These data correspond to values of $\alpha$ of 1.06 and 1.03, again indicating that exchange is complete before chlorination takes place.

While these experiments are of a preliminary nature they do indicate that in the present favorable case there is no indication but that the velocity of the exchange reaction is far greater than that of the chlorination. As an investigation of chlorination reactions the method seems to hold little promise. It is, of course, not impossible that the $Br_2$–$Br^-$ or the $I_2$–$I^-$ exchanges might be sufficiently slow so that significant measures of rates of halogenation could be obtained in these cases.

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## Crystal Form of Sucrose Octaacetate

BY C. D. WEST

When an acetone solution of sucrose octaacetate[1] is allowed to stand at room temperature for several weeks, well-formed prisms of m. p. 84.5°, density 1.335, develop which are suitable for goniometric measurement. The expectation that this optically active molecule would crystallize in one of the enantiomorphous symmetry classes is confirmed by the finding of orthorhombic bisphenoidal symmetry (point group V), as shown by the accompanying orthogonal projection.
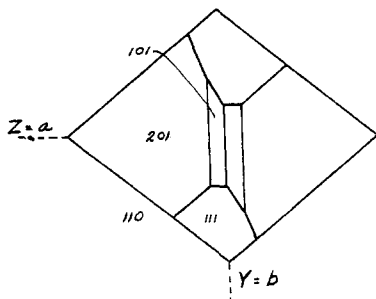


Fig. 1.—End faces and optical orientation.

The elements as calculated for a single crystal, the best of several measured, are set out in the table.

(1) From Niacet Chemicals Corporation, Niagara Falls, N. Y.

### TABLE I

TWO CIRCLE ANGLES OF SUCROSE OCTAACETATE

$a:b:c = 1.2156:1:0.4605;$  $p_0:q_0:r_0 = 0.3789:0.4605:1$

| hkl | Faces | Measured $\varphi$ | Mean $\rho$ | | Calculated $\varphi$ | $\rho$ |
|-----|-------|---------|------|------|---------|------|
| 110 | 4 | 39°27′ | 90°0′ | | 39°26$\frac{1}{2}$′ | 90°0′ |
| 101 | 2 | 90  0 | 20 16 | | 90  0 | 20 45 |
| 201 | 2 | 90  0 | 37 10 | | 90  0 | 37  9 |
| 111 | 2 | 39 26 | 30 48 | | 39 26$\frac{1}{2}$ | 30 48$\frac{1}{2}$ |

Cleavage planes are absent, and doubly terminated prisms were not noted. Crystals from ethanol solution have the same melting point and show the same faces. Optically the crystals are biaxial negative with the orientation $\alpha//c$, $\beta//b$, $\gamma//a$; the refractive indices are identical with Brandt's published values, namely, $\alpha = 1.470$, $\beta = 1.488$, $\gamma = 1.500$, all $\pm 0.002$ (2). Dispersion of the optic axes was not observed. The present crystals are thus identified with the stable, unsolvated form of sucrose octaacetate of m. p. 89° in the pure state.[2]

For the glassy form of this material the reported constants $n^{20}$D $= 1.4660$, $d. = 1.28^3$ yield the specific refraction $\dfrac{n^2 - 1}{n^2 + 2} \cdot \dfrac{1}{d} = 0.2164$. The same constant for the crystals, as calculated from the average refractive index 1.486, is 0.2150.

The writer is indebted to Dr. Harry Berman for the opportunity to make the foregoing angle measurements at the Harvard Mineralogical Laboratories.

(2) In Linstead, Rutenberg, Dauben and Evans, THIS JOURNAL, **62**, 3260 (1940).
(3) Cox, Ferguson and Dodds, *Ind. Eng. Chem.*, **25**, 968 (1933).

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## NEW COMPOUNDS

### Derivatives of Piperazine. XIX. Reactions with Aryl Sulfonyl Chlorides and Aryl Sulfonic Acids

**N,N′-Bis-(arylsulfonyl)-piperazines.[1]**—The benzene, p-toluene, p-bromobenzene, and 2-nitrotoluene sulfonyl derivatives were prepared by adding with stirring a hot ethanolic solution of 0.1 mole of the appropriate sulfonyl chloride to an ethanolic solution of 0.05 mole (9.7 g.) of piperazine hexahydrate which contained a suspension of 6 g. of anhydrous sodium carbonate. The o-toluenesulfonyl derivative was prepared by gently refluxing 34 g. of o-toluenesulfonamide, 65 g. of ethylene bromide, and 30

(1) The melting points of N,N′-bis-(benzenesulfonyl)-piperazine[1] and N,N′-bis-(p-toluenesulfonyl)-piperazine[2] have been reported in the literature as being 282–283 and 286°, respectively, values which are lower than those which we have found for these compounds.