

Oxidation with copper acetate of the mixture of  $\alpha$ -ketols obtained from estrone methyl ether gave 16-ketoestrone methyl ether, obtained as flat needles, orange in color (m. p. 176–178°). It gives a dioxime (m. p. 230°) identical with that prepared by oximating 16-isonitrosoestrone methyl ether. The dione gives an intense violet color with concentrated sulfuric acid.

The dioxime of 16-ketoestrone (free phenol) was also prepared in the hope that this derivative might be useful in detecting the dione in tissue or fluid, should it be present as predicted by Marrian. 16-Ketoestrone dioxime (m. p. 230–231°) gives no colored complex with nickelous or cobaltous ions, but produces a yellowish-green solution with alcoholic copper acetate. The copper complex may be extracted with chloroform, and the color intensity is such that the eye can detect it at the level of 10  $\gamma$  per cc.

The melting points listed above are uncorrected.

We are now attempting to prepare the other isomeric 16-hydroxyestrone and the remaining two isomeric estriols.

DEPARTMENT OF BIOCHEMISTRY      MAX N. HUFFMAN  
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AND SURGEONS      Medical Sciences, 1941–1942  
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### THE CRYSTAL STRUCTURE OF $\beta$ -GLYCYLGLYCINE

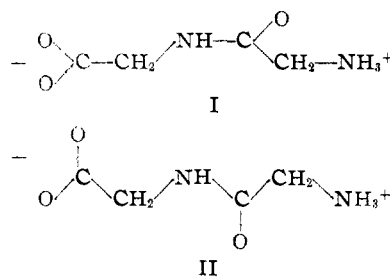
Sir:

Some time ago the determination of the crystal structure of glycylglycine was undertaken as a continuation of the X-ray diffraction studies<sup>1</sup> which are a part of a program of research upon the constitution and configuration of proteins. This determination, the first concerned with a linear peptide, was stopped last year because of the war, and in view of the uncertainty of completing the work the results at hand are briefly described in this letter.

The crystals were grown from aqueous *n*-propyl alcohol and all three modifications described by Bernal<sup>2</sup> were eventually obtained, although not simultaneously as in his crystallization. Because of the shortness of the *b* axis the needle-like beta form was selected for investigation. Using Cu K $\alpha$  rays complete sets of oscillation pictures were

prepared about *a* and *b* and some oscillations were made about *c* to confirm the length of that axis. Weissenberg pictures of the [010] zone were made for intensity comparisons. The cell has *a* = 17.89 Å., *b* = 4.62 Å. and *c* = 17.06 Å., with  $\beta$  = 125°10', and contains eight molecules. This *c* is twice that given by Bernal and the space-group instead of P2<sub>1</sub>/*a* as given by him, is either *Aa* or *A2/a*. The latter was tentatively assumed and present results indicate it is probably correct.

A Patterson projection parallel to *b* yielded preliminary *x* and *z* parameters. The configurations I, suggested by Bernal, and II were tried, and it was found that only with II could the projection be interpreted. The parameters were



improved by Fourier projections and least squares,<sup>3</sup> the latter to resolve CO groups not resolved in the projections. The present agreement between calculated and observed intensities in this zone is good, but minor discrepancies indicate that there may be a few errors in signs or that the contributions from hydrogen atoms should be considered. A tentative set of *y* parameters has also been selected so as to yield reasonable interatomic distances both within and between molecules and these give qualitative agreement between calculated and observed intensities in the [100] zone.

Because of the approximate nature of the parameters, particularly the *y*'s, there is no point in listing interatomic distances. The chief results are first, that the configuration is that of II with the molecule coplanar within present errors except for the terminal nitrogen atom, which lies out of the plane by several tenths of an ångström, and, second, that the zwitterion form is correct. The terminal nitrogen is surrounded at the usual N–H ... O distance by three oxygen atoms of other molecules, two carboxyl and one carbonyl. All the indicated hydrogen bond angles about the nitrogen are tetrahedral to within 10°. The imino nitrogen is also forming a hydrogen bond to a carboxyl oxygen of a neighboring molecule.

(1) Diketopiperazine, R. B. Corey, *THIS JOURNAL*, **60**, 1598 (1938); glycine, G. Albrecht and R. B. Corey, *ibid.*, **61**, 1087 (1939); *dl*-alanine, H. A. Levy and R. B. Corey, *ibid.*, **63**, 2095 (1941).

(2) J. D. Bernal, *Z. Krist.*, **78**, 363 (1931).

(3) E. W. Hughes, *THIS JOURNAL*, **63**, 1737 (1941).

The hydrogen bonds satisfactorily account for the binding together of the crystal.

