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Dispositif supprimant l'asymétrie des diagrammes due à la hauteur du faisceau issu d'un monochromateur.

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Un cristal courbé, utilisé comme monochromateur, focalise les rayons X non en un point, mais sur une petite droite. Il s'ensuit qu'on ne peut bénéficier de la focalisation pour obtenir des raies à la fois intenses et fines que dans le plan perpendiculaire à la hauteur du faisceau: en dehors de l'équateur des diagrammes, les raies s'élargissent jusqu'à atteindre la hauteur du faisceau (Fig. 2 (*a*)). C'est là un grave inconvénient dans tous les cas où le diagramme de l'échantillon n'a pas une symétrie de révolution. Ainsi les arcs renforcés par suite d'orientations privilégiées ont un noircissement dépendant de la largeur de la raie, donc de leur azimuth (Fig. 2 (*a*)). Le diagramme Fig. 2 (*b*) est donné par un cristal unique d'alliage contenant de nombreux précipités orientés: il est difficile de trouver les indices des taches du précipité quand elles sont éloignées de l'équateur, car deux anneaux consécutifs ne sont plus suffisamment séparés.

Le dispositif représenté sur la Fig. 1 élimine ces inconvénients. Le film photographique et l'échantillon, solidaires l'un de l'autre, tournent autour du rayon moyen du faisceau primaire. Un écran fixe masque la plaque, sauf sur un secteur de 30° d'ouverture et à bissectrice horizontale. Ainsi on n'enregistre le diagramme que dans les conditions où les raies sont fines. Les Fig. 3 (*a*) et (*b*) montrent le résultat obtenu par comparaison avec les diagrammes ordinaires Fig. 2 (*a*) et (*b*). En enregistrant la trace du faisceau direct dans deux positions rectangulaires, on définit le centre du diagramme à 0,1 mm. près et toutes les taches de la Fig. 3 (*b*) peuvent être pointées avec grande précision. Dans le diagramme Fig. 3 (*a*), le noircissement de l'anneau en chaque point représente correctement l'intensité sur la sphère de pôles.

Le temps de pose doit être multiplié théoriquement par 6, mais pratiquement la perte est beaucoup moindre dans le cas où les raies larges sont remplacées par des raies fines. D'ailleurs c'est parce que nous disposons d'un tube à anticathode tournante que nous avons envisagé d'adopter ce montage.

Il serait possible de faire des diagrammes de cristal tournant en ajoutant un mouvement de rotation de l'échantillon autour de YY' non synchronisé avec la rotation autour de XX' (Fig. 1).

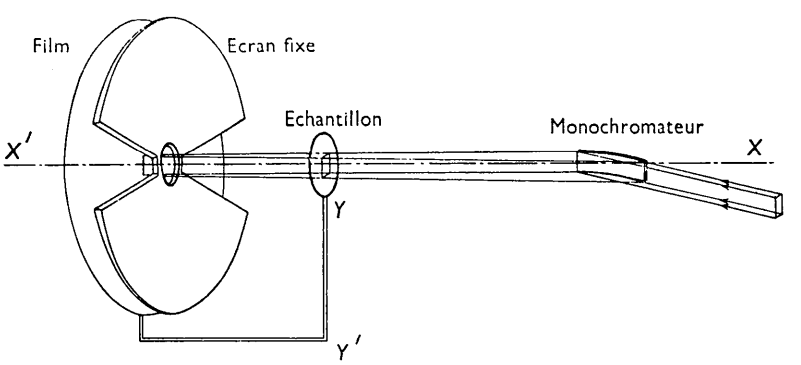

Fig. 1. Schéma de la chambre tournante.

Acta Cryst. (1952). 5, 150

The unit-cell dimensions and the space groups of some simple peptides of glycine, alanine and leucine.*

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The data presented in this paper were obtained from samples of D,L-alanylglycine, glycyl-D,L-alanine, glycyl-D,L-leucine, D,L-leucylglycine, L-leucylglycine and D,L-leucylglycylglycine;† they represent a continuation of a survey (Pasternak & Leonard, 1952) which is being made in these laboratories for the purpose of selecting crystalline peptides suitable for complete X-ray analysis, and were obtained by use of the experimental techniques described in the preceding publication.

D,L-*Alanylglycine* crystallized from water–methylcellosolve mixtures in the form of tabular monoclinic crystals. They cleaved very easily along {001}, and this probably accounts for the relatively poor photographs obtained. On the Weissenberg photographs the only systematic absences found were ($h0l$) with h odd, and ($0k0$) (for the

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† The first three of these peptides were purchased from Amino Acid Manufacturers, University of California at Los Angeles, and the remainder from Delta Chemical Works, New York, N.Y.

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

Acta Cryst. (1952). **5**, 151

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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