CHARLES C. PRICE

1.5569). The alkylated aminoquinoline was very susceptible to air-oxidation, and it was necessary to preserve it under nitrogen in the cold.

Anal. Calcd. for  $C_{22}H_{77}N_2O$ : C, 74.34; H, 10.04. Found: C, 74.50; H, 10.14.

Attempts to prepare the dihydrochloride were unsuccessful, but it was found that the dihydrobromide could be prepared easily. To 10 g. of the free amine was added a solution of 10 ml. of 40% hydrobromic acid (sp. g. 1.38) in 100 ml. of absolute ethanol and 100 ml. of absolute ether. The salt separated as a fine, yellow powder, m. p. 192.5-193.5°, when the solution was cooled. The analytical sample was crystallized from hot absolute ethanol.

Anal. Calcd. for C23H39Br2N3O: C, 51.79; H, 7.34; Br, 29.97. Found: C, 51.71; H, 7.11; Br, 29.97.

NOVES CHEMICAL LABORATORY

UNIVERSITY OF ILLINOIS

HARRY F. HERBRANDSON URBANA, ILLINOIS

**RECEIVED FEBRUARY 4, 1946** 

# COMMUNICATIONS TO THE EDITOR

## FRACTIONATION OF CELLULOSE

Sir:

In the course of our work on the fractionation of cellulose by a refined procedure using cuprammonium solvent at low temperatures (below  $0^{\circ}$ ), we have observed phenomena similar to those recently reported by Morey and Tamblyn<sup>1</sup> for cellulose acetate and cellulose acetate butyrate which demonstrate the importance of the choice of solvent and precipitant in fractionation work.

With cellulose the choice of solvent is limited, but with cuprammonium, which is recognized as the best solvent, we have found the choice of precipitant to be important.

For example, when a 5% aqueous solution of sodium potassium tartrate (Rochelle salt) was used as the precipitant, precipitation of fractions took place on a weight basis, but the respective fractions did not show a significant amount of resolution on a molecular weight basis. However, by selecting other precipitants, varying degrees of resolution on a molecular weight basis became possible, as illustrated in Table I.

#### TABLE I

## FRACTIONATION OF CELLULOSE

Experimental rayon tire yarn; 100% wood pulp base; av. basic D. P. = 490.

	Sodium potassium tartrate soln.		Acetone		n-Propyl alcohol	
Frac- tion	Recov.	Basic D. P.	Recov.	Basic D. P.	Recov.	Basic D, P.
1	19.7	480	46.6	535	18.8	615
<b>2</b>	19.3	478	13.2	392	20.2	465
3	18.3	473	8.5	314	20.8	316
4	17.5	470	18.4	250	16.6	272
5	13.4	470	8.1	142	9.7	247
6	13.7	478	••		6.1	132

The average molecular chain length values of the fractions are expressed as Basic D. P.'s.<sup>2</sup>

The same general phenomena have been observed for the fractionation of viscose rayons, and

(1) D. R. Morey and J. W. Tamblyn, J. Phys. Chem., 50, 12-22 (1946). (2) O. A. Battista, Ind. Eng. Chem., Anal. Ed., 16, 351-354 (1944).

viscose rayon pulps, respectively, from cuprammonium solvent.

Further work is in progress in which additional precipitants possessing wide variations in molecular structure are being used with the hope that resolving power can be correlated with molecular structure.

CHEMICAL RESEARCH DEPARTMENT

AMERICAN VISCOSE CORPORATION O. A. BATTISTA WAYNE A. SISSON MARCUS HOOK, PENNSYLVANIA RECEIVED APRIL 20, 1946

## A REACTION VELOCITY WITH LARGE NEGATIVE TEMPERATURE COEFFICIENT

Sir:

Iodine shows very large departures from Raoult's law in a number of solvents, and it occurred to me to use iodine solutions to decide between activity and concentration as the determinant for reaction velocity. I chose the reaction between iodine and chlorine to form ICl, in CCl<sub>4</sub> to begin with, as one likely to meet the specifications. As so often happens in scientific research, it is not answering the original question, but has yielded results of such interest as to seem worth this brief preliminary report. They are as follows:

(1) The reaction is accelerated by light, but this is neither surprising nor disturbing, since one can let it proceed in the dark.

(2) It is enormously sensitive to minute traces of water. Although the solubility of water in CCl<sub>4</sub> is extremely small, the reaction runs to completion in minutes if the solutions are not carefully dried, whereas hours are required if the solutions are mixed after being kept sealed for a day in contact with  $P_2O_5$ .

(3) The "wet" reaction appears to be bimolecular, but the "dry" reaction is monomolecular with respect to chlorine through a wide range of iodine concentrations.

(4) And this is the truly astonishing feature, the wet reaction not only proceeds several times