

(4) The absorption kinetics of metal ions were studied in relation to the pH of the medium. It is pointed out that the rate of metal ion recovery and film absorption depend directly on the pH.

(5) A study was made of the desorption kinetics of copper ions from the film. It was shown that the rate of desorption of copper ions from the film is higher than the rate of absorption of copper ions by the film.

(6) Electron microscopic investigations of the structure of the copolymer obtained were carried out. It was indicated that in strongly acid and strongly alkaline media the copolymer macromolecules have a fibrillar structure and in the pH range of 3–8 a globular structure.

(7) Optical microscopy in an amorphous copolymer revealed large oriented structures exhibiting optical anisotropy.

Translated by E. SEMERE

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COPOLYMERIZATION OF SOME *p*-SUBSTITUTED STYRENE AND α -METHYLSTYRENE DERIVATIVES WITH STYRENE AND METHYLMETHACRYLATE*†

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(Received 5 April 1967)

QUANTITATIVE considerations concerning the effect of substituents on the reactivity of organic compounds, as is well known, were developed in greatest detail for di-substituted benzene derivatives, and in particular substituted benzoic acids and their derivatives, since the most detailed and accurate physicochemical parameters were available for these substances.

* *Vysokomol. soyed.* **A10**: No. 2, 438–445, 1968.

† 2nd Paper from the series "Relation of relative activities during radiation copolymerization with parameters of the Hammett equation".

For the same reasons substituted styrene derivatives can be conveniently used for the quantitative evaluation of the effect of substituents on the activity of vinyl monomers in homo- and copolymerization. Several attempts have so far been made to correlate with the Hammett σ -constants the rate constant of growth during radical homopolymerization of some *p*-substituted styrenes [1, 2], the relative activity constant of substituted styrenes during copolymerization with styrene [2-4] and methylmethacrylate [3], and the relative activity constants of *p*-substituted derivatives of α -methylstyrene during copolymerization with maleic anhydride [5].

When plotting these correlations Walling [3, 5] and Imoto [1, 2] used a set of σ -constants for the Hammett substituents [6], and Bagdasar'yan [7] used a set of Taft σ -constants [8].

New equations were derived in our former study [9], which relate both copolymerization constants (r_1 and r_2) with the σ -constants of substituents, thus providing additional information from copolymerization data. In view of the fact that when considering substituted styrenes the vinyl group (reaction centre), which enters into direct conjugation with the *para*-substituents of both the donor and acceptor type, is fixed, we thought it advisable to use for the correlations a set of electrophilic constants (σ^+) for electron-donor and nucleophilic (σ^-) constants for electron-acceptor substituents (see for example [10]). The use of σ^{+-} makes it possible to obtain a clearer correlation pattern, a slighter scatter of points in relation to the regression lines.

Detailed experimental data are given in this report of the copolymerization of some *para*-substituted derivatives of styrene and α -methylstyrene (substituents: COCH_3 , COOH and $\text{C}(\text{CH}_3)=\text{NOH}$) with styrene and methylmethacrylate. Preliminary data on the copolymerization of *p*-vinylacetophenone and *p*-vinylbenzoic acid with styrene and methylmethacrylate were reported previously [9, 11].

EXPERIMENTAL

Copolymerization of p-vinylacetophenone (M_2) in binary systems with styrene (M_1) and methylmethacrylate (M_1). *p*-Vinylacetophenone was obtained by Kenyon's method [12, 13], purified by distillation and repeated recrystallization from hexane; m.p. 36-37°, d_4^{20} 1.0095; n_D^{40} 1.5681. Styrene and methylmethacrylate, freed from inhibitor and distilled, had refractive indices n_D^{20} 1.5468 and 1.4135, respectively.

Copolymerization in this and subsequent cases was carried out in sealed ampoules without air and light at 60°. Azobisisobutyronitrile (0.05-0.1 mole-%) was used as initiator. After attaining a certain conversion, *p*-vinylacetophenone-styrene copolymer specimens were isolated by dilution of the reaction mixture with benzene, or methylethylketone (MEK) (according to copolymer composition), followed by decantation into a large amount of methanol or hexane. Specimens of *p*-vinylacetophenone-methylmethacrylate copolymers were isolated by dilution of the contents of the ampoules with a benzene-methylethylketone mixture, followed by precipitation with the excess methanol.

