six observable orders) with k odd. These absences are required only by the space group $P2_1/a$.

Glycyl-D,L-*alanine* proved to be very similar to D,L-alanyl glycine. Crystals were obtained from the same solvent, and they showed similar habit and cleavage. The same absences were found on the Weissenberg photographs, giving again the space group $P2_1/a$. There was, however, no simple relationship between the unit-cell dimensions of the two crystals. Data for both are presented in Table 1.

L-*Leucylglycine* crystallized from water–acetone mixtures in the form of triclinic plates containing water of crystallization. No elements of symmetry or systematic absences were observed on the Weissenberg photographs. From the possible space groups $P1$ and $P\bar{1}$, the latter can be excluded because the crystal is composed of only one optical isomer, and, moreover, the unit cell contains only one molecule of leucylglycine. The space group is therefore undoubtedly $P1$.

Attempts were made to obtain racemic crystals of D,L-*leucylglycine* by crystallizing a synthetic mixture of the optical isomers. Monoclinic crystal plates which cleaved easily along {100} were obtained from water–methylcellosolve mixtures. The Laue photographs showed the symmetry $2/m$ and the Weissenberg photographs had systematic absences only among the six observable orders of $(0k0)$, which were found to be present only when k was even. These absences are required only by the space groups $P2_1$ and $P2_1/m$. However, the number of molecules in the unit cell, as calculated from the unit-cell dimensions and density, is only two, and the space group is therefore undoubtedly $P2_1$. Since this space group excludes a racemic crystal, resolution must have taken place during crystallization. We were unable to obtain the same crystal form directly by crystallization of the L-isomer of leucylglycine.

D,L-*Leucylglycylglycine* crystallized from water–ethanol mixtures in monoclinic prisms. The only systematic absences found were $(h0l)$ with h odd and $(0k0)$ with k odd. The space group is therefore unambiguously $P2_1/a$. Data for these leucyl peptides are presented in Table 1.

Several attempts to obtain crystals of glycyl-L-leucine, suitable for X-ray examination, were unsuccessful.

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

Reference

PASTERNAK, R. A. & LEONARD, J. E. (1952). *Acta Cryst.* **5**, 152.

Table 1. *Crystallographic data*

Peptide	Crystal system	Space group	General positions in unit cell	Molecules in unit cell	Unit-cell dimensions a (Å)	b (Å)	c (Å)	Density (g.cm.$^{-3}$)
D,L-Alanylglycine	Monoclinic	$P2_1/a$	4	4·01	15·72	5·41 $\beta=92°$	8·00	1·429
Glycyl-D,L-alanine	Monoclinic	$P2_1/a$	4	3·95	9·20	7·75 $\beta=92°$	9·44	1·425
L-Leucylglycine $2H_2O$	Triclinic	$P1$	1	1·01	5·98 $\alpha=90°$	7·95 $\beta=108°$	6·89 $\gamma=96°$	1·214
D- or L-Leucylglycine	Monoclinic	$P2_1$	2	1·98	11·45	5·42 $\beta=103°$	7·89	1·294
D,L-Leucylglycylglycine	Monoclinic	$P2_1/a$	4	4·00	9·60	12·24 $\beta=101°$	11·41	1·236

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Space group and unit cell of beryllium borohydride.*

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The material used in this investigation was a small sample of freshly fractionated beryllium borohydride, $Be(BH_4)_2$, furnished by Prof. A. B. Burg of this laboratory. Because of the explosive reactivity of this material with water or oxygen, all transfers of the material had to be accomplished using a vacuum system, and all X-ray diffraction specimens had to be in sealed containers. The volatility near room temperature is sufficiently high to permit relatively rapid sublimation, and by this method a number of samples, suitable for powder photographs, were prepared in thin Pyrex capillaries. The material in one of these capillaries was caused to grow into a single needle-shaped crystal large enough for single-crystal photographs, free from overgrowths, and with the needle axis nearly parallel to the capillary axis. Optical examination with a polarizing microscope showed parallel extinction for the single crystal, discounting effects caused by the enclosing Pyrex capillary. The shape of the single crystal was that of an elongated, rectangular, nearly square prism with poorly developed pyramid faces at the ends.

Powder photographs were taken both with filtered and with monochromatic copper X-rays. The single-crystal specimen was used for rotation and for equi-inclination Weissenberg photographs of the levels $hk0$, $hk1$, $hk2$ and $hk3$. These photographs were in accord with a tetragonal unit cell having

$$a_1 = 13{\cdot}59 \pm 0{\cdot}05, \quad a_3 = 9{\cdot}22 \pm 0{\cdot}05 \text{ Å},$$

based on λCu $K\alpha = 1{\cdot}540$ Å.

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Characteristic extinctions were noted as follows:

hkl present only when $h+k+l=2n$,
$0kl$ present only when $l=2n$,
hhl 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.*

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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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