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Elementarzellen und Raumgruppen der Peptidderivate Carbobenzoxy-L-leucyl-L-tyrosyl-L-leucinmethylester und Carbobenzoxyglycyl-L-alaninäthylester.*

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Gillessen (1961) synthetisierte das Peptid L-Leucyl-L-tyrosyl-L-leucin, dessen Derivat Carbobenzoxy-L-leucyl-L-tyrosyl-L-leucinmethylester aus Essigester in verhältnismässig gut ausgebildeten gedrungenen hexagonalen Pyramiden kristallisierte. Ausgebildet waren ausschliesslich (001)- und (10$\bar{1}$0)-Flächen. Eine Drehkristallaufnahme um die *c*-Achse mit vanadiumgefilterter Chromstrahlung und Präzessionsaufnahmen nach Buerger mit den Einstrahlungsrichtungen parallel zur *a*-Achse sowie parallel zur Winkelhalbierenden zweier Nebenachsen mit nickelgefilterter Kupferstrahlung ergaben eine hexagonale Elementarzelle mit den Translationsperioden

$$a_0 = b_0 = 7{,}22 \pm 0{,}02;\ c_0 = 36{,}31 \pm 0{,}1\ \text{Å}$$

und den Winkeln $\alpha = \beta = 90°$; $\gamma = 120°$.

Daraus wird unter Annahme von 6 Molekülen in der Elementarzelle die Dichte zu 1,12 g.cm.$^{-3}$ berechnet. Gefunden wurde nach der Verdrängungsmethode 1,11 g.cm.$^{-3}$. Die Laue-Symmetriegruppe 6/*mm* und die ausschliesslich beobachteten 000*l*-Reflexe mit $l = 3n$ lassen die Wahl zwischen den Raumgruppen $P6_222$ und $P6_422$. Im reziproken Gitter treten parallel zur *c*-Achse zwischen den Schichtlinien schwache und diffuse Reflexserien auf, deren Bedeutung und Herkunft unklar ist.

Im Zusammenhang mit Arbeiten zur Synthese von Peptiden mit Sequenzen des Seidenfibroins fiel das Peptidderivat Carbobenzoxyglycyl-L-alaninäthylester in Form nadeliger Einkristalle an. Eine Drehkristallaufnahme sowie Weissenbergaufnahmen um die Nadelachse (*c*-Achse) der rhombischen Prismen und Präzessionsaufnahmen nach Buerger mit der Einstrahlungsrichtung parallel zu den Flächendiagonalen der Prismenendfläche (*a*- und *b*-Achse) ergaben eine orthorhombische Elementarzelle mit folgenden Dimensionen:

$$a_0 = 21{,}17 \pm 0{,}05;\ b_0 = 9{,}78 \pm 0{,}03;\ c_0 = 16{,}10 \pm 0{,}04\ \text{Å}$$

und den Winkeln $\alpha = \beta = \gamma = 90°$.

Es waren praktisch ausschliesslich (001)- und (110)-Flächen ausgebildet. Allgemeine systematische Auslöschungen traten nicht auf. Die fehlenden Reflexe *h*00 mit $h = 2n+1$, 0*k*0 mit $k = 2n+1$ und 00*l* mit $l = 2n+1$ lassen auf die Raumgruppe $P2_12_12_1$ schliessen.

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Literatur

Gillesen, Dieter (1961). Diplom-Arbeit Abteilung Chemie der TH Aachen.

* 25. Mitt. über Peptide; 24. Mitt., vgl. H. Zahn & M. Heinz, *Liebigs Annalen*, im Druck.

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The atomic position parameter in alpha uranium-room temperature and above.

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Alpha uranium, which is orthorhombic, space group D_{2h}^{17}–*Cmcm*, with 4 U per unit cell at $0, y, \frac{1}{4}$; $0, \bar{y}, \frac{3}{4}$; $\frac{1}{2}, \frac{1}{2}+y, \frac{1}{4}$; $\frac{1}{2}, \frac{1}{2}-y, \frac{3}{4}$; can be described in terms of corrugated sheets parallel to the *b* face in which the *y* parameter is a measure of the degree of corrugation—the larger the value of *y* the greater the degree of corrugation. It becomes of interest to determine the change in *y* with temperature since an increase in *y* makes the orthorhombic uranium lattice more nearly hexagonal.

Previously reported and presently determined values of *y* are shown in Table 1. Most of the results are in fairly good agreement near room temperature, however, the value of *y* at elevated temperatures has not been very thoroughly investigated. Therefore this investigation was undertaken to determine especially values at the elevated temperatures.

For the space group concerned the structure factor, *F*, can be stated as $F = 4(f_U \text{ or } b_U) \cos 2\pi hx \cos 2\pi ky \cos 2\pi lz$, where f_U is the X-ray and b_U the neutron scattering factors for uranium. Since there is only one positional parameter, *y*, $F = 4(f_U \text{ or } b_U) \cos 2\pi ky$, therefore the (0*k*0) reflections are particularly suitable for its determination. This technique was used recently by Sturcken & Post (1960) in their determination of the *y* positional parameter at 25 °C. Since their technique involved the determination of a minimum value for the agreement factor, *R*, for selecting the best value of *y* using a predetermined value of the temperature factor, *B*, it was decided at first to recheck the room temperature values of *y* and *B* using the Busing–Levy (1959) least-squares program.

X-ray data were first obtained at room temperature in a manner similar to that used by Sturcken & Post (1960) using a small single crystal approximately 3 mm. on an edge with a polished (0*k*0) face. These results together with a redetermination of *y* from the Sturcken & Post data using the Busing–Levy program are shown in Table 1.

