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## Dilute Solution Properties and Chain Dimensions of Poly(2-alkoxyethyl methacrylates)

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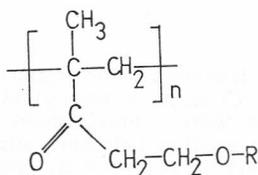
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The solution properties of poly(2-methoxyethyl methacrylate) (PMEMA), poly(2-ethoxyethyl methacrylate) (PEEMA) and poly(2-*n*-butoxyethyl methacrylate) (PBEMA) have been compared. The respective  $\Theta$ -temperatures in *n*-butanol are 64, 38 and 17 °C. From the relationships between intrinsic viscosity and molecular weight in a thermodynamically good solvent, methyl ethyl ketone (MEK), the respective characteristic ratios  $C_\infty = 11.1, 12.6,$  and 15.5 were estimated. The unperturbed dimensions of PBEMA depend on the nature of the solvent, being lower in *n*-butanol (12.3) than in MEK. The conformational properties of poly(2-alkoxyethyl methacrylates) resemble those of poly(*n*-alkyl methacrylates) with a comparable side-chain length.

### INTRODUCTION

Poly(2-alkoxyethyl methacrylates)



(R = alkyl) can be regarded as polymers formally derived from the widely applied poly(2-hydroxyethyl methacrylate) (R = H, PHEMA). These polymers, which in many respects resemble by their structure and properties poly(*n*-alkyl methacrylates), have been applied in practice only recently. This is particularly true of ethoxy and butoxy derivatives, which are used as comonomers in the modification of various polymers. Homopolymers, i. e., poly(2-ethoxyethyl methacrylate) (R = ethyl, PEEMA) and poly(2-*n*-butoxyethyl methacrylate) (R = *n*-butyl, PBEMA) have not yet been systematically investigated.

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This study is concerned with a comparison of the solution properties of PEEMA and PBEMA with poly(2-methoxyethyl methacrylate) (R = methyl, PMEMA), investigated earlier.<sup>1</sup> The main attention is concentrated on the solution characterization of these polymers by the light-scattering and viscometric methods, and on the determination of some molecular parameters, e. g., unperturbed dimensions of polymer coils in solution, and on their dependence on the side-chain length.

#### EXPERIMENTAL

##### *Preparation of Monomers and Polymers*

The monomers were prepared by esterification of methacrylic acid (Fluka AG, Switzerland) with 2-ethoxyethanol (ethylcellosolve, Lachema, Czechoslovakia) and 2-butoxyethanol (butylcellosolve, Fluka AG). The esterification was catalyzed with *p*-toluenesulfonic acid, the forming monomer was stabilized with hydroquinone (0.5 wt. % in the reaction mixture). Water formed in the reaction was separated by azeotropic distillation with benzene. The esterification proceeded for some 15 h. After that, the reaction mixture was diluted with *n*-hexane, and residues of the starting compounds and the stabilizer were extracted with a 2% aqueous solution of sodium hydroxide, and then with water to neutral reaction. After drying with magnesium sulfate, benzene and *n*-hexane were removed by distillation and the raw product, stabilized with 0.1 wt. % of octylpyrocatechol, was distilled on a laboratory column; b. p. 72–73 °C/2 kPa (15 mm Hg) for 2-ethoxyethyl methacrylate and 54–55 °C/0.013 kPa (0.1 mm Hg) for 2-butoxyethyl methacrylate. Purity of the monomers was checked by gas chromatography (>99.95%). The respective yields were 35 and 40%.

Both monomers were polymerized at 60 °C in an inert nitrogen atmosphere. Each time, three samples (A, B, C), having different molecular weight ( $M_w \sim 1 \times 10^5$ ,  $5 \times 10^5$  and  $10 \times 10^5$ ), were prepared. The molecular weight was regulated by the concentration of the initiator, azobisisobutyronitrile ( $10^{-3} - 2 \times 10^{-2}$  mol/dm<sup>3</sup>), and by the content of the diluent, toluene (50–90 vol. %). The polymerization time, hours by the order of magnitude, was estimated so as to make the resulting polymerization conversion 20–30 wt. %. The PMEMA samples were prepared earlier by a similar procedure.<sup>1</sup>

##### *Fractionation*

The PEEMA and PBEMA fractions were prepared by the precipitation fractionation of three samples (A, B, C) using a methyl ethyl ketone (MEK) — methanol system at 25 °C. The fractions were reprecipitated from a dilute acetone solution into excess of *n*-hexane. Since swollen polymers adhere to glass and their isolation becomes therefore difficult, work in polyethylene vessels can be recommended. The fractions were dried at 60 °C in vacuo to constant weight.

