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Crystallographic data for some idose derivatives.

By SHIRLEY S. C. CHU and G. A. JEFFREY, *The Crystallography Laboratory, The University of Pittsburgh, Pittsburgh* 13, *Pa., U.S.A.*

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The suggestion by Bentley (1960) that the β-idopyranoside molecules exist in solution in a half-chair configuration, *HC*3, has prompted the examination of the crystal data of some methylbenzal derivatives. The data are given in Table 1. They were measured from single-crystal oscillation and Weissenberg photographs with Cu *K*α radiation. The limits on the unit-cell dimensions are ±0·05%. The densities were measured by flotation. The space groups were determined from the systematic extinctions. The derivative with the short *c* axis is being further studied.

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References

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Table 1. *Crystal data of some methylbenzal idosides*

	Methyl-4, 6-benzal α-D-idoside	Methyl-4, 6-benzal β-D-idoside H_2O	Methyl-4, 6-benzal- -3-methyl β-idoside	2, 3-dimethyl- -4, 6-benzal- methyl β-idoside
a	16·72	22·22	22·38	20·76
b	10·21	11·31	13·00	9·10
c	7·92	11·05	5·00	8·40
D_m	1·389	1·386	1·359	1·295
D_x	1·387	1·389	1·353	1·299
Z	4	8	4	4
Space group	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$

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Données cristallographiques sur quelques diamides organiques.

Par MICHEL HOSPITAL et JACQUES HOUSTY. *Laboratoire de Minéralogie et Cristallographie Physique, Faculté des Sciences de Bordeaux, France*

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Malonamide, $CONH_2 . CH_2 . CONH_2$

Le malonamide cristallise à partir d'une solution aqueuse dans le système monoclinique sous forme de fines et longues aiguilles transparentes allongées suivant la direction [001].

Les paramètres obtenus par oscillation d'un cristal autour de sa rangée [001] dans une chambre de Bragg sont les suivants:

$$a=13{,}63\pm0{,}02,\ b=9{,}47\pm0{,}01\ ,\quad c=8{,}06\pm0{,}01\ \text{Å},\ \beta=113°\ 10'\ .$$

Nombre de molécules par maille: 8
Densité calculée: 1,416
Groupe spatial: $P2_1/c$

Adipamide, $CONH_2 . (CH_2)_4 . CONH_2$

L'adipamide est obtenu sous forme d'aiguilles cristallines par sublimation. La direction d'allongement est [010] dans le système monoclinique.

Les paramètres sont les suivants:

$$a=6{,}89\pm0{,}01,\ b=5{,}14\pm0{,}01\ ,\quad c=10{,}67\pm0{,}01\ \text{Å};\ \beta=111°\ .$$

Nombre de molécules par maille: 2
Densité calculée: 1,367
Groupe spatial: $P2/c$

Suberamide, $CONH_2 . (CH_2)_6 . CONH_2$

Le suberamide cristallise à partir d'une solution dans un mélange d'alcools amylique, propylique et éthylique, sous forme de plaquettes facilement clivables suivant la direction [010]. Le système est monoclinique.

Les paramètres obtenus par oscillation d'un cristal dans une chambre de Bragg sont les suivants:

$$a=14{,}44\pm0{,}02,\ b=5{,}13\pm0{,}01\ ,\quad c=14{,}17\pm0{,}02\ \text{Å};\ \beta=117°\ 30'\ .$$

Nombre de molécules par maille: 4
Densité calculée: 1,225
Groupe spatial: $C2/c$

Toutes les mesures cristallographiques ont été réalisées avec la radiation Cu *K*α.

La détermination des structures cristallines de ces composés est actuellement en cours.

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The crystal structure of lithium glycollate monohydrate.

By R. H. COLTON and D. E. HENN, *Basic Physics Division, National Physical Laboratory, Teddington, Middlesex, England*

(*Received* 20 *October* 1964)

Introduction

Pooley & Whiffen (1961) have studied by electron spin resonance the $H\dot{C}(OH)CO_2^-$ radical-ion in γ-irradiated crystals of both anhydrous lithium glycollate and its monohydrate. In order to resolve the ambiguity of the sign of the isotropic H(O) coupling it was necessary to determine the structure of one of these crystals. This paper describes the determination of the crystal structure of the monohydrate.

Crystal data

At temperatures below 40 °C lithium glycollate crystallizes from aqueous solution as the monohydrate, often in the form of twinned crystals. Above 40 °C the anhydrous salt is formed.

