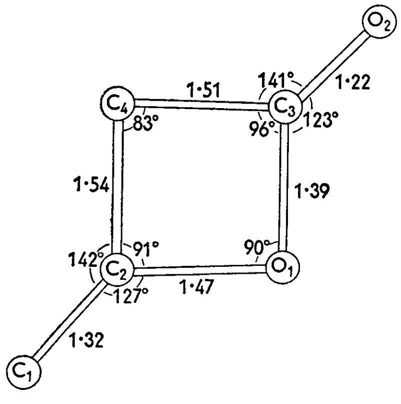

Fig. 1. Dimensions of diketene molecule.

of Katz and Lipscomb were used for the refinement (non-observed reflections were omitted). R dropped from 0·14 to 0·11 in 6 cycles. Numbering the atoms as in Fig. 1, the new coordinates are listed in Table 1. The new bond lengths and angles are indicated in Fig. 1 and, together with the old values of Katz and Lipscomb and the electron diffraction results of Bauer & Bregman, are given in Table 2. Standard deviations for the new results are about 0·03 Å for bond lengths, 1·5° for bond angles.

The C–C single bond lengths are in the usual range, confirming the absence of an appreciable amount of double bond character. The C_2–O_1 and the C_3–O_1 single bonds are, however, shorter and longer, respectively, than the normal C–O single bond distance of 1·42 Å. While the differences fall within twice the standard deviations, it should be noted that such shortening and lengthening of C–O single bonds α and β to a C–O double bond have been observed in methyl acetate, methyl formate, and methyl chloroformate (O'Gorman, Shand & Schomaker, 1950); in dimethyl terephthalate (Bailey, 1949); and in dimethyl oxalate (Dougill & Jeffrey, 1953). This effect in esters is probably a result of the usual resonance in carboxyl groups (Pauling, 1940). It is also of interest to note that the temperature factors for the atoms outside the ring are higher than those for the atoms in the ring.

Table 2. *Bond lengths and angles from old X-ray determination (K and L), electron diffraction (B and B), this refinement (K and K)*

Bond lengths	*K* and *L*	*B* and *B*	*K* and *K*
C_1–C_2	1·35 Å	1·31 Å	1·32 Å
C_2–C_4	1·48	1·52	1·54
C_3–C_4	1·46	1·52	1·51
C_3–O_2	1·24	1·19	1·22
C_3–O_1	1·40	1·41	1·39
C_2–O_1	1·39	1·41	1·47
Bond angles			
C_1–C_2–O_1	130°	130°*	126·9°
C_1–C_2–C_4	136	136*	141·6
C_4–C_2–O_1	94	95	91·3
C_2–C_4–C_3	83	81	83·0
C_4–C_3–O_1	94·5	95	95·8
C_4–C_3–O_2	145	145*	140·9
O_2–C_3–O_1	121	121*	123·1
C_3–O_1–C_2	89	89	90·0

* Assumed value.

We would like to express our thanks to Prof. V. Vand of the Pennsylvania State University, who ran the refinement with his modification of Sayre's 704 least-squares program. This work was supported in part by the United States Atomic Energy Commission.

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Space group of *o*-tolidine.

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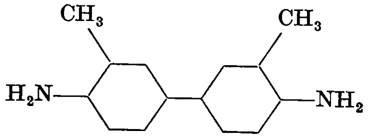
Clark & Pickett (1931) examined *o*-tolidine $C_{14}H_{16}N_2$ with structural formula



by both X-ray and optical methods and found it to be orthorhombic with cell dimensions: $a = 6·50$; $b = 7·48$ and $c = 23·62$ Å, with four molecules per unit cell. The space group was found to be either Q^3 or Q^4 depending upon the presence or absence of $(0k0)$ with k odd. The present work was undertaken to find out the correct space group of *o*-tolidine in preparation for complete structure determination.

Good crystals of *o*-tolidine, slightly brown in colour, were obtained from distilled alcohol. They exhibited extinctions parallel to the axes under the polarizing microscope. Employing unfiltered Co $K\alpha$ radiation, rotation and Weissenberg zero-layer and equi-inclination photographs were taken. The crystal was found to be orthorhombic. The systematic absenses are: $(h00)$ when h is odd, $(00l)$ when l is odd. Over-exposed a axis zero-layer Weissenberg photograph showed a sharp reflexion in the (050) position and very weak impression of a reflexion in the (010) position; these, however, did not appear in even highly over-exposed c-axis zero-layer Weissenberg photograph.