##### *Light Scattering*

Solutions of PEEMA and PBEMA were optically purified by centrifugation in a Beckman L8–55 ultracentrifuge in a swinging-bucket rotor SW 28 at 15 000 rpm for one hour, or in the case of samples of a lower expected molecular weight ( $< 3 \times 10^5$ ) by pressure filtration through a sintered glass filter G5 (VEB Jenear Glasswerk, GDR).

Light scattering from these solutions was measured at 25 °C with a Sofica 42.000 apparatus or, at elevated temperatures, with a FICA 50 apparatus, with unpolarized primary light of the wavelength 546 nm. The results were evaluated by the Zimm method.

The refractive index increments,  $dn/dc$  (Table I), were determined by a differential refractometer Brice Phoenix BP-2000-V.

TABLE I

Refractive Index Increments  $dn/dc$  of Poly(2-alkoxyethyl methacrylates) for the Wavelength 546 nm in MEK at 25 °C and in *n*-Butanol at the  $\Theta$ -Temperature Given in Parenthesis

| Solvent           | $(dn/dc)/\text{cm}^3 \cdot \text{g}^{-1}$ |               |               |
|-------------------|---|---------------|---------------|
|                   | PMEMA                                     | PEEMA         | PBEMA         |
| MEK               | 0.108 <sup>a</sup>                        | 0.100         | 0.099         |
| <i>n</i> -Butanol | 0.085 (64 °C)                             | 0.078 (38 °C) | 0.076 (17 °C) |

<sup>a</sup> Taken from ref. 1.

### Viscometry

The specific viscosities of polymer fractions in MEK and *n*-butanol at 25 °C were determined with a modified Ubbelohde viscometer of the capillary diameters 0.47 and 0.60 mm, respectively. The kinetic energy corrections could be neglected in all cases. The intrinsic viscosities were obtained by a linear extrapolation of the concentration dependences of reduced viscosity to zero polymer concentration. The viscosity behaviour was Newtonian within the whole range of molecular weights.

## RESULTS AND DISCUSSION

### The Solubility of Polymers and $\Theta$ -Solvents

The solubility parameters  $\delta$  calculated for PMEMA, PEEMA and PBEMA by a method according to Small<sup>2</sup> are, respectively, 18.8, 17.8 and 17.3 (J/cm<sup>3</sup>)<sup>1/2</sup>. Hence, with respect to their solubility behaviour, poly(2-alkoxyethyl methacrylates) resemble poly (*n*-alkyl methacrylates) having a comparable side-chain length.<sup>3</sup> The replacement of the methylene group with the ether group in the side chains has no pronounced effect.

All polymers investigated in this study can be readily dissolved in aromatic and halogenated hydrocarbons, aliphatic ketones and common organic acids. They do not dissolve in water, aliphatic ethers and hydrocarbons. Extension of the side chain somewhat reduces the polar character of the polymer ( $\delta$  parameter decreases), so that, e. g., PBEMA is soluble in cyclohexane at room temperature. A solubility behaviour which is difficult to predict is met with esters as solvents (e. g., unlike other polymers, the high-molecular-weight PBEMA is macroscopically insoluble in *n*-butyl acetate) and in cyclic ethers (PEEMA is insoluble in dioxan, PBEMA in tetrahydrofuran, etc.).

All three polymers — PMEMA, PEEMA and PBEMA — are soluble in aliphatic alcohols at elevated temperature; PBEMA dissolves in *n*-butanol also at room temperature. Alcohols form cosolvent mixtures with water. Often even a low amount of water in alcohols may considerably affect the solubility behaviour of the polymers.