After three re-precipitations the copolymers were dried *in vacuo* at 45° to constant weight. Copolymer composition was determined by elementary analysis. Results of copolymerization are shown in Table 1.

TABLE 1. COPOLYMERIZATION OF *p*-VINYLACETOPHENONE (M_2)

Composition of the initial mixture, mol. fractions		Degree of polymerization, %	Carbon content of the copolymer, %	Composition of the reaction mixture for a given degree of polymerization, mol. fractions		[η] of the copolymer (MEK, 25°), dl/g
M ₁ ⁰	M ₂ ⁰			M ₁	M ₂	
With styrene (M ₁)						
0.9487	0.0513	19.70	90.37	0.7867	0.0247	—
0.8478	0.1522	19.00	88.53	0.7220	0.0997	—
0.8059	0.1941	20.80	88.01	0.6766	0.1272	0.531
0.5770	0.4230	16.60	85.51	0.5150	0.3308	—
0.5559	0.4441	13.90	85.50	0.5014	0.3654	—
With methylmethacrylate (M ₁)						
0.9407	0.0593	18.91	63.90	0.7805	0.0358	—
0.8682	0.1318	21.00	67.73	0.7231	0.0782	0.670
0.6590	0.3410	15.24	73.35	0.5862	0.2654	0.568

Copolymerization of *p*-vinylbenzoic acid (M_2) in binary systems with styrene (M_1) and methylmethacrylate (M_1). *p*-Vinylbenzoic acid is obtained by the Merrill [13] method and is recrystallized from alcohol and ether; m.p. 140°.

Copolymerization was carried out in a solution in ethyl alcohol (or in an alcohol-benzene mixture). During the separation of copolymers solvent-precipitant combinations were varied according to the number of *p*-vinylbenzoic acid units in the copolymer.

With up to ~25 mole-% *p*-vinylbenzoic acid units in the copolymers, to separate the copolymer a combination of benzene, or MEK (solvent)-methanol (precipitant) was used. With a higher number of *p*-vinylbenzoic acid units satisfactory precipitation of copolymers could only be achieved by the following methods. The contents of the ampoule were diluted with ethyl alcohol and decanted into an excess of benzene or petroleum ether. The liquid phase was then rapidly decanted and the transparent copolymer stuck to the vessel walls treated with diethyl ether. Copolymer composition was determined by elementary analysis and potentiometric titration of carboxyl groups (copolymer batches were dissolved in dioxane and titrated with 0.01 N NaOH). Results of copolymerization are shown in Table 2.

Copolymerization of *p*-vinylacetophenoneoxime (M_2) in binary systems with styrene (M_1) and methylmethacrylate (M_1). *p*-Vinylacetophenoneoxime was obtained by oximation of *p*-vinylacetophenone [14] and purified by recrystallization from ethyl alcohol; m.p. 115–116°. Copolymerization was carried out in solution in ethyl alcohol, or in an alcohol-benzene mixture. *p*-Vinylacetophenoneoxime-styrene copolymers were re-precipitated from benzene (MEK) with methanol and *p*-vinylacetophenoneoxime-methylmethacrylate copolymers from MEK with petroleum ether. Copolymer composition was found by determination of carbon and nitrogen. Results of copolymerization are shown in Table 3.

Copolymerization of *p*-isopropenylacetophenone (M_2) with styrene (M_1). *p*-Isopropenylacetophenone was obtained by the method previously described, by acetylation of poly- α -methylstyrene, by the Friedel-Crafts method, followed by depolymerization [15]. *p*-Isopropenylacetophenone was purified by recrystallization from hexane, or aqueous ethyl alcohol; m.p. 52.6–53°, d_4^{25} 0.9935, n_D^{25} 1.5547. Copolymerization, separation of copolymers and determination of composition were effected by the same method as that used for copolymerization of *p*-vinylacetophenone. Results of copolymerization are shown in Table 4.

