

choice of the gain parameter of the system can serve that desired purpose (Biswas, 1966).

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## SPACE GROUP OF O-BENZOYL BENZOIC ACID

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The crystals of o-benzoyl benzoic acid, (chemical formula  $C_6H_5COC_6H_4COOH$  and m.p.  $127^\circ C$ ), obtained from its saturated solution both in absolute alcohol and benzene by slow evaporation, are transparent prismatic needles. Crystals from alcohol show, in general, eight faces about the needle axis, but those from benzene show six faces. The preliminary optical study of a crystal was carried out with an optical goniometer and since all the faces did not give prominent reflections, interfacial angles could only be measured approximately.

The axial lengths,  $a = 7.71 \text{ \AA}$ ,  $b = 8.28 \text{ \AA}$  and  $c = 9.95 \text{ \AA}$  were determined from the rotation photographs about the proposed [100]-, [010]- and [001]- axes. The zero-level normal beam weissenberg photographs were also taken about these three axes. From the symmetry of the weissenberg photographs and other considerations it was confirmed that the crystal belongs to the triclinic system. The positive directions of  $a$ ,  $b$  and  $c$  axes with the condition  $a < b < c$ , were chosen according to the standard practice in right handed system.

The angles between the faces (100), (010) and (001), obtained from the zero-level weissenberg photographs, are

$$\alpha^* = 76^\circ, \quad \beta^* = 96^\circ \quad \text{and} \quad \gamma^* = 92^\circ 30'$$

The values of the axial angles, calculated directly from these values with the help of standard formulae, are

$$\alpha = 103^\circ 50', \quad \beta = 84^\circ 26' \quad \text{and} \quad \gamma = 88^\circ 56'$$

The axial lengths calculated from weissenberg photograph are consistent with those obtained from rotation photographs. The calculated values are

$$a = 7.72 \text{ \AA}, \quad b = 8.27 \text{ \AA}, \quad c = 9.93 \text{ \AA}$$

The volume of the unit cell ( $V$ ) was found to be  $609 \text{ \AA}^3$ .

The density of the crystal, measured by floatation method using a mixture of carbon tetrachloride and benzyl chloride, was  $1.29 \text{ gm/c.c.}$  The number of molecular units per unit cell ( $Z$ ) was found to be two and the measured density is consistent with the theoretical density obtained from the X-ray measurement on the basis that  $Z = 2$  and  $V = 609 \text{ \AA}^3$ .

Since the crystal belongs to the triclinic system, no systematic extinctions are possible and this was also confirmed from the indices of the spots on the weissenberg photographs. Hence the space group of the crystal is either  $P1$  or  $P\bar{1}$ . The presence of centre of symmetry in the unit cell was confirmed by the statistical tests of the intensity distributions of  $okl$  and  $hol$  reflections. For this purpose, the integrated intensities of the X-ray reflections were estimated visually by comparison with a standard graded intensity scale. The intensities were corrected for Lorentz and polarisation factors. The results of the statistical tests applied to  $okl$  and  $hol$  reflections are shown in the fig. 1.

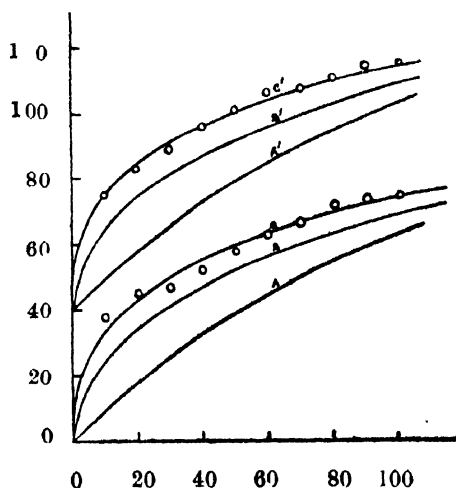


Fig. 1—Intensity distribution curves for O-benzyl benzoic acid.

A, A'—theoretical acentric, B, B'—theoretical centric,

C—experimental hypercentric for  $okl$  reflections,

C'—experimental hypercentric for  $hol$  reflections,

A', B' and C' are displaced 40% vertically.

Ordinate— $N(Z)\%$ , Abscissa— $Z\%$

Results show that the experimental curves are hypercentric in nature. Further Wilson's ratios for  $okl$  and  $hol$  reflections were found to be 0.540 and 0.500 res-

pectively as against the theoretical value 0.637 for centrosymmetrical crystals. So, the space group of the crystal must be centrosymmetric i.e.  $\bar{P}T$ .

Hypercentric nature of the intensity distribution curves not only shows the presence of centre of symmetry in the space group but also indicates the presence of a molecular centre of symmetry in the structure (Lipson *et. al.*, 1952). But for this particular case, the presence of molecular centre of symmetry does not follow the chemical structure of the compound. Molecular centre of symmetry, in this case, may be explained on the basis that two molecular units in a unit cell form a dimer and centre of symmetry of the dimer thus coincides with that of the unit cell.

Further work is in progress.

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## RENNINGER EFFECT IN QUINALDIC ACID

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While the study of space-group of quinaldic acid was made some weak reflections different from the nature of the normal ones were observed in the position (105) and (10 $\bar{5}$ ). Thus their presence was a hurdle in ascertaining the space-group of the crystal. A critical study of the space-group and extinction condition was made which reveals the origin due to double reflection from a pair of strong plane (Srivastava 1957).

The forbidden reflection  $s$  (105) and (10 $\bar{5}$ ) was not accompanied by its usual  $\text{CuK}_\beta$  reflection using unfiltered  $\text{CuK}$  radiation while other reflections are accompanied with the usual  $\text{CuK}_\beta$  reflections. The study of the reflection conditions of the reciprocal lattice confirms the presence of Renninger's reflection (1937). The mechanism of the formation of double reflections can be understood by constructing the reciprocal lattice for this crystal. As an example we shall consider the forbidden reflection (105) in details.

The unit cell dimensions of quinaldic acid (Khan, 1967) were determined by oscillation and Weissenberg photograph by standardising the camera diameter, and from a consideration of the high angle spots (where  $K_{\alpha_1\alpha_2}$  doublet is well re-