The  $\Theta$ -temperatures were determined in *n*-butanol for all polymers investigated in this study by interpolating the temperature dependences of the second virial coefficient,  $A_2$ , measured by light scattering to its zero value (Figure 1). The respective  $\Theta$ -temperatures thus obtained for PMEMA, PEEMA

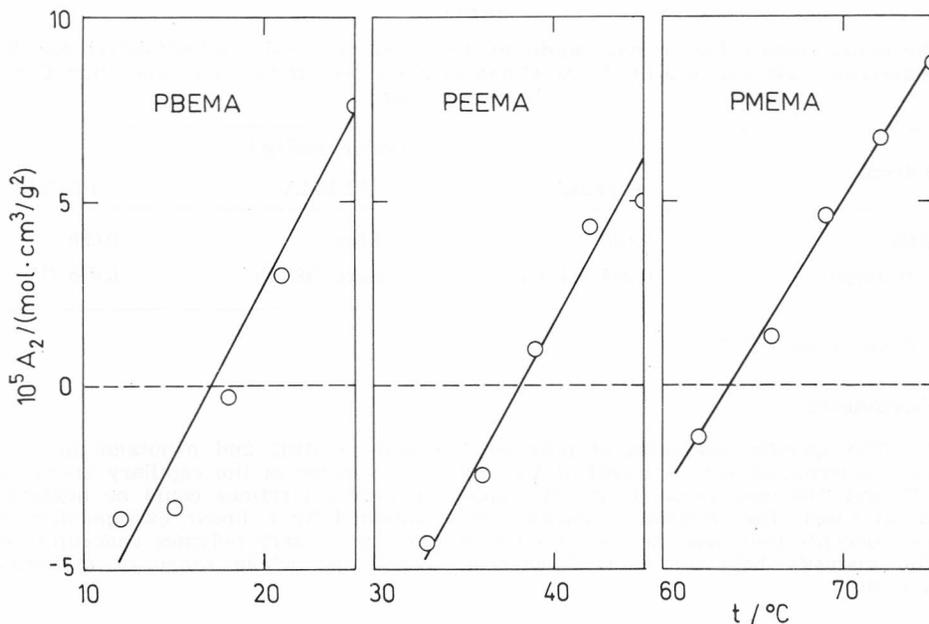


Figure 1. Determination of the  $\Theta$ -temperature of poly(2-alkoxyethyl methacrylates) in *n*-butanol from the dependence of the second virial coefficient,  $A_2$ , on temperature,  $t$ . Molecular weight of the polymers  $M_w \sim 1 \times 10^5 - 1.5 \times 10^5$ .

and PBEMA are 64, 38 and 17 °C. For comparison: the  $\Theta$ -temperature for poly(2-hydroxyethyl methacrylate) in *n*-butanol<sup>4</sup> is 89 °C. Thus, the  $\Theta$ -temperatures in *n*-butanol decrease monotonically with increasing length of the side chain.

The finding that, e.g., the  $\Theta$ -temperature of poly(*n*-octylmethacrylate)<sup>5</sup> is 16.8 °C in the same solvent, additionally illustrates the fact reported earlier, viz., that the solution behaviour of poly(*n*-alkyl methacrylates) and poly(2-alkoxyethyl methacrylates) is similar. Quite understandably, the closer this similarity, the longer are the side chains and, consequently, the weaker the relative effect of one ether group.

#### Viscosity — Molecular Weight Relations

Methyl ethyl ketone (MEK) was selected as solvent for the solution characterization of both polymers, PEEMA and PBEMA. The same solvent had been used earlier to characterize PMEMA.<sup>1</sup> With respect to the determination of the conformation characteristics of the polymers, it is advantageous to use  $\Theta$ -solvents, where the excluded volume effect can be neglected. No thermodynamically poor solvent could be found for PEEMA in the vicinity of room temperature. For PBEMA, *n*-butanol is such a solvent, and measurements for PBEMA were therefore performed also in *n*-butanol. To compare the results of measurements in various solvents, all intrinsic viscosities were determined at 25 °C. The results of characterization of the PEEMA and PBEMA fractions are summarized in Tables II and III, respectively.