TABLE 2. COPOLYMERIZATION OF *p*-VINYLBenzoic Acid (M_2)

Composition of the initial mixture, mol. fractions		Degree of polymerization, %	Carbon content of the copolymer, %	Content of <i>p</i> -vinylbenzoic acid units in the copolymer (potentiometric titration), %	Composition of the reaction mixture for a given degree of polymerization, mol. fractions		[η] of the copolymer (MEK), 25°, dl/g
M ₁ ^o	M ₂ ^o				M ₁	M ₂	
With styrene (M ₁)							
0.9548	0.0452	29.18	89.56	14.00	0.6986	0.0160	—
0.9327	0.0673	25.75	88.41	19.74	0.7199	0.0305	0.783
0.7926	0.2074	37.80	83.70	42.45	0.5557	0.0845	0.440
0.4009	0.5991	25.29	77.83	74.49	0.3198	0.4330	—
0.3949	0.6051	28.91	77.73	74.75	0.3031	0.4141	—
With methylmethacrylate (M ₁)							
0.8408	0.1592	21.84	64.34	35.41	0.6886	0.1028	—
0.8344	0.1656	29.96	63.46	34.00	0.6205	0.0911	0.65
0.5683	0.4317	53.23	67.99	62.27	0.3256	0.1610	—
0.5846	0.4154	53.21	67.62	59.27	0.3241	0.1593	—

TABLE 3. COPOLYMERIZATION OF *p*-VINYLAcetophenoneoxime (M_2)

Composition of the initial mixture, mol. fractions		Degree of polymerization, %	Elementary composition of the copolymer, %		Composition of the reaction mixture for a given degree of polymerization, mol. fractions		[η] of the copolymer (MEK, 25°), dl/g
M ₁ [°]	M ₂ [°]		C	N*	M ₁	M ₂	
With styrene (M ₁)							
0.9533	0.0467	42.65	90.56	1.00	0.5623	0.0169	0.335
0.9092	0.0908	39.50	89.13	1.58	0.5685	0.0426	—
0.8125	0.1875	35.97	85.95	3.04	0.5557	0.0973	0.630
0.6662	0.3338	55.80	83.45	4.24	0.3304	0.1228	0.438
0.5219	0.4781	39.74	81.14	5.58	0.3381	0.2725	0.580
0.2300	0.7700	38.63	77.25	7.55	0.1517	0.4657	0.467
With methylmethacrylate (M ₁)							
0.8577	0.1423	31.33	64.48	2.70	0.6228	0.0767	0.920
0.8593	0.1407	56.98	63.82	2.30	0.4035	0.0390	—
0.7617	0.2383	13.40	66.12	3.68	0.6744	0.1986	0.733
0.7476	0.2524	26.55	66.77	4.07	0.5933	0.1682	—
0.4520	0.5480	58.62	69.54	5.73	0.1697	0.2080	—
0.4571	0.5429	45.74	69.35	5.60	0.2287	0.2859	—
0.2271	0.7729	35.43	72.36	7.35	0.1396	0.4681	0.792

* Determined by the Kjeldahl method.

TABLE 4. COPOLYMERIZATION OF *p*-ISOPROPENYLACETOPHENONE (M_2) WITH STYRENE (M_1)

Composition of the initial mixture, mol. fractions		Degree of polymerization, %	Carbon content in the copolymer, %	Composition of the reaction mixture at a given degree of polymerization, mol. fractions		[η] of the copolymer (MEK, 25°), dl/g
M_1°	M_2°			M_1	M_2	
0.8900	0.1100	29.38	89.54	0.6707	0.0551	—
0.7545	0.2055	25.19	88.19	0.6347	0.1318	0.305
0.5422	0.4578	31.86	85.92	0.4019	0.2907	—
0.5353	0.4647	19.39	85.88	0.4498	0.3610	—

Copolymerization of *p*-isopropenylacetophenoneoxime (M_2) with styrene (M_1). *p*-Isopropenylacetophenoneoxime was obtained by oximation of *p*-isopropenylacetophenone [14] and purified by recrystallization from ethyl alcohol, m.p. 139.5–140.5°. Copolymerization, isolation of copolymers and the determination of composition were effected as in the above cases of copolymerization of *p*-vinylacetophenoneoxime. Results of copolymerization are shown in Table 5.

