Characteristic extinctions were noted as follows:

$$hkl \text{ present only when } h+k+l=2n,$$
$$0kl \text{ present only when } l=2n,$$
$$hhl \text{ present only when } 2h+l=4n.$$

These extinctions are those uniquely required by the space group C_{4v}^{12}–$I4cd$, which has only eightfold and sixteenfold positions. If this space group is accepted, the number of molecules in the unit cell must be a multiple of eight.

The density of beryllium borohydride was not known, and none of the customary techniques to measure it was convenient because of the small amount of sample and the high reactivity of the substance. Assuming sixteen molecules per unit cell the calculated density is 0·604 g.cm.$^{-3}$. This can be compared with the density of a comparable substance, liquid propane, C_3H_8, just under its normal boiling point. At −44·5° C. liquid propane has a density of 0·585 g.cm.$^{-3}$ (*Handbook of Chemistry and Physics*, 1943). From this comparison it is clear that the unit cell contains sixteen molecules rather than some other multiple of eight molecules.

The probable space group can account for sixteen molecules in two sets of eight in positions 8 (a), or in one set of sixteen in positions 16 (b). Spatial considerations as well as the appearance of strong reflections such as (310) require the molecules to be in general sixteenfold positions. Since each atom of the molecule is in a general position no conclusions concerning molecular symmetry can be reached. In particular, the two boron atoms need not be crystallographically equivalent.

Indirect evidence, namely the rapid decline of intensity with increasing $\sin\theta/\lambda$, indicates a structure with discrete molecules held by secondary valence forces rather than an ionic structure with strong electrostatic forces. At room temperature no reflections were observed with $\sin\theta$ greater than 0·61 (using copper $K\alpha$). Under similar conditions lithium hydride produces strong reflections out to $\sin\theta \approx 1$. The rapid decline of intensity for beryllium borohydride cannot be ascribed solely to the small and rapidly declining atomic scattering factors, but is evidence for large thermal motions characteristic of molecular crystals.

In spite of the observation that the probable space group lacked a center of symmetry, thereby limiting the use of Fourier-series methods, and that the molecules were in general positions, the intrinsic interest in the structure of the molecules made it worth trying to get a complete structure. In the hope of finding the structure a number of Patterson sections, lines, and projections were calculated. These included the projection $P(x, y)$; the sections $P(x, y, 0)$ and $P(x, 0, z)$; and the lines $P(0, 0, z)$, $P(\frac{1}{4}, \frac{1}{4}, z)$, and $P(x, x, 0)$. By symmetry the section $P(x, y, \frac{1}{2})$ was obtained from the section $P(x, y, 0)$. These calculations did not suggest a structure.

Reference

Handbook of Chemistry and Physics (1943). 27th ed. Cleveland: Chemical Rubber Publishing Company.

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The unit-cell dimensions and the space groups of some alanyl peptides.*

By R. A. Pasternak and John E. Leonard, *Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena* 4, *California, U.S.A.*

(*Received* 19 *June* 1951)

The data presented in this paper were obtained from samples of D,D- and L,L-dialanine and D,D,D- and L,L,L-trialanine kindly provided by Prof. E. Brand of Columbia University. They comprise a portion of a survey which is being made for the purpose of selecting crystalline peptides suitable for complete X-ray analysis.

Rotation and zero-layer (occasionally first-layer) Weissenberg photographs were taken around at least two crystallographic axes of different crystal specimens of each substance. Dimensions of the unit cells (accurate to about ±0·5%) were obtained from measurements of the Weissenberg photographs. The space groups were determined from the systematic absences found on the Weissenberg, supplemented by observations made on Laue photographs.

The densities of the crystals were measured by the usual flotation procedure; the results are estimated to be accurate to better than 1%.

D,D-*Dialanine* crystallized from water–ethanol mixtures in the form of tetragonal needles on which {110} predominated. The Laue photographs showed the symmetry $4/m$, and the only systematic absences found on the Weissenberg photographs were those for which $h+k+l$ was odd. These observations are compatible only with the space groups $I4$, $I\bar{4}$ or $I4/m$. Since the crystal contains only one kind of asymmetric molecule, the presence of a plane of symmetry is excluded and the space group is doubtless $I4$.

L,L,L-*Trialanine* crystallized from water in the form of thick monoclinic plates, on which {001} predominated. The only systematic absences observed were in the first ten orders of $(0k0)$, which were found to be present only when k was even, compatible with the space groups $P2_1$ or $P2_1/m$. Because the crystal is composed of only one optical isomer, the space group is doubtless $P2_1$.

Of these two crystals, only D,D-dialanine seems to be feasible for X-ray analysis; the short four fold axis indicates little molecular overlapping in this direction, and the number of molecules in the unit cell equals the number of general positions. Data for both crystals are presented in Table 1.