TABLE II

Molecular Characteristics of Poly(2-ethoxyethyl methacrylate) (PEEMA): Molecular Weight,  $M_w$ , and the Second Virial Coefficient,  $A_2$ , of Fractions Determined by Light Scattering in MEK,  $M_w/M_n$  Ratio Estimated by Means of GPC and Intrinsic Viscosity  $[\eta]$  in MEK at 25 °C

| Fraction <sup>a</sup> | $10^3 \cdot M_w$ | $10^4 \cdot A_2^b$<br>mol·cm <sup>3</sup> /g <sup>2</sup> | $M_w/M_n$ | $[\eta]_{\text{MEK}}$<br>cm <sup>3</sup> /g |
|-----------------------|------------------|---|-----------|---|
| A-1                   | 4130             | 1.7   | —         | 309   |
| A-2                   | 2610             | 2.0   | —         | 258   |
| B-1                   | 2290             | 2.0   | —         | 161   |
| A-3                   | 2120             | 2.2   | —         | 201   |
| B-2                   | 1770             | 2.4   | —         | 169   |
| A-4                   | 1050             | 3.1   | —         | 152   |
| B-3                   | 1010             | 2.3   | —         | 135   |
| B-4                   | 895              | 3.0   | 1.17      | 91.9  |
| A-5                   | 830              | 2.6   | 1.32      | 106   |
| B-5                   | 445              | 3.2   | 1.25      | 62.9  |
| A-6                   | 420              | 3.1   | 2.07      | 54.6  |
| B-6                   | 385              | 3.3   | 1.30      | 71.8  |
| C-1                   | 330              | 4.0   | 1.51      | 51.9  |
| C-2                   | 300              | 3.4   | 1.60      | 40.1  |
| C-3                   | 210              | 4.2   | 1.56      | 35.3  |
| B-7                   | 210              | 3.7   | 1.62      | 37.5  |
| C-4                   | 150              | 6.2   | 1.34      | 30.0  |
| C-5                   | 105              | 6.4   | 1.27      | 21.2  |
| C-6                   | 90               | 7.3   | 1.19      | 17.9  |
| C-7                   | 53               | 8.5   | 1.20      | 11.4  |

<sup>a</sup> Number of fraction obtained from the starting polymer A, B, or C.

<sup>b</sup>  $A_2 = 3.34 \times 10^{-2} M_w^{-0.35}$ .

TABLE III

Molecular Characteristics of Poly(2-butoxyethyl methacrylate) (PBEMA): Molecular Weight,  $M_w$ , and the Second Virial Coefficient,  $A_2$ , of Fractions Determined by Light Scattering in n-Butanol,  $M_w/M_n$  Ratio Estimated by Means of GPC and Intrinsic Viscosity,  $[\eta]$ , in n-Butanol and in MEK at 25 °C

| Fraction <sup>a</sup> | $10^3 \cdot M_w$ | $10^4 \cdot A_2^b$<br>mol·cm <sup>3</sup> /g <sup>2</sup> | $M_w/M_n$ | $[\eta]$ in BuOH<br>cm <sup>3</sup> /g | $[\eta]$ in MEK<br>cm <sup>3</sup> /g |
|-----------------------|------------------|---|-----------|--|---------------------------------------|
| A-2                   | 6600             | 0.27  | —         | 113                                    | 330                                   |
| B-1                   | 3500             | 0.21  | —         | 87.1                                   | 242                                   |
| A-3                   | 2550             | 0.39  | —         | 67.7                                   | 177                                   |
| A-4                   | 2500             | 0.41  | —         | 77.2                                   | 187                                   |
| A-5                   | 1700             | 0.44  | —         | 64.0                                   | 158                                   |
| A-6                   | 1100             | 0.61  | —         | 52.2                                   | 125                                   |
| B-2                   | 970              | 0.44  | 1.31      | 48.2                                   | 106                                   |
| B-3                   | 700              | 0.54  | 1.22      | 41.5                                   | 87.5                                  |
| B-4                   | 550              | 0.35  | 1.18      | 36.5                                   | 72.0                                  |
| A-7                   | 525              | 0.81  | 1.33      | 33.4                                   | 64.4                                  |
| B-5                   | 315              | 0.74  | 1.15      | 27.9                                   | 55.4                                  |
| C-1                   | 305              | 0.56  | 1.58      | 24.4                                   | 40.2                                  |
| B-6                   | 155              | 0.35  | 1.45      | 16.6                                   | 32.4                                  |
| C-2                   | 120              | 0.76  | 1.28      | 17.3                                   | 27.2                                  |
| C-3                   | 90               | 0.73  | 1.20      | 13.3                                   | 24.5                                  |
| C-4                   | 78               | 0.94  | 1.15      | 11.9                                   | 20.9                                  |
| C-5                   | 55               | 0.80  | 1.37      | 10.4                                   | 16.3                                  |

<sup>a</sup> Cf. Table II.