TABLE 5. COPOLYMERIZATION OF *p*-ISOPROPENYLACETOPHENONEOXIME (M_2) WITH STYRENE (M_1)

Composition of the initial mixture, mol. fractions		Degree of polymerization, %	Elementary composition of the copolymer, %		Composition of the reaction mixture at a given degree of polymerization, mol. fractions		[η] of the copolymer (MEK, 25°), dl/g
M_1°	M_2°		C	N*	M_1	M_2	
0.9475	0.0525	48.68	90.50	0.94	0.4988	0.0192	0.960
0.8969	0.0031	32.71	88.99	1.79	0.6222	0.0585	—
0.8270	0.1730	34.33	86.98	2.53	0.5632	0.1011	0.322
0.7034	0.2966	33.62	84.34	3.65	0.4857	0.1850	0.216
0.5000	0.5000	13.17	81.78	4.97	0.4330	0.4348	0.170

* Determined by the Kjeldahl method.

Copolymerization constants r_1 and r_2 of styrene and α -methylstyrene derivatives were determined by the Mayo and Lewis method of intercepts [16] and by the Shtraikhman analytical method [17, 18] and the Alfrey-Price parameters, Q and e , calculated. The results are given in Table 6.

DISCUSSION OF RESULTS

It was pointed out in a former report [9] that there is a linear correlation between the relative activity constants for substituted styrenes (M_2) in copolymerization with styrene (M_1) and methylmethacrylate (M_1).

TABLE 6. COPOLYMERIZATION CONSTANTS r_1 AND r_2 OF p -SUBSTITUTED DERIVATIVES OF STYRENE (M_2) AND α -METHYLSTYRENE (M_2) IN BINARY SYSTEMS WITH STYRENE (M_1) AND METHYLMETHACRYLATE (M_1)

Monomer	Substituent	Styrene M_1				Methylmethacrylate M_1			
		r_1	r_2	Q_2	e_2	r_1'	r_2'	Q_2	e_2
p -Substituted styrene (M_2)	COCH ₃	0.25	1.15	1.64	0.32	0.32	1.37	1.61	-0.51
	COOH	0.28	1.04	1.47	0.31	0.40	1.20	1.31	-0.46
	C=NOH	0.54	1.04	1.01	0.04	0.50	0.98	1.06	-0.45
	 CH ₃								
p -Substituted α -methylstyrene (M_2)	COCH ₃	0.37	0.95	1.20	0.22	—	—	—	—
	C=NOH	0.64	0.91	0.96	0.06	—	—	—	—
	 CH ₃								

The correlation takes the form of the equation

$$\log r_{2(H)} \frac{r_2(HY)}{r_2(Y)} = \log \frac{K_{XY}}{K_{HY}} - \log \frac{K_{XH}}{K_{HH}} = (\rho_Y - \rho_H) \sigma, \quad (1)$$

where $r_{2(H)}$ and $r_{2(Y)}$ are the relative activity constants of monomers (M_2) having different substituents (X) in copolymerization with standard monomers (H) (M_1) (e.g. with styrene) and (Y) (M_1) (e.g. with methylmethacrylate), respectively; $r_{2(HY)}$ is the relative activity constant of the standard monomer (H) (M_2) (for example, styrene) in copolymerization with a standard monomer (Y) (M_1) (e.g. methylmethacrylate); ρ_Y and ρ_H are the reaction constants of addition of radicals to monomers (Y) and (H), respectively; σ —substitution constant.

This relation can now be statistically analysed for the case of copolymerization of substituted styrenes with styrene and methylmethacrylate, using literature data [19] and experimental data of this paper. The correlation coefficient (r), standard deviation (s) and the value of $(\rho_Y - \rho_H)$, calculated by the method of least squares (see [20]) are: 0.961; 0.07 and -0.322, respectively. Figure 1 shows relation (1) graphically.

