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

RECEIVED APRIL 30, 1946

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.[1] 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
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.[2] 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 β-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%.[2] 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.

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

[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[1]

Sir:

Publications by Spinrad[2] and Stevens[3] 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

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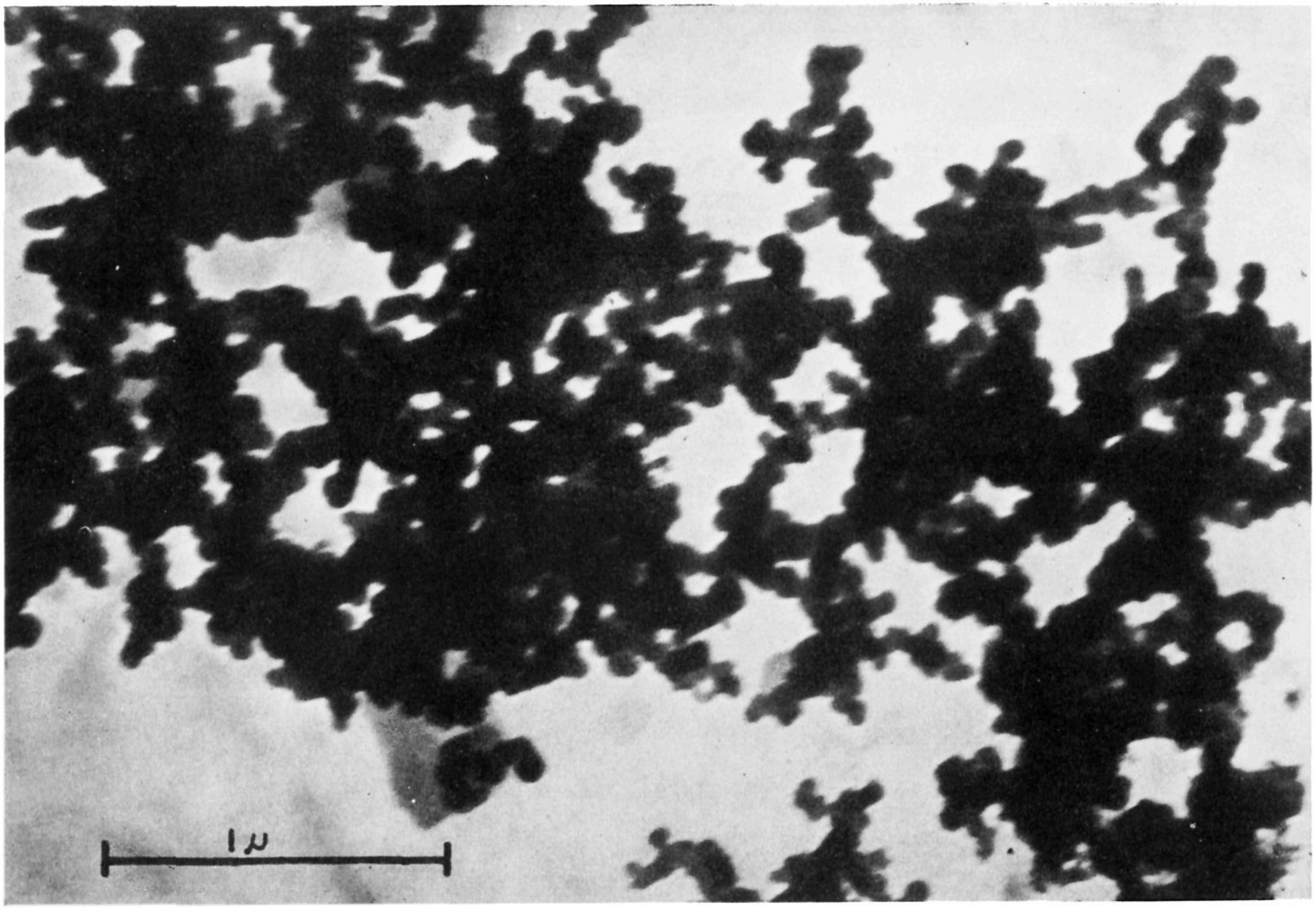
[1] Contribution No. 1066 from The Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena, California.