The monoclinic cell contains four molecules of lithium glycollate and four water molecules and has dimensions

$$a=11{\cdot}21\pm0{\cdot}01,\ b=6{\cdot}80\pm0{\cdot}01,\ c=5{\cdot}97\pm0{\cdot}01\ \text{Å};\quad \beta=108{\cdot}2°\pm0{\cdot}1°\,.$$

The measured density is 1·542 g.cm^{-3}; the calculated density is 1·546 g.cm^{-3}.

Reflexions *hkl* are absent for odd $h+k$, suggesting one of three space groups *C*2, *Cm*, or *C*2/*m*. Electron spin resonance studies show that the radical ion formed from the glycollate ion on γ-irradiation lies in a plane of symmetry perpendicular to the *b* axis. These results are consistent with the space group *C*2/*m* with the glycollate ion lying in the plane of symmetry.

Determination of the structure

The intensities of some 385 independent reflexions were determined by photometric measurement on multiple-film integrated Weissenberg photographs (Cu $K\alpha$ radiation). Equi-inclination photographs were taken about the *b* axis for layers with $k=0$ to $k=6$. Intensities were corrected for Lorentz and polarization factors but no correction was made for absorption.

Preliminary atomic coordinates for the carbon and oxygen atoms were determined from the three-dimensional Patterson function. An F_o Fourier synthesis phased on the carbon and oxygen atom positions indicated the position of the lithium ion. Atomic positional parameters were refined by a series of Fourier syntheses and difference Fourier syntheses. Positional and anisotropic thermal-vibration parameters were further refined on the DEUCE computer by means of the least-squares program of Rollett (1961). The weighting scheme throughout was $\sqrt{\omega}=1$ if $|F_o|<F^*$ otherwise $\sqrt{\omega}=F^*/|F_o|$ where F^* is a selected constant. In the later stages of refinement the contributions of the hydrogen atoms, based on estimated hydrogen atom positions, were included in the structure factor calculations but their parameters were not refined. Refinement ceased at $R=0{\cdot}11$ when the least-squares shifts were less than one third the corresponding standard deviations. Tables 1 and 2 list the final fractional atomic coordinates and anisotropic vibration parameters.

Table 1. *Final fractional atomic coordinates*

Atom	*x/a*	*y/b*	*z/c*
C(1)	0·2378	0	0·6921
C(2)	0·3040	0	0·4955
O(1)	0·1235	0	0·6347
O(2)	0·3095	0	0·9078
O(3)	0·2119	0	0·2698
OH_2	0	0·2206	0
Li^+	0·0310	0	0·2595

Table 2. *Vibration parameters*

$$f=f_o[\exp\{-\ln 2(h^2b_{11}+k^2b_{22}+l^2b_{33}+klb_{23}+lhb_{31}+hkb_{12})\}]$$

Atom	b_{11}	b_{22}	b_{33}	b_{23}	b_{31}	b_{12}
C(1)	0·00549	0·00101	0·01883	0	0·00099	0
C(2)	0·00523	0·01052	0·01937	0	0·00323	0
O(1)	0·00553	0·01375	0·01820	0	0·00451	0
O(2)	0·00628	0·01057	0·01712	0	0·00124	0
O(3)	0·00598	0·03163	0·01671	0	0·00286	0
OH_2	0·00638	0·00300	0·02372	0	0·00069	0
Li^+	0·00535	0·01331	0·01786	0	0·00634	0

Discussion

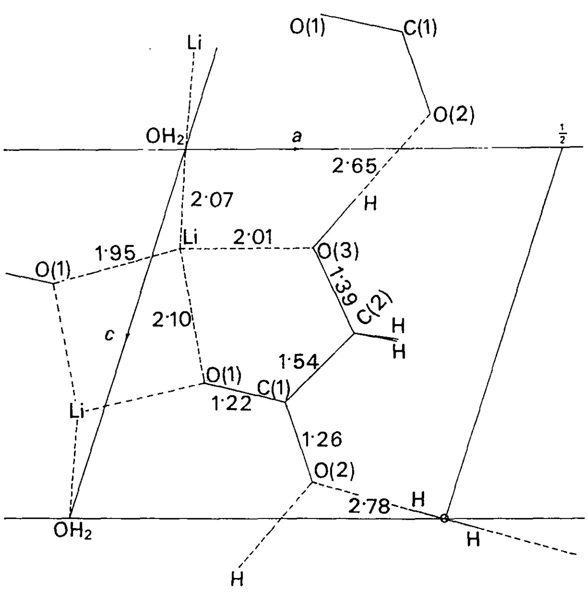

Fig. 1.