For *para*-substituents, owing to the above reasons, the large constants (σ^{+-}) were used, and for *meta*-substituents the Taft σ^0 -constants, where there was no direct polar conjugation of substituents with the reaction centre.

Thus, a study of the copolymerization of monomers with different substituents (substituted styrenes) in binary systems with two standard monomers (e.g. styrene and methylmethacrylate) indicates that both copolymerization constants (r_1 and r_2) are related to the σ -constants of the substituents. The existence of this correlation, in addition to those already known [3, 9], opens up the possibility for the approximate evaluation of the relative activity con-

stants of monomers (r_1 and r_2) (in this case substituted styrenes) from known σ -constants of substituents. These correlations can, apparently, also be used for solving the inverse problem: from the known copolymerization constants of substituted monomers the unknown σ -constants of substituents can be approx-

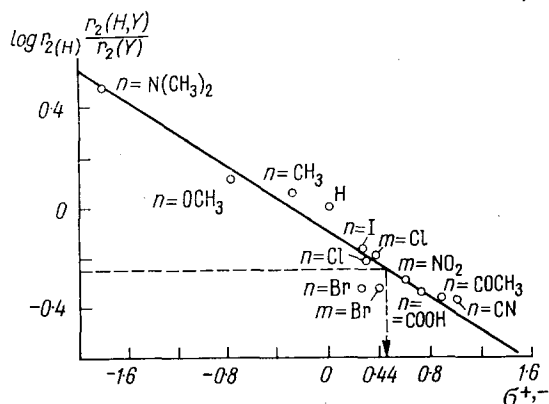


FIG. 1. Dependence of constants of substituents ($\sigma^{+,-}$) of $\log \frac{r_2(H,Y)}{r_2(Y)}$ for the copolymerization of substituted styrenes (M_2) with styrene (M_1) and methymethacrylate (M_1) (= should be corrected to - in Figs. 1 and 2)

imately estimated. The nucleophilic constant (σ^-) for the p - $C(CH_3)=N-OH$ group can be estimated by this method using data for the copolymerization of p -vinylacetophenoneoxime in binary systems with styrene and methymethacrylate ($\sigma_{C(CH_3)=NOH}=0.44$; see Figs. 1 and 2).

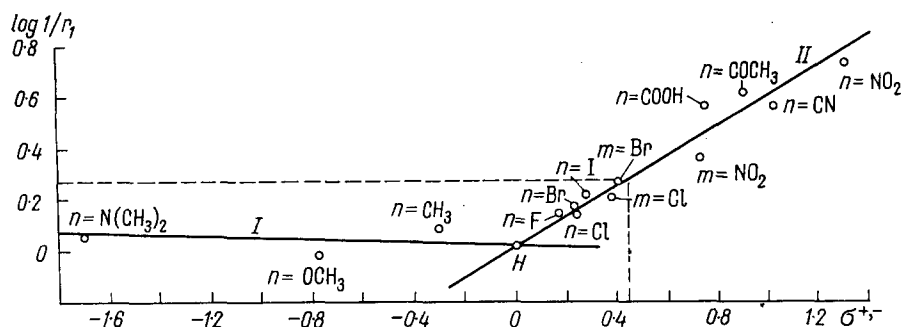


FIG. 2. Dependence of $\log 1/r_1$ for the copolymerization of substituted styrenes (M_2) with styrene (M_1) on constants of substituents ($\sigma^{+,-}$). The reaction constant (ρ_{II}) and the correlation coefficient (r_{II}) are 0.575 and 0.968, respectively.

Lowry [21] and Ham [22] noted that the behaviour of α -methylstyrene in copolymerization cannot be described by a conventional equation of copolymer composition [16], based on the assumption of four stages of chain growth in binary copolymerization. This does not allow for the fact that when three or more

α -methylstyrene molecules are sequentially joined in the polymer chain, the end is depolymerized. According to this theory, the maximum number of sequentially joined α -methylstyrene units in the chain is two [19]. However, the equations derived, which take into account the depolymerization factor of the monomer, according to the author [21], are complicated and hardly suitable for practical calculations.