Since the presence of planes or centers of symmetry often facilitates the determination of a crystal structure, several attempts were made to obtain the racemic crystals of both D,D-L,L-dialanine and D,D,D-L,L,L-tri-

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* Contribution No. 1580 from the Gates and Crellin Laboratories. This investigation was supported by a research grant from the National Institutes of Health, Public Health Service.

alanine by preparing mixtures containing equal quantities of the pure optical isomers and crystallizing these mixtures from various solvents. With dialanine two types of triclinic crystals were obtained from water–methylcellosolve and water–dioxane solutions respectively. The crystals from the former mixture were anhydrous, while those from the latter contained one-half molecule of water for each molecule of peptide. The Weissenberg photographs of both forms revealed no symmetry elements or systematic absences, consequently the space groups of both crystals were either $P\bar{1}$ or $P1$. Unfortunately, we were unable to obtain data which would serve to distinguish between these two space groups, and since resolution could easily have taken place during crystallization, it was uncertain whether these were crystals of racemates or of pure optical isomers. Data for these crystals are listed in Table 1. Mixtures of the trialanines yielded only very poor crystals which were unsatisfactory for X-ray examination.

We wish to thank Dr Robert B. Corey, who suggested this investigation, for helpful advice and discussion.

Table 1. *Crystallographic data*

Peptide	Crystal system	Space group	General positions in unit cell	Molecules in unit cell	Unit-cell dimensions			Density (g.cm.$^{-3}$)
					a (Å)	b (Å)	c (Å)	
D,D-Dialanine	Tetragonal	$I4$	8	8·05	18·00	18·00	5·16	1·280
L,L,L-Trialanine	Monoclinic	$P2_1$	2	4·01	9·86	10·04	11·88	1·333
						$\beta=101°$		
Dialanine (D,D- or racemate)	Triclinic	$P1$	1	8·00	14·10	12·02	10·51	1·261
		or $P\bar{1}$	2		$\alpha=105°$	$\beta=101°$	$\gamma=90°$	
Dialanine $\frac{1}{2}H_2O$ (D,D- or racemate)	Triclinic	$P1$	1	2·00	5·15	13·34	13·47	1·277
		or $P\bar{1}$	2		$\alpha=102°$	$\beta=96°$	$\gamma=101°$	

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Hexagonal $CaAl_2Si_2O_8$.

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(*Received* 11 *October* 1951)

$CaAl_2Si_2O_8$, which in nature is triclinic (anorthite), has been synthesized recently in two polymorphic forms (Davis & Tuttle, 1951). Dr N. L. Bowen kindly supplied a sample of one of the forms for single-crystal work. Equi-inclination Weissenberg photographs show the crystals to be hexagonal with diffraction symbol $6/mmm\ C\text{–}/\text{–}\text{–}\text{–}$. The cell dimensions are

$$a = 5{\cdot}10 \pm 0{\cdot}01, \quad c = 2 \times 7{\cdot}36 \pm 0{\cdot}01 \text{ Å}.$$

Reflections with l odd are few and very weak, many of them barely discernible, indicating marked pseudo-halving of c. With two formula units per cell,

$$d_{\text{calc.}} = 2{\cdot}78 \text{ g.cm.}^{-3}$$

as compared with

$$d_{\text{meas.}} = 2{\cdot}7 \pm 0{\cdot}1 \text{ g.cm.}^{-3}.$$

W. L. Bond (Bell Telephone Laboratories) kindly performed pyroelectric and piezoelectric tests, and could not rule out a center of symmetry. The most probable space group is therefore $C6/mmm$.

Ito (1950) has proposed a structure for α-celsian, the high-temperature modification of $BaAl_2Si_2O_8$. Because this barium compound and the new calcium compound have similar physical properties and cell dimensions, and because they belong to the same space group, they were suspected of being isostructural. Structure-factor calculations, however, have disproved this hypothesis.

Perfect cleavage on (00.1) indicates a layer structure. The a-axis Weissenberg photographs of the odd (1st and 3rd) levels (taken by Mr K. Güler) show streaks along the festoons representing reciprocal-lattice rows parallel to [00.1]*. Mistakes in the stacking of the layers may therefore be expected. The structure determination will be attempted.

References

Davis, G. L. & Tuttle, O. F. (1951). *Two New Crystalline Phases of the Anorthite Composition*, $CaO.Al_2O_3.2SiO_2$. In preparation.

Ito, T. (1950). *X-Ray Studies on Polymorphism.* Tokyo: Maruzen.

Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. Copy should be sent direct to the British Co-editor (R. C. Evans, Crystallographic Laboratory, Cavendish Laboratory, Cambridge, England).

Acta Crystallographica: important notice

The Executive Committee has decided to entrust the publication of this journal to Messrs Ejnar Munksgaard of Copenhagen as from 1 January 1952. The subscription price per volume is now 100 Danish crowns post free, or its equivalent in other currencies. (At the present rates of exchange American and British subscriptions will be accepted at \$15 and £5 respectively.) Orders should be placed direct with the publisher (Ejnar Munksgaard, Nörregade 6, Copenhagen, Denmark) or with any bookseller. Advance orders for complete volumes from subscribers in the United States of America, its territories and