The (050) spot is a sharp one and is not accompanied by the usual β spot which other equally intense normal spots have. So these two reflexions were suspected to be double reflexions (Renninger, 1937). They were verified to be so by drawing the reciprocal lattice and considering the reflexion conditions graphically and also analytically. The pairs of reflexions responsible for the formation of (050) are ($12\bar{2}$, $\bar{1}32$) and (203, $\bar{2}5\bar{3}$), and that responsible for (010) reflexion is ($1\bar{2}3$, $\bar{1}3\bar{3}$). The normal ($0k0$) reflexions with k odd are thus absent. No other systematic absences are found. Hence the space group of o-tolidine is $P2_12_12_1(Q^4\text{–}D_2^4)$.

Unit-cell dimensions were measured from carefully calibrated rotation and zero-layer Weissenberg photographs. High-angle reflexions, where the $K\alpha_1\alpha_2$ doublets were well resolved, were used. The dimensions were found to be: $a = 6{\cdot}412$, $b = 7{\cdot}490$ and $c = 23{\cdot}758$ Å. (The wave lengths of Co $K\alpha_1$ and $K\alpha_2$ have been taken as 1·78892 Å and 1·79278 Å respectively.) The density of o-tolidine is 1·234 g.cm.$^{-3}$ as obtained by the flotation method using $ZnSO_4$. The number of molecules per unit cell is 4 and the density calculated is 1·236 g.cm.$^{-3}$. The observed and calculated interfacial angles are:

Angle	Obs.	Calc.
(001) : (012)	57° 51′	57° 47′
(001) : (101)	74° 50′	74° 54′

The author wishes to express his thanks to Prof. B. N. Srivastava, D.Sc., F.N.I., for his keen interest in the problem and to Dr B. V. R. Murty, for suggesting the problem and guidance throughout the piece of work.

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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. Copy should be sent direct to the Editor (P. P. Ewald, Polytechnic Institute of Brooklyn, 333 *Jay Street, Brooklyn* 1, *N.Y., U.S.A.) or to the Technical Editor (R. W. Asmussen, Chemical Laboratory B of the Technical University of Denmark, Sølvgade* 83, *Copenhagen K, Denmark)*

Tables of Interatomic Distances and Configuration in Molecules and Ions

The Editors of Volume III of the International Tables for X-ray Crystallography have had a difficult task in deciding where to stop. This Volume is intended to give Tables of physical and chemical data which will be of value in crystal structure determination and in related fields. Obviously it is of the greatest importance in crystal analysis to have information about interatomic distances and molecular configurations in previously analysed structures. Yet to give them all would mean a serious loss of balance and increase of size of the Volume.

This problem has now been solved by the appearance of Special Publication No. 11 of the Chemical Society of London: '*Interatomic Distances and Configuration in Molecules and Ions*', which at £ 2.2.0 (U.S.A. $ 6·00) is such excellent value that every crystallographer should have a personal copy as well as recommending it to any library used by science students, teachers or research workers. The Scientific Editor, Dr. L. E. Sutton, gave a lecture at the Fourth International Congress in Crystallography held in Montreal in 1957, on the general subject of this most noteworthy publication.

Kathleen Lonsdale

Book Reviews

Works intended for notice in this column should be sent direct to the Editor (P. P. Ewald, Polytechnic Institute of Brooklyn, 333, *Jay Street, Brooklyn* 1, *N.Y., U.S.A.). As far as practicable books will be reviewed in a country different from that of publication.*

Structure Reports for 1951. Volume 15. A. J. C. Wilson, general editor. Published for the International Union of Crystallography. Utrecht: N.V. A. Oosthoek's Uitgevers Mij. 1957, vii+588 pp., 145 figs. Price (bound) 110 Dutch florins, £10.10.0, $29·00.

A concise summary of the results attained by the X-ray analysis of various substances began as Strukturbericht in 1931 (for 1913–1928), continued as Structure Reports, and has by now covered the long period of X-ray studies of structures from 1913 to 1951. The present volume, marked as Volume 15 for 1951 follows Volume 13 for 1950, but as mentioned in the prospectus—but not in the volume itself—the vacant position to be filled by volume 14 is reserved for cumulative indexes for the previous volumes. The laborious task of presenting the data is done by a team of editors (one general, three sectional) and twenty six abstractors. As on former occasions the volume is divided into three sections: