

From the point of view of the scientist, however, the use of this film offers the great advantage that one can do the processing oneself. This is of particular value, because it permits one to ascertain immediately if the microphotographs are satisfactory or not. In the latter case, a new picture can be exposed before the preparation, which frequently changes quite rapidly with time, has become useless.

We thought that this information would be of interest to those who are already following the suggestions we made in the above-mentioned publication.

DEPARTMENT OF CHEMICAL ENGINEERING  
MASSACHUSETTS INST. OF TECH. ERNST A. HAUSER  
CAMBRIDGE 39, MASS. DESIRÉE S. LE BEAU

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### RECRYSTALLIZATION OF AMORPHOUS CELLULOSE Sir:

Treating very dry cellulose in an efficient grinding device, such as a vibrating ball mill, yields a powder whose X-ray diffraction pattern reveals that all three-dimensional lattice order has disappeared. Upon wetting the powder with hot water a recrystallization occurs, the crystalline diffraction pattern which then appears being invariably that of cellulose II (hydrate cellulose) when either native or regenerated fibers are used as starting material.<sup>1</sup> We have repeated such experiments with ramie, woodpulp and viscose rayon. Diffraction patterns of the amorphous powders show a broad band whose maximum occurs slightly within the angular position of the  $A_3(10\bar{1})$  interference of cellulose II. Upon recrystallization in water the band's intensity is considerably reduced, the spectrum of cellulose II being superimposed on it.

The degree of crystallization has been computed in three ways. In one method the original powder was assumed to be 100% amorphous and the percentage of amorphous substance in the recrystallized powder was estimated to be 62% from the photometrically determined ratio of the background intensities at the site of the aforementioned maximum. Correction was made for scattering due to air and to the thin-walled capillary containing the sample.

In the second and third methods the integral heats of wetting and the sorption isotherms of the very dry amorphous and recrystallized powders are employed. The data obtained (corrected for a small amount of porcelain contamination resulting from grinding) were:

	Sorption ratio	Integral heat of wetting, cal./g.
Wood Pulp		
Original fibers	1.25	14.3
Ground product	2.12	29.0
Recrystallized product	1.65	18.8

(1) Cf. K. Hess, H. Kiessig and J. Gundermann, *Z. physik. Chem.*, **49**, 64 (1941).

### Viscose Rayon

Original fibers	1.88	21.7
Ground product	1.98	30.0
Recrystallized product	1.70	20.0

A relative measure of amorphous substance is given by the sorption ratio, which is the ratio of sorptive capacity to that of native cotton at equal relative humidity, while density determinations have indicated that 40% of native cotton and ramie is amorphous.<sup>2</sup> Thus, from the average of the above sorption ratios the quantity of amorphous substance in the recrystallized powders is estimated at  $1.68 \times 40 = 67\%$ .

The heats of wetting of the amorphous and recrystallized samples differ by 10 cal./g. or 1.62 kcal./mole, which is a measure of the heat of crystallization of the crystalline portion in both wetted samples. Assuming that cellulose II and  $\beta$ -glucose have similar heats of crystallization (5.5 kcal./mole), the percentage of amorphous substance is calculated to be  $(5.5 - 1.62)/5.5 = 72\%$ .

The average value for the three methods is 67%. The percentage of amorphous substance in rayon was previously estimated from density to be 75-80%.<sup>3</sup> According to the sorption ratio of rayon given above, this figure should be  $(1.88:1.68) \times 67 = 75\%$ , providing additional confirmation.

The X-ray method seems the more general procedure for estimating the amorphous portion in cellulose. The work is being continued.

(2) P. H. Hermans, "Contribution to the Physics of Cellulose Fibres," Elsevier, Amsterdam or New York, 1946.

LABORATORY FOR CELLULOSE RESEARCH  
AKU AND AFFIL. COMPANIES P. H. HERMANS  
UTRECHT, NETHERLAND A. WEIDINGER

RECEIVED MAY 11, 1946

### THE STRUCTURE OF CHLORINATED CYCLOPROPANES<sup>1</sup>

Sir:

Publications by Spinrad<sup>2</sup> and Stevens<sup>3</sup> of discussion on the exterior valence angle of the cyclopropane ring, based on dipole moment measurements of *trans*-1,2-dichlorocyclopropane, prompt us to report some results obtained in an electron diffraction investigation of the structures of a series of chlorinated cyclic hydrocarbons which is under way in these Laboratories at the present time. We have completed the investigation of monochlorocyclopropane and of 1,1-dichlorocyclopropane, using samples furnished by Dr. J. D. Roberts. The cyclopropane ring was assumed to be equilateral, and the C-H bond distance was assumed to be 1.09 Å. Both compounds led to the

(1) Contribution No. 1066 from The Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena, California.

(2) B. I. Spinrad, *THIS JOURNAL*, **68**, 617 (1946).

(3) P. G. Stevens, *ibid.*, **68**, 620 (1946).