Although the structure has not yet been completely checked by quantitative comparisons of intensities, particularly with regard to the  $\gamma$

parameters, it is probably substantially correct.

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## NEW BOOKS

**Thorpe's Dictionary of Applied Chemistry.** By JOCELYN FIELD THORPE and M. A. WHITELEY, Assisted by Eminent Contributors. Fourth Edition (Revised and Enlarged), Vol. V, FEH.-Glass, including an Abridged Index to Volumes I-V of the New Edition. Longmans, Green and Company, Inc., 55 Fifth Avenue, New York, N. Y., 1942. xxiii + 610 pp. With illustrations. 15.5 × 23 cm. Price, \$25.00.

The earlier volumes of this Fourth Edition have already been reviewed in *THIS JOURNAL* (59, 2477; 61, 222; 62, 237; 63, 884). This fifth volume of 609 pages covers a part of the alphabet which required 275 pages in the earlier edition. The new edition is almost completely rewritten. There has been a noticeable deterioration in the quality of the paper which is far from uniform in color and texture in the different parts of the book. But there has been no deterioration in the quality of the text. This excellent book has been issued promptly in spite of difficulties modestly described, "When, owing to war conditions, the work of the Dictionary could no longer be carried on in London it was transferred to the University Chemical Laboratory, Cambridge." Another obstacle was the death of the Editor-in-Chief, Sir Jocelyn Thorpe, which occurred on June 10, 1940, the day of publication of Volume IV of the Dictionary. M. A. Whiteley, formerly Associate Editor, has had the editorial responsibility for the completion of this volume, which, however, was well advanced at the time of the death of Sir Jocelyn Thorpe. It is a pleasure to express appreciation and admiration for the typically British determination to carry on successfully in spite of hindrances which would stop many a less resolute people.

GRINNELL JONES

**Liebig and after Liebig.** A Century of Progress in Agricultural Chemistry. Publication of the American Association for the Advancement of Science, No. 16. Publication Committee: CHARLES A. BROWNE, Chairman, RICHARD BRADFIELD, HUBERT B. VICKERY. Edited by FOREST RAY MOULTON. American Association for the Advancement of Science, Smithsonian Institution Building, Washington, D. C., 1942. 111 pp. 19.5 × 26.5 cm.

The ten papers which make up this volume constitute a well-integrated account of the announced subject of the book, namely, of a century of progress in agricultural

chemistry. The Introduction by Charles A. Browne on "Justus von Liebig—Man and Teacher" is followed by Section I, "Organic Chemistry, Enzymes and Nutrition," which contains four papers, "Liebig's Influence in the Promotion of Agricultural Chemical Research" by Henry R. Kraybill, "Liebig and the Chemistry of Proteins" by Hubert B. Vickery, "Liebig and the Chemistry of Enzymes and Fermentation" by Arnold K. Ball, and "Liebig and the Chemistry of Animal Nutrition" by Paul E. Howe, and Section II, "Soils, Fertilizers and the Mineral Requirements of Plants," which contains five papers, "Liebig and the Chemistry of the Soil" by Richard Bradfield, "Liebig—The Humus Theory and the Rôle of Humus in Plant Nutrition" by Selman A. Waksman, "Liebig and the Chemistry of Mineral Fertilizers" by Harry A. Curtis, Liebig and the Law of the Minimum" by Charles A. Browne, and "Liebig and the Mineral Requirements of Plants as Indicated by Means of Solution Cultures" by Burton Livingston. For the chemist who is not familiar with the subject of agricultural chemistry, the book will supply an interesting introduction to it, a general account of the field and of the manner in which it has been cultivated and made to fructify. It is a book which is not by any means to be reserved for the library of the specialist.

The book is clearly printed, two columns to the page, and is fully documented. It is illustrated with five pictures and ten diagrams.

TENNEY L. DAVIS

**The Amphoteric Properties of Proteins.** Vol. XLI, Art. 4 of the Annals of the New York Academy of Sciences. By R. Keith Cannon, A. Kibrick, John G. Kirkwood, L. G. Longsworth, A. H. Palmer and Jacinto Steinhardt. The New York Academy of Sciences, care of the American Museum of Natural History, New York, N. Y. 87 pp. Price \$1.25. This monograph is one of several, which if purchased as a set, may be had at a reduced price.

This contribution to protein literature consists of a series of papers presented at a recent symposium sponsored by the New York Academy of Sciences. It conforms to the usual high standards of other symposia sponsored by this Academy.

The first paper of the series, titled "The Amphoteric Properties of Egg Albumin," by Cannon, Kibrick and Palmer "is devoted to a discussion of the contribution