

de cassure. Le tube contenant les cristaux et 5 cm.³ de liquide est refroidi ou chauffé par un courant d'air à débit constant. Dans le premier cas, on peut faire bouillir de l'air liquide avec une résistance électrique contrôlée par un transformateur variable si l'on veut descendre à de basses températures, sinon, on peut refroidir de l'air comprimé dans un bain de neige carbonique/éther ou glace/sel selon la température voulue. Dans l'autre cas l'air comprimé peut être chauffé au chalumeau.

Pour avoir une précision de 0,01 g.cm.⁻³ sur la densité du liquide il suffit de supposer que la densité varie linéairement avec la température.

Il suffit que la température de l'expérience soit à $\pm 30^\circ$ C. environ de celle à laquelle ont été mesurés les paramètres de la maille, pour obtenir une précision de 0,01 g.cm.⁻³.

Des mesures faites sur des corps de densité connue ont donné des résultats à $\pm 0,01$ g.cm.⁻³. La précision sur les densités des hydrates de soude (mesures faites entre -60 et -100° C.) était aussi de $\pm 0,01$ g.cm.⁻³, sauf dans le cas de NaOH.H₂O, où la différence était de 0,03 g.cm.⁻³.

Les halogénures aliphatiques et aromatiques, ainsi

que le sulfure de carbone permettent de mesurer des densités variant de 0,8–2,5 g.cm.⁻³ à des températures descendant jusqu'à -110° C. environ. Pour ces liquides le coefficient d'expansion thermique est égal à $1,5 \times 10^{-3}$ cm.³/°C. environ. Le 'Fréon' (CF₂Cl₂) fait exception ne gelant qu'à -160° C., et variant en densité de 1,48 à 1,90 g.cm.⁻³.

Cette méthode n'est pas limitée aux mesures à basse température mais peut servir également à la température ambiante et jusqu'à $+200^\circ$ C. environ.

Dans le Tableau I il est présenté une liste de liquides (nullement complète), qui se prêtent bien à cette méthode. Les valeurs dans la troisième colonne ont été extrapolées d'après les valeurs de densité mesurées à plusieurs températures (Timmermans, 1950). Les valeurs dans les autres colonnes ont été tirées du même ouvrage.

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Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.

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Crystal data of Cu-glycylglycine. By Y. HERMODSSON and B. STRANDBERG, *Institute of Chemistry, University of Uppsala, Uppsala, Sweden*

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Metal complexes of dipeptides have aroused interest because of differences in the activation of the enzymatic hydrolysis of dipeptides caused by different metal ions. An attempt to explain the role of metal ions in these hydrolytic reactions reveals that the metal ion forms a bridge in the enzyme-substrate complex. In the case of glycylglycine, Smith (1948) assumes that in the cobaltous complex Co²⁺ links to hydrogen atoms of the two amino groups and possibly also to the hydrogen of the carboxylic group. To clarify this coordination problem, infrared studies on metal complexes of amino acids and dipeptides have been carried out by Rosenberg (1956, 1957) at the Institute of Biochemistry, Uppsala University, and X-ray investigations of complexes with glycylglycine have been started at the Institute of Chemistry, Uppsala. We have chosen to begin with Cu-glycylglycine,



The crystals used were obtained from a sample prepared and analyzed by Rosenberg (1957). The prismatic

crystals were deep blue in colour. The dimensions of the crystal used in the X-ray investigation were $0.10 \times 0.06 \times 0.05$ mm.

The cell dimensions were first approximately settled from measurements on rotation and zero-level Weissenberg photographs. The cell is monoclinic. An accurate determination from powder photographs would be very complicated and a single-crystal method first described by Weiss, Cochran & Cole (1948), called by them the θ -method, was used. Systematic errors in our measurements were eliminated by a certain kind of extrapolation worked out by Löfgren (1957). According to the last named publication, the rotation (b) axis is obtained directly with good accuracy from higher levels with equi-inclination setting without remounting the crystal.

The camera used was an ordinary Weissenberg camera with which it was possible to obtain inclination angles up to 40° . The collimator had a diameter of 0.15 mm. The reflexions chosen had $\theta > 75^\circ$ (in the case of b^* : $\theta = 69^\circ$). The crystal orientation at maximum reflexion was determined by interpolation of estimated intensities

in a series of exposures between each of which the crystal was turned $10'$. The total errors in crystal positions lead to $\Delta\theta = 5'$. No significant errors were to be found in the eccentricity and graduation of the circular scale. The difference between $\theta_{15,0,15}$ and $\theta_{15,0,15}^-$ was only $2'$.

