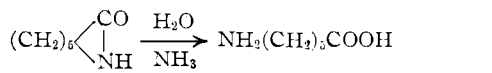
leads directly to the free and pure ϵ-aminocaproic acid in a single step. The conversion after five days at 80° was about 20%, but the unreacted ϵ-caprolactam can be simply and quantitatively recovered, so that the actual yield is close to the theoretical. Undoubtedly speedier and higher conversions can be obtained with higher temperatures and pressures but this was not investigated.



Experimental

ϵ-Aminocaproic Acid.—Eleven and three-tenths grams (0.1 mole) of ϵ-caprolactam[2] was heated in an oven with 55 ml. of concentrated ammonia in a pressure-bottle for five days at 80°. The contents of the bottle were then evaporated to dryness, treated with 30 ml. of *i*-propanol, the precipitated ϵ-aminocaproic acid filtered off, washed with *i*-propanol and dried: yield 2.6 g.; m. p. 201–202° (dec.). On distillation to dryness, the mother liquors yielded an oily residue which promptly crystallized; it had a m. p. of 65–68° and thus proved to be pure ϵ-caprolactam.

(2) ϵ-Caprolactam was generously supplied by E. I. du Pont de Nemours & Co., Inc.

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X-Ray Crystallographic Data for Sodium 1-Decanesulfonate Hemihydrate

BY L. H. JENSEN AND E. C. LINGAFELTER

X-Ray crystallographic data for the hemihydrates of sodium 1-dodecane-, 1-tetradecane-, 1-hexadecane- and 1-octadecanesulfonate have been reported.[1] The hemihydrate of sodium 1-decanesulfonate was now been obtained as extremely small single crystals suitable for X-ray diffraction studies.

Conditions for the growth of sodium 1-decanesulfonate hemihydrate differ from those for the hemihydrates previously reported. Sodium 1-decanesulfonate hemihydrate crystallized from a solution of approximate composition 15% water, 85% 1,4-dioxane at *ca* 5°. Work is in progress at the present time to delineate the temperature and solvent conditions for the formation of this and the other hydrates of the sodium 1-alkanesulfonates.

In addition to the habits observed for the other hemihydrates, sodium 1-decanesulfonate hemihydrate crystallizes as rhomb-shaped tablets, extremely thin parallel to {001} and outlined by (12 *l*).

X-Ray crystallographic data were obtained as previously reported.[1] Cell constants for the monoclinic unit cell are: $a_0 = 6.82$ kX; $b_0 = 15.36$ kX; $c_0 = 25.69$ kX; $\beta = 90° 45'$.

The X-ray value for a_0/b_0 is 0.4440. The goniometric value of a/b is 0.4402. The density calculated from the unit cell constants assuming four molecules of $2C_{10}H_{21}SO_3Na \cdot H_2O$ per unit cell is 1.261 g./cc. From the size of the unit cell and the intensity distribution of the diffraction effects, the structure of sodium 1-decanesulfonate hemihydrate seems identical with the other members of the hemihydrate series.

(1) Jensen and Lingafelter, THIS JOURNAL, **68**, 1729 (1946).

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Electron Microscopic Structure of Cellulose Powder from Wood Pulp Ground in Very Dry Condition*

BY P. H. HERMANS

In a previous paper[1] we have shown that on grinding of very dry cellulose fibers in a vibrating ball mill two characteristic types of disintegration product are observed, fibrillar ones still showing a crystalline nature on X-ray examination and an amorphous powder without clearly recognizable structure. The former disappears more and more on prolonged grinding and the latter apparently represents the final product, probably consisting of crumpled or otherwise deformed fibrillar elements admixed with porcelain powder from mill abrasion. The electron micrograph reproduced in Fig. 5 of the paper cited showed powder from ramie fibers containing about 20% of porcelain.

Using balls of a harder ceramic material it was possible to reduce the ash content of the grinding products considerably. Powder from woodpulp fibers ground five hours and containing less than 3% ash was used for the recrystallization experiments referred to in our previous paper. In the present note we shall briefly deal with a further electron microscopic study on the morphological structure of this product in the original and in the recrystallized state.

As stated before, the particle size of the powder largely depends on the method of previous treatment. In order to get the best possible dispersion for examination with the electron microscope, a suspension of the powder in water was exposed to ultrasonic radiation (200,000 cycles). Particles ranging between about 1–10 μ were so obtained.

Figure 1 shows a typical particle of the original powder so dispersed in water and then dried on the collodion film. Figures 3 and 4 show typical particles of the same powder which had previously been heated in water at 100° (recrystallized sample). There is no observable difference between the original and the recrystallized preparations. Both consist of rather compact aggregates whose actual structure is difficult to recognize. Since there is only 3% ash present, their substance must be almost entirely cellulose. It will be seen that there are some indications of a distorted and crumpled fibrillar structure. Figure 2 shows a

(1) P. H. Hermans and A. Weidinger, THIS JOURNAL, **68**, 2547 (1946).