Fig. 1 shows the arrangement of the lithium and glycollate ions in the mirror plane. The water molecules lie on the twofold axes symmetrically on either side of the plane and serve to bind the sheets together by hydrogen bonding to the carboxyl oxygen atoms in one layer while completing the lithium coordination in the layer above. The lithium ion is at the centre of a slightly distorted trigonal bipyramid of oxygen atoms. Table 3 lists some of the interatomic distances and angles with their standard deviations. Copies of the list of final structure factors may be obtained from the authors. The electron spin resonance studies are in complete agreement with the structure found and the

Table 3. *Interatomic distances and angles*

Bond	*d*	σ
C(1) C(2)	1·538 Å	0·0117 Å
C(1) O(1)	1·220	0·0096
C(1) O(2)	1·260	0·0095
C(2) O(3)	1·393	0·0102
Li OH_2	2·074	0·0104
Li O(1)	2·098	0·0148
Li O(3)	2·011	0·0145
O(2) · · · OH_2	2·784	0·0047
O(3) · · · O(2)	2·647	0·0070

Angle		σ
O(3) C(2) C(1)	107° 52′	35′
O(1) C(1) C(2)	120 21	37
O(2) C(1) C(2)	115 20	35
O(1) C(1) O(2)	124 19	34

ambiguity of the sign of the H(O) isotropic coupling has been resolved (Henn & Whiffen, 1964).

This work forms part of the research programme of the Basic Physics Division, National Physical Laboratory and is published by permission of the Director of the Laboratory.

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Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the General Secretary of the International Union of Crystallography (D. W. Smits, Rekencentrum der Rijksuniversiteit, Grote Appelstraat 11, Groningen, The Netherlands). Publication of an item in a particular issue cannot be guaranteed unless the draft is received 8 weeks before the date of publication.

Summer school on OD arrangements and structure analysis

A Summer School on OD Arrangements and Structure Analysis will be arranged by the Institut für Strukturforschung, Deutsche Akademie der Wissenschaften zu Berlin, at this Institute, Berlin-Adlershof, from 30 July to 13 August 1965.

The aim of the School is to make crystallographers who are already familiar with the concepts and international nomenclature of space groups, reciprocal space, Fourier and Patterson methods, acquainted with the concepts of OD arrangements and to give them a working knowledge of methods for the determination of the OD-groupoid family and its parameters from the distribution of diffuse streaks and sharp points in reciprocal space and the symmetry of the intensity distribution. Patterson and Fourier methods specialized for OD arrangements will be discussed.

Enrolment forms and further information may be obtained from the Secretary, Institut für Strukturforschung, Berlin-Adlershof, Rudower Chaussee 5, Germany.

Book Reviews

Works intended for notice in this column should be sent direct to the Editor (A. J. C. Wilson, Department of Physics, Georgia Institute of Technology, Atlanta, Georgia 30332, *U.S.A.). As far as practicable books will be reviewed in a country different from that of publication.*

Colloques internationaux du Centre National de la Recherche Scientifique. No. 126. La diffraction et la diffusion des neutrons, Grenoble 3–5 Septembre 1963. Pp. 236. Paris: Éditions du Centre National de la Recherche Scientifique, 1964. Price 25 F.

A colloquium, attended by about 60 participants and 40 'auditeurs', was held at Grenoble from 3 to 5 September 1963, under the auspices of the Centre National de la Recherche Scientifique. The President of the committee was Professor Néel, and the Secretary, largely responsible for the organization, was Dr E. F. Bertaut. The proceedings of the colloquium were published in the *Journal de Physique* for May 1964, and are here conveniently reprinted.

The publication contains 59 papers, about equally divided between French and English. Their length varies considerably; some, intended for full publication elsewhere, are little more than extended abstracts, whereas others run to seven or eight large pages. Each paper is followed by a report of the discussion on it.

The published record of the colloquium is very impressive. Rather less than one-third of the papers are concerned with the solution of what might be called conventional structural problems by neutron diffraction, and rather more than one-third with the solution of magnetic structures. The remaining papers are concerned with instrumentation, anomalous dispersion, and scattering. Neutron crystallographers, and perhaps even more non-neutron crystallographers interested in learning something of the scope and power of neutron methods, will find this reprint of the papers extremely useful.

A. J. C. Wilson

University College
Cardiff
Wales

Practical optical crystallography. By N. H. Hartshorne and A. Stuart. Pp. vii + 326 with 1 folding chart, 2 pl. + 253 figs. London: Arnold. Price 40*s*.

This book represents a new approach to the problem of teaching optics to students working in the fields of geology, ceramics, crystallography, and chemistry who need to use a polarizing microscope. It is intended to bridge the long felt gap between optical theory and the practical manipulation of crystalline material upon the microscope stage and as such is to be welcomed.