by slow evaporation of an aqueous solution of the substance. The unit cell dimensions and space group were determined from oscillation and Weissenberg photographs taken about crystallographic axes using CuK α radiation. The crystal and physical data of the compound are as follows:

Crystal system	orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁
Lattice parameters	$a = 9.72 \text{ \AA}$ $b = 10.03$ $c = 10.73$
Number of molecules in the unit cell	$Z = 4$
Measured density	$d_m = 1.39 \text{ gm/cc.}$
Calculated density	$d_{cal} = 1.38 \text{ gm/cc.}$

The crystal structure has been determined by symbolic addition method. The positional parameters of all non-hydrogen atoms are given in Table I and the bond lengths and bond angles in the molecule in Table II. A detailed discussion of the stereochemistry will be presented elsewhere.

TABLE I
Fractional atomic coordinates

Atom	Conventional notation*	x/a	y/b	z/c
N ₁	N ₁	0.7083	0.0466	0.6251
C ₁	C ₁ ^{α}	0.6138	0.1182	0.5390
C ₂	C ₁	0.6783	0.2515	0.4987
O ₁	O ₁	0.8021	0.2765	0.5208
N ₂	N ₂	0.5924	0.1323	0.401
C ₃	C ₂ ^{α}	0.543	0.5757	0.4363
C ₄	C ₃ ^{β}	0.6179	0.4539	0.2401
C ₅	C ₂ ^{γ}	0.7144	0.3528	0.1786
O ₂	C ₂ ^{γ} ¹	0.4734	0.1252	0.2130
C ₆	C ₂	0.6304	0.4595	0.3869
O ₃	O ₂ ¹	0.5852	0.6585	0.4110
O ₄	O ₂ ¹¹	0.4433	0.5482	0.5071
OW1		0.351	0.8167	0.7661
OW2		0.641	0.2281	0.8275

* IUPAC—IU, Commission on Biochemical Nomenclature, *Biochem. Biophys. Acta.*, 1971, **229**, 1-17.

TABLE II
Bond lengths and bond angles

N ₁ —C ₁	1.49 Å	N ₁ —C ₁ —C ₂	110.0°
C ₁ —C ₂	1.54	C ₁ —C ₂ —O ₁	120.8
C ₂ —O ₁	1.25	C ₁ —C ₂ —N ₂	114.1
N ₂ —C ₃	1.44	O ₁ —C ₂ —N ₂	125.0
C ₃ —C ₄	1.51	N ₂ —C ₃ —C ₆	114.1
C ₄ —O ₃	1.22	N ₂ —C ₃ —C ₄	110.0
C ₅ —O ₄	1.50	C ₃ —C ₄ —C ₅	113.9
C ₃ —C ₄	1.57	C ₃ —C ₄ —O ₂	106.2
C ₄ —C ₅	1.53	C ₅ —C ₄ —O ₂	111.8
C ₄ —O ₂	1.46	C ₃ —C ₆ —O ₅	118.9
		C ₃ —C ₆ —O ₄	117.1
		O ₃ —C ₆ —O ₄	123.9

DL-Leucyl-DL-leucine.—Fragile, platy crystals of DL-leucyl-DL-leucine were grown by slow evaporation at room temperature either from alcohol or water solution. The unit cell dimensions and space group were determined from oscillation and Weissenberg photographs taken about crystallographic axes using CuK α radiation. The crystal and physical data of the compound are as follows:

Crystal system	Monoclinic
Space group	P2 ₁ /a
Lattice parameters	$a = 11.68 \text{ \AA}$ $b = 25.52$ $c = 10.52$ $\beta = 101.9^\circ$

Number of molecules in the unit cell	$Z = 8$
Measured density	$= 1.052 \text{ gm/cc.}$
Calculated density	$= 1.058 \text{ gm/cc.}$

Three-dimensional intensity data have been collected and the structure analysis is in progress.