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

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



Swan soap section × 50,000.

following results: C–C, 1.52 ± 0.02 Å.; C–Cl, 1.76 ± 0.02 Å.; angle between the C–Cl bond and the plane of the ring, 56 ± 2°. There were no unusual difficulties in the determination, which made use of photographs showing features out to $q = 100$, and hence we feel that these results justify confidence.

It seems probable that the angle between the C–Cl bond and the plane of the ring in other chlorocyclopropanes, including *trans*-1,2-dichlorocyclopropane, is little different from 56° and that the value 48° found by Spinrad[2] is in error, due perhaps to the presence of impurities, a possibility mentioned by Spinrad[2] and Stevens,[3] or to inadequacies of the interpretation of the dipole moment data.

THE GATES AND CRELLIN LABORATORIES OF CHEMISTRY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA JOHN MICHAEL O'GORMAN
VERNER SCHOMAKER

RECEIVED MAY 1, 1946

THIN UNDISTORTED SECTIONS FOR ELECTRON MICROSCOPY

Sir:

A serious limitation on electron microscopy results from the fact that sections of only a few tenths of a micron in thickness must be obtained if they are to transmit electron beams. The invention of the high speed ultramicrotome[1] by O'Brien and McKinley in 1943 showed that under certain conditions such sections could be produced.

However, the opinion is still current that such sections may be so distorted that they are artifacts. For optical microscopy, sections are frequently exposed to great stresses and submitted to drastic treatments without losing their value. The high speed microtome should give better sections because the inertia of the material tends to conserve the structure as compared with the relatively slow dragging of an ordinary microtome.

Here the accompanying electron micrograph gives for the first time a conclusive demonstration that the high speed ultramicrotome can yield thin sections of a comparatively soft plastic body without smearing or distortion.

The specimen is from a commercial, floating, tallow coconut oil soap manufactured by drastic working in the neighborhood of 100° (water content about 20%) while air is incorporated, with subsequent cooling to room temperature during which phase changes induce further fine subdivision of the soap and of the air. It had been surmised from optical and ultraviolet microscopy that the texture was submicroscopic and possibly finely granular. Now it is apparent at a glance that this is indeed the case.

The "converter" soap is very clearly resolved into primary particles of soap about 1000 Å. in diameter, and these are seen to be loosely aggregated into an open mass enmeshing the air. No smearing of the soap is noticeable, nor any directional effects caused by the microtome except for a regular variation in thickness caused by unevenness of the microtome blade itself. Even the air spaces show no signs of having been distorted or crushed.

It is an important circumstance that these sections are caught directly upon 200-mesh wire gauze without coming into contact with any surface, and then directly examined.

Opportunity may be taken to record a suggestion made by one of us (J. W. M.) that the enclosed cell of McBain and Abrams[2] devised for liquids can to great advantage be used with such solid sections as are given by the high speed microtome. If the sections are caught directly in such a cell, which is vacuum tight and also impermeable to water vapor, while transparent to electrons, they may be preserved intact and examined without exposure to vacuum or desiccation, thus opening valuable new possibilities for the examination of solid colloids and of biological material.

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DEPARTMENT OF CHEMISTRY
STANFORD UNIVERSITY JAMES W. MCBAIN
STANFORD UNIV., CALIF.

RECEIVED APRIL 30, 1946

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[1] O'Brien and McKinley, *Science*, **98**, 455 (1943); see also the modifications by Ladd and Braendle, *Rubber Age*, **57**, 681 (1945) and Fullam and Gessler, *Rev. of Sci. Instruments*, **17**, 23 (1946).

[2] Abrams and McBain, *J. Appl. Physics*, **15**, 607 (1944); *Science*, **100**, 273 (1944).