seal broken. The contents of the tubes were dissolved in methyl ethyl ketone containing hydroquinone, and this solution was slowly added with stirring to a 3 times excess of methanol. The precipitated polymer was filtered, washed copiously with methanol and allowed to dry to constant weight in a vacuum oven at 60°.

Chlorine Analysis.—The per cent. chlorine was determined using the Parr bomb.

Results and Discussion

The experimental data are summarized in Table I. The copolymerization rate constants were calculated according to the method of Alfrey and Goldfinger[1] in the usual manner.

TABLE I

DETERMINATION OF r_1 AND r_2

THE COPOLYMERIZATION RATE CONSTANTS SYSTEM 2,5-DICHLOROSTYRENE–METHYL ACRYLATE

Monomer composition				Polymer composition (Exp.)	
Mole fraction 2,5-dichlorostyrene	Mole fraction methyl acrylate	Yield, %	Chlorine, %	Mole fraction 2,5-dichlorostyrene	Mole fraction methyl acrylate
0.90	0.10	4.4	39.95	0.950	0.050
.75	.25	3.0	39.15	.913	.087
.50	.50	3.7	37.20	.829	.171
.25	.75	4.4	32.15	.643	.357
.10	.90	4.9	22.30	.371	.629
.05	.95	4.3	14.15	.206	.794

The smooth curve in Fig. 1 represents the theoretical curve calculated from the values $\alpha = 0.25$ and $\beta = 0.15$. These correspond to the values, according to the latest nomenclature,[2] $r_1 = 4$ and $r_2 = 0.15$, respectively. The encircled points are experimentally determined from the data in Table I.

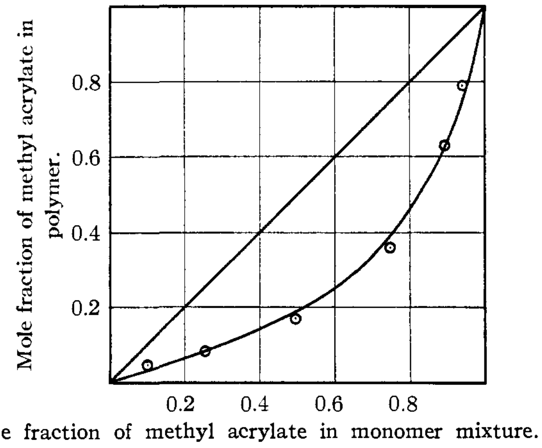

Fig. 1.—Copolymerization composition curve.

The value $r_1 = 4$ indicates that the 2,5-dichlorostyrene monomer adds to the 2,5-dichlorostyrene free radical four times as fast as the methyl acrylate monomer adds. The value $r_2 = 0.15$ indicates that the methyl acrylate monomer adds to the methyl acrylate free radical 0.15 as fast as the 2,5 dichlorostyrene monomer adds to the methyl acrylate free radical.

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(1) T. Alfrey and G. Goldfinger, *J. Chem. Phys.*, **12**, 205 (1944).

(2) Alfrey, Mayo and Wall, *J. Polymer Sci.*, **1**, 581 (1946).

The Preparation of 2,3,5-Triphenyltetrazolium Chloride

BY A. M. MATTSON, C. O. JENSEN AND R. A. DUTCHER

Synthesis of 2,3,5-triphenyltetrazolium chloride by the method of Pechman and Runge as modified by Bamberger and Billeter and by Kuhn and Jerchel[1] resulted in poor yields of preparations which had a greater chloride content than the desired monobasic compound. A better yield of the compound with the theoretical chloride content and a saving of time were achieved by modifying the previous procedures.

Experimental

Triphenylformazan.—Twenty-one and two-tenths grams (0.2 mole) of freshly distilled benzaldehyde was dissolved in 125 ml. of methanol. To this solution 21.6 g. (0.2 mole) of phenylhydrazine was added during mechanical agitation. The hydrazone, dissolved in one liter of methanol, was added to a solution of 50 g. of sodium hydroxide and 70 g. of sodium acetate in one liter of methanol. To this solution, cooled to 20°, benzenediazonium chloride prepared from 18.6 g. of aniline (0.2 mole), 50 ml. of concentrated hydrochloric acid, 50 ml. of water and 14–15 g. of sodium nitrite, was added slowly during agitation. Formazan was precipitated as small red crystals. A yield of 15.8 g. (23%) of triphenylformazan was obtained, m. p. 170°.

2,3,5-Triphenyltetrazolium Chloride.—Fifteen grams (0.05 mole) of triphenylformazan were dissolved in 100 ml. of chloroform and the solution was cooled to 20°. Lead tetraacetate (30 g.) was added until the red color disappeared. The chloroform was evaporated and the residue taken up in water. Hydrochloric acid was added and the lead chloride was removed by filtration. The monobasic triphenyltetrazolium chloride was removed from the filtrate by three successive extractions with chloroform (water–chloroform ratio of 3:1), leaving the more acid salt in the water. The chloroform solution was concentrated on the steam-bath. Addition of ether to this solution precipitated the tetrazolium salt in long, silky needles. Nine and seven-tenths grams (57.7% based on formazan) of 2,4,5-triphenyltetrazolium chloride was obtained, m. p. 245° (d.) (Pechman and Runge 243°).

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(1) Pechman and Runge, *Ber.*, **27**, 2920 (1894); Bamberger and Billeter, *Helv. Chim. Acta*, **14**, 232 (1931); Kuhn and Jerchel, *Ber.*, **74B**, 941 (1941).

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Effects of Inhibitors on the Polymerization of Styrene[1]

BY FRANK R. MAYO AND R. A. GREGG

Inhibitors of free radical polymerization are regarded as materials which, by transfer or addition, are converted to radicals so stable they do

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(1) This paper was presented before the Division of Organic Chemistry at the New York City Meeting of the American Chemical Society, September 15, 1947.