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INFRARED SPECTRA OF SOME BROMOXYLENES IN THE LIQUID STATE

IN continuation of our work on vibrational spectra of some substituted xylenes¹ the infrared spectra of 1-bromo-2, 3-, 1-bromo-2, 4- and 1-bromo-2, 6-xylenes are reported here. Using samples supplied by Koch-Light and Co., the infrared spectra of the above compounds were recorded in the pure liquid state in the region 650–4000 cm⁻¹ on a Perkin-Elmer, Model 221 double beam automatic spectrophotometer

TABLE I
Vibrational frequencies of 1-bromo-2,3-, 1-bromo-2,4- and 1-bromo-2,6-xylenes

1-bromo-2, 3-xylene	1-bromo-2, 4-xylene	1-bromo-2, 6-xylene	Assignment
3055 (m)	3050 (sh)	3045 (m)	C—H stretching
..	3025 (sh)	3035 (sh)	do.
3005 (m)	3005 (m)	3005 (w)	do.
2965 (m)	2970 (m)	2970 (s)	C—H asymmetric stretching (in CH ₃ group)
2940 (s)	2940 (m)	2945 (vs)	do.
2910 (s)	2915 (s)	2915 (s)	do.
2855 (m)	2855 (s)	2850 (m)	C—H symmetric stretching (in CH ₃ group)
1590 (s)	1590 (m)	1585 (ms)	C—C stretching
1550 (sh)	1580 (sh)	1570 (ms)	do.
1485 (sh)	1475 (sh)	1485 (sh)	do.
1465 (sh)	1465 (s)	1465 (ms)	do.
1460 (ms)	1445 (w)	1455 (ms)	C—H asymmetric bending (in CH ₃ group)
1435 (s)	1430 (s)	1425 (s)	do.
1395 (s)	1390 (vs)	1390 (s)	C—H symmetric bending (in CH ₃ group)
1290 (m)	1285 (m)	1275 (m)	C—C stretching
1280 (ms)	1240 (s)	1270 (m)	C—CH ₃ stretching
1180 (s)	1170 (vs)	1200 (s)	do.
1145 (ms)	1145 (s)	1170 (ms)	C—H in-plane bending
1090 (ms)	1090 (m)	1110 (w)	CH ₃ rocking
1020 (sh)	1030 (sh)	1030 (vs)	do.
1010 (s)	1025 (s)	1015 (s)	C—H in-plane bending
1000 (vs)	1005 (s)	1000 (s)	C—C in-plane bending
910 (w)	920 (ms)	930 (w)	C—H out-of-plane bending
890 (m)	875 (s)	910 (ms)	do.
830 (s)	830 (m)	880 (w)	do.
765 (vs)	765 (vs)	760 (vs)	C—C stretching
785 (s)	800 (vs)	805 (m)	C—CH ₃ out-of-plane bending
695 (vs)	720 (s)	695 (s)	C—C out-of-plane bending
655 (w)	670 (m)	660 (w)	C—C in-plane bending

vs=very strong; s=strong; ms=medium strong; m=medium; w=weak; sh=shoulder;
sb=strong and broad.

with a NaCl prism-grating interchange using cells of thickness 0.050 mm and 0.025 mm.

Of the three bromoxylenes studied, 1-bromo-2,3- and 1-bromo-2,4-xylenes belong to the point group C_{2v} and 1-bromo-2,6-xylene belongs to C_{2v} symmetry.

The analyses of the bands and assignments of the fundamental frequencies have been made essentially on the basis of intensities of the observed bands and also with the help of the infrared spectra of xylenes²⁻⁴ and chloroxylenes¹.

A correlation of all the observed fundamental frequencies is given in Table I.

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Dharwar-3, July 7, 1972.

X-RAY DETERMINATION OF THE THERMAL EXPANSION OF LEAD MOLYBDATE

LEAD molybdate, a tetragonal crystal belonging to space group $14_1/a$, is an isotype of scheelite (CaWO_4). Argyle and Hummel¹ have measured the linear expansion of a polycrystalline sample using a dilatometer. Brower² has communicated to the present authors his results on the directional thermal expansion of lead molybdate, as determined by an interferometric method. With the exception of some reports on the determinations of the room temperature values of the lattice parameters, there is no published work available on their temperature variation.

Hence, we have undertaken the precise determination of the lattice parameters and the evaluation of the principal coefficients of linear thermal expansion of PbMoO_4 , using an X-ray method, as a part of our general programme of 'X-ray studies on scheelite type compounds'. Similar studies on KIO_4 ³, NaIO_4 ⁴, CaMoO_4 ⁵, CdMoO_4 ⁶, SrWO_4 ⁷, BaWO_4 ⁸, PbWO_4 ⁹, SrMoO_4 ¹⁰ and CaWO_4 ¹¹ have already been published.

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