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VISCOSITY OF POLYTRIFLUOROCHLOROETHYLENE IN o-CHLOROBENZOTRIFLUORIDE

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Viscosity data for polytrifluorochloro-ethylene is a chlorofluorobutane solvent have been presented by H. S. Kaufman and M. S. Muthana.[1] We present here the results of measurements in the solvent *o*-Chlorobenzo-trifluoride at a temperature of 137.10°. The measurements were made with a Bingham-type viscometer[2] in which flow times are measured as a function of driving pressure.

Experimental

The silicone oil constant temperature bath was electronically controlled at a temperature of $137.10 \pm 0.02°$. The viscometer was patterned after the one shown in ref. 2. With these modifications: (1) large stopcocks were placed in the arms approximately 10 cm. Above A and M of the figure; and (2) a side arm containing a fritted glass disc and a 15-cc. Bulb in which polymer and solvent were placed for dissolution was attached just above M. A weighed amount of polymer and 10cc. (volume at 20°) of solvent were introduced into this bulb, a cap placed on the opening to the bulb and the stopcocks closed to keep solvent vapor from escaping. The solvent and polymer were left in the bulb until solution was complete. After solution, nitrogen gas under pressure was used to drive the solution through the fritted glass into the viscometer proper. Excess solution was withdrawn from the viscometer through a small stainless steel tube whose tip reached to point A in the left-hand arm. The solution was kept under an atmosphere of nitrogen at all times to prevent oxidation of the solvent.

A water manometer was used to determine driving pressure. Time of flow data as a function of driving pressure were obtained in the manner indicated in Appendix A of ref. 2. The viscometer was calibrated with water and sucrose solutions according to the methods outlined by Hall and Fuoss.[3] The volume of the dry bulb of the viscometer was 2.13cc., the capillary radius was 0.015 cm., and the length of the capillary was approximately 10 cm.

Pressures were varied between 50 and 150 cm. of water in six approximately equal steps and the corresponding flow times recorded. In ref. 3 it is shown that the behavior of a solution in a Bingham-type viscometer can be expressed by the relationship

$$1/pt = A_0/h_¥ + (k - l)\, p \qquad (1)$$

in which p is the driving pressure, t is the flow time, A_0 is a constant depending only on viscometer dimensions, $h_¥$ is the viscosity of the solution at zero driving pressure (infinite time), and k and l are functions of viscometer dimensions and the nature of the solution. For the viscometer used A_0 was 9.57×10^{-7} poise-cm.^{2}/g. sec.

The polytrifluorochloroethylene used was made by the Kellogg Corporation. The three molecular weight ranges used were designated Kel-F 240, lot no. 124; Kel-F 270, lot no. 126 and Kel-F 300, lot no. 162-L.

Results and Discussion

Plots of $1/pt$ *vs. p* were constructed and the viscosities at zero rate of shear for various concentrations of the polymer obtained from the intercepts. From the viscosities the reduced viscosities were calculated and plotted in the usual fashion as a function of concentration. The plots are shown in Fig. 1. The viscosity of the solvent *o*-Chlorobenzotrifluoride at 137.10° is 0.3595 centipoise. The density is 1.20.

Molecular weights of 56,600, 76,000 and 100,000 are given for Kel-F 240, 270 and 300, respectively, by Kaufman and Muthana. The relationship between intrinsic viscosity [*h*] (obtained from the intercepts in Fig. 1) and the

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[1] Paper No. 26, Division of High Polymer Chemistry, American Chemical Society Meeting, September 5, 1950.

[2] Eugene C. Bingham, "Fluidity and Plasticity," McGraw-Hill Book Co., Inc., New York, N. Y., Fig. 29, p. 76.

[3] H. T. Hall and R. M. Fouss, This Journal, 73, 265 (1951).

molecular weight M is expressed by the equation[4]

$$[h] = KM^a \qquad (2)$$

in which K is a constant and α is characteristic of the polymer and usually has a value between 0.3
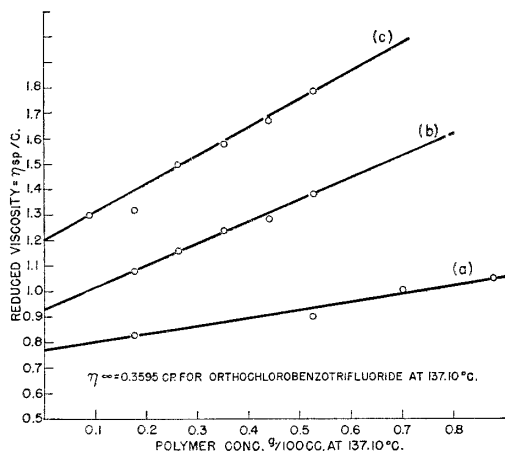

Fig. 1.—Reduced viscosity as a function of concentration for (a) Kel-F 240, (b) Kel-F 270 and (c) Kel-F 300 in ortho-chlorobenzotrifluoride at 137.10°.

and 1.2. Substituting the molecular weight values of Kaufman and Muthana into (2) and plotting log $[h]$ *vs.* log M gives values of 1.71 X 10^{-4} and 0.77 for K and a, respectively, when the concentration is expressed as grams of polymer per 100 cc. of solvent. The value a is somewhat of an indication of chain stiffness. The value obtained here is midway between that for a flexible chain polymer such as a polyester and that of a stiff chain polymer such as a cellulose derivative. This is consistent with what might be expected when a Hirschfelder model of the polymer is examined. The model shows that some restriction to rotation about C-C bonds should be expected.

It was not possible to evaluate shear constants for this polymer because of a reduction in the precision resulting from difficulties introduced by the high temperature.

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[4] H. Mark, "Die feste Körper," Verlag S. Hirzel, Leipzig, 1948, p. 103.