The uranium crystal was then mounted in a high

Table 1. *y Positional parameter for alpha uranium as reported by various investigators*

Investigator	Material	Temperature	*y*
Jacob & Warren (1937)	Powder	R.T.	0·105 ±0·005
Konobeevsky *et al.* (1958)	Powder	20 °C.	0·107 ±0·003
Chebotarev (1961)	Powder	R.T.	0·105
Sturcken & Post (1960)	Single crystal	25 °C.	0·1025±0·0003*
Cash *et al.* (1961)	Powder	R.T.	0·102 ±0·002
Present invest. (X-ray)	Single crystal	25 °C.	0·1025±0·0005
Present invest. (neutron)	Single crystal	25 °C.	0·1024±0·0003
Konobeevsky *et al.* (1958)	Powder	500 °C.	0·115 ±0·003
Chebotarev (1961)	Powder	640 °C.	0·112
Present invest. (neutron)	Single crystal	625 °C.	0·1057±0·0006

* A value of 0·1024±0·0005 was obtained as redetermined with the same data using the least-squares program.

temperature furnace designed primarily for polycrystalline X-ray studies. The crystal was held in an open section in the center of a large silver plate by very light tantalum springs. Silver served as a good holder since it is a good thermal conductor and does not react with uranium. The plate and crystal with suitable thermocouples were held in the center of a resistance furnace. An ion getter pump attached to the top of the furnace eliminated all vacuum lines during runs and produced a vacuum of 10^{-6} to 10^{-7} mm. Hg.

Except for a few preliminary X-ray measurements, data were obtained by neutron diffraction since it had advantages over X-ray diffraction such as: larger volume sampling, smaller absorption by the surface irregularities and inappreciable surface oxide effect, all due to the greater penetration of neutrons; no drop off of neutron scattering amplitudes as a function of angle; and easier alignment of the sample due to larger instrument geometry.

The furnace with crystal was then carefully aligned on the neutron diffractometer. The correct θ angle was established by means of an adjustable omega motion in the base of the furnace and the diffracted beam was centered in the counter by means of a shutter assembly which permitted observing the left, right, top and bottom quadrants separately. Integrated intensities were then obtained by step scanning 2θ using a moving crystal-moving counter technique.

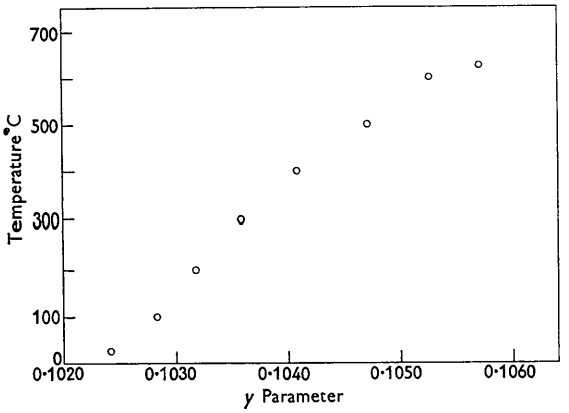

Fig. 1. Plot of *y* positional parameter for alpha uranium as a function of temperature.

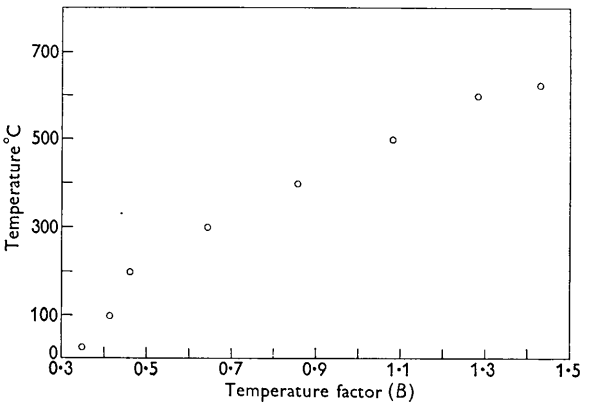

Fig. 2. Plot of temperature factor *B* for alpha uranium as a function of temperature.

Intensity data were obtained for the (040), (060), (080) and (0,10,0) for approximately 100 °C. intervals using a neutron wavelength of 0·98 Å. The (020) was partially masked by the edge of the furnace window.

Plots of *y* and *B* versus temperature are shown in Figs. 1 and 2 respectively, and both *y* and *B* appear to rise gradually with some flattening near 625 °C. Since the interdependence of these two variables was of interest, additional calculations were made on the 625 °C. data holding *B* constant at 1·1 and 0·8. The corresponding *y* values were 0·1057 and 0·1058 indicating little dependence. As shown in Table 1 the room temperature *y* values determined by X-ray and neutron diffraction are in excellent agreement. The results obtained with neutron diffraction at elevated temperatures, however, are considerably lower than the previously reported values.

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References

Busing, W. R. & Levy, H. A. (1959). Oak Ridge National Laboratory Report 59-4-37.

Cash, A. H., Hughes, E. W. & Murdock, C. C. (1961). *Acta Cryst.* **14**, 313.

Chebotarev, N. T. (1961). *Atomnaya Energiya*, **10** (1), 43.

Jacob, C. W. & Warren, B. E. (1937). *J. Amer. Chem. Soc.* **59**, 2588.

Konobeevsky, S. T. *et al.* (1958). Second United Nations International Conference on Peaceful Uses of Atomic Energy.

Sturcken, E. F. & Post, B. (1960). *Acta Cryst.* **13**, 852.