It may be pointed out that the probability of the formation of units consisting of three or more links of the α -methylstyrene component (M_2) is slight in the composition range of 0–50 mole-% α -methylstyrene monomer in the initial mixture (during copolymerization with styrene (M_1) or methylmethacrylate (M_2)).

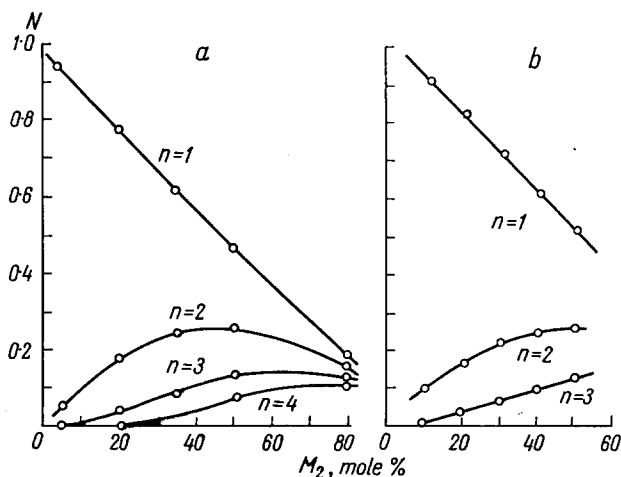


FIG. 3. Numerical functions of the longitudinal distribution of a unit consisting of p -links of p -vinylacetophenone in the copolymer, according to the composition of the initial monomer mixture for a styrene (M_1)– p -vinylacetophenone (M_2) system (a) and p -isopropenylacetophenone in a copolymer, in accordance with the composition of the initial mixture for the styrene (M_1)– p -isopropenylacetophenone (M_2) (b) system.

Figure 3a shows numerical functions of the dimensional distribution of a unit consisting of p -links of p -vinylacetophenone (M_2) in the copolymer for any initial mixture composition. The probability was determined by the formula [23]:

$$N = P_{22}^{(n-1)} (1 - P_{22}) . \quad (2)$$

It can be seen that in the range indicated the probability N of the formation of units with n of three and more, is slight. Consequently, in the case of a less active homologue, p -isopropenylacetophenone (M_2) (see Table 6) the probability of formation of these units is even less (Fig. 3b). Therefore for the copolymerization of derivatives of α -methylstyrene (M_2) with styrene (M_1) and methylmethacrylate (M_1) in the range of 0 to 50 mole % α -methylstyrene content, in the initial mixture the depolymerization factor of the monomer M_2 has not been taken into account. Copolymerization constants were calculated from a conventional equation of

copolymer composition and parameters Q and e were calculated according to Alfrey and Price (Table 6). A comparison of data on the copolymerization of styrene and α -methylstyrene derivatives, having the same substituents, indicates that α -methylstyrene monomers are of somewhat lower activity.

CONCLUSIONS

(1) A study was made of radical copolymerization of some styrene and α -methylstyrene derivatives (substituents: p -COCH₃, p -COOH and p -C(CH₃)=N—OH) in binary systems with styrene and methylmethacrylate, the copolymerization constants (r_1 and r_2) were determined and the parameters Q and e calculated according to the method used by Alfrey and Price.

(2) A statistical analysis was made of the correlation between the relative activity constant (r_2) and the σ -constants of the substituents for the copolymerization of substituted styrenes (M_2) with styrene (M_1) and methylmethacrylate (M_1).

(3) It was proved that the dependence of the copolymerization constant on the σ -constant of the substituents can be used in the approximate evaluation of unknown copolymerization constants from known σ -constants and vice versa.

(4) It was shown that in a certain composition range of the initial mixture the conventional equation of copolymer composition can be used to determine the relative activity constants (r_1 and r_2) in copolymerization of α -methylstyrene derivatives, not allowing for the depolymerization of the monomer.

Translated by E. SEMERE

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