The following direct lattice elements were obtained:

$$a = 14.95 \pm 0.02, \quad b = 7.54 \pm 0.03, \quad c = 15.80 \pm 0.02 \text{ \AA}; \\ \beta = 102^\circ 13' \pm 7'.$$

They were measured with both $\text{Cu } K\alpha_1$, and $\text{Cu } K\alpha_2$ radiations. The agreement is very good: the differences are smaller than the theoretical errors obtained with $\Delta\theta = 5'$.

The equatorial vectors chosen have approximately the directions of a , c and the normal to the bisector of β^* . Owing to this, the errors in the direct cell constants are of the same order of magnitude as the measured reciprocal vectors.

Systematic extinctions were found for $h0l$ reflexions with $h+l$ odd and for $0k0$ reflexions with k odd. The space group is thus $P2_1/n$, in agreement with the absence of a piezoelectric effect. (We are indebted to Mr J. Baeklund for the use of his apparatus.)

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Unit cell and space group for sodium tetraborate, $\text{Na}_2\text{O} \cdot 4\text{B}_2\text{O}_3$. By J. KROGH-MOE, *Department of Ceramic Technology, The Pennsylvania State University, University Park, Pa., U.S.A.*

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Morey & Merwin (1936) have reported two modifications of sodium tetraborate. The stable modification at high temperatures separates at 816°C . from a melt of the stoichiometric composition $\text{Na}_2\text{O} \cdot 4\text{B}_2\text{O}_3$. Single crystals suitable for X-ray work were grown in a platinum crucible at 700°C . from a melt containing an excess of boron oxide. X-ray powder patterns established these crystals as identical with the phase crystallizing at 816°C . from the melt of stoichiometric composition.

Oscillation and Weissenberg exposures around three axes revealed a monoclinic symmetry. The following dimensions for the unit cell were found:

$$a = 6.50, \quad b = 17.80, \quad c = 8.37 \text{ \AA}, \quad \beta = 96.5^\circ.$$

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The crystal structure of urea phosphate. By R. V. G. SUNDERA-RAO, JUNE W. TURLEY and RAY PEPINSKY, *X-Ray and Crystal Analysis Laboratory, Department of Physics, The Pennsylvania State University, University Park, Pa., U.S.A.*

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A preliminary analysis, in this laboratory, of the crystal structure of urea phosphate, $\text{OC}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4$, has been reported by Sundera-Rao & Pepinsky (1954). That analysis was based upon (010) and (001) projections. Indications of a very interesting hydrogen-bond system prompted a complete three-dimensional study. Some results of the complete analysis are reported here.

The density was determined by the flotation and the pycnometric methods, both of which gave $1.89 \pm 0.02 \text{ g.cm.}^{-3}$. On the basis of eight formula units per unit cell the calculated density is 1.89 g.cm.^{-3} . The number of formula units in the unit cell indicates that there are either two non-equivalent molecules or a dimeric molecule in the crystalline state. A complete crystal-structure determination has been started at this Institute.

We wish to thank Prof. G. Hägg for all the facilities placed at our disposal and Prof. A. Tiselius for his great interest. Thanks are also due to Dr I. Lindqvist, Mr T. Löfgren and A. Rosenberg for valuable discussions. These investigations were made possible by a grant from The Royal Swedish Academy of Sciences (Wallmarkska fonden) for which we express our thanks.

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These figures are estimated to be accurate within 0.5%. With four $\text{Na}_2\text{O} \cdot 4\text{B}_2\text{O}_3$ in the unit cell, the calculated density is 2.35 g.cm.^{-3} . A density of 2.32 g.cm.^{-3} was observed by the flotation method.

Systematic extinctions were observed only in the $0k0$ reflections, the extinctions occurring for $k = 2n + 1$. This is compatible with the space group $P2_1$ or $P2_1/m$.

Reference

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The crystals are orthorhombic with

$$a = 17.68, \quad b = 7.48, \quad c = 9.06 \text{ \AA};$$

the observed density is 1.770 g.cm.^{-3} . With $Z = 8$ molecules per unit cell, the asymmetric unit contains one $\text{OC}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4$. Systematic extinctions indicate space group $Pbca$.