<sup>b</sup>  $A_2 = 9.08 \times 10^{-4} M_w^{-0.22}$ .

TABLE IV

Constants of the Mark-Houwink Equation  $K$  and  $a$ , Viscometric Constant  $K_o$ , Characteristic Ratio  $C_\infty$  and Steric Factor  $\sigma$  of Poly(2-alkoxyethyl methacrylates) at 25 °C

| Polymer            | Solvent | $a$             | $10^3 \cdot K$<br>cm <sup>3</sup> /g | $10^3 \cdot K_o^b$<br>cm <sup>3</sup> /g | $C_\infty$ | $\sigma$ |
|--------------------|---------|-----------------|--------------------------------------|--|------------|----------|
| PMEMA <sup>a</sup> | MEK     | $0.71 \pm 0.01$ | 7.3                                  | 55                                       | 11.1       | 2.35     |
| PEEMA              | MEK     | $0.75 \pm 0.02$ | 3.8                                  | 58                                       | 12.6       | 2.51     |
| PBEMA              | MEK     | $0.65 \pm 0.01$ | 14.1                                 | 62                                       | 15.5       | 2.78     |
| PBEMA              | n-BuOH  | $0.54 \pm 0.01$ | 30.0                                 | 44                                       | 12.3       | 2.48     |

<sup>a</sup> Results taken from ref. 1.

<sup>b</sup> Error of determination  $\pm 5\%$  on the average.

The constants of the Mark-Houwink equation,  $[\eta] = K M^a$  (Table IV), were determined from a logarithmic plot of intrinsic viscosity,  $[\eta]$ , vs. molecular weight,  $M_w$  (Figure 2). Correction for the polydispersity of fractions was comparable with the experimental error of molecular-weight determination by light scattering. That is why the viscosity-average molecular weight,  $M_\eta$ , is regarded below as equal to the weight average,  $M_w$ .

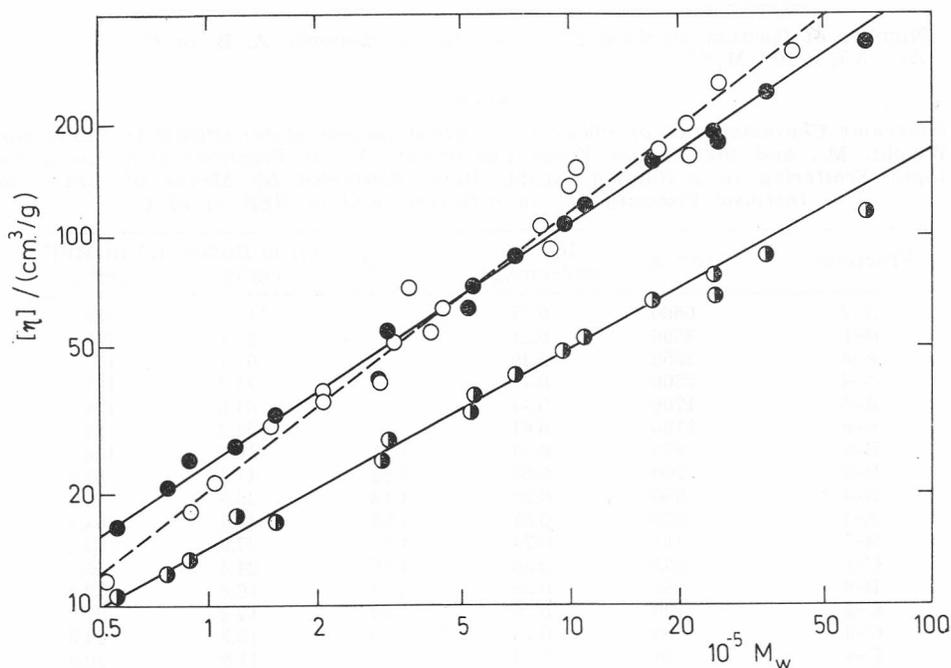


Figure 2. Relation between the intrinsic viscosity,  $[\eta]$ , and the weight-average molecular weight,  $M_w$ , for PEEMA (---) in MEK (○) and for PBEMA (—) in MEK (●) and in *n*-butanol (●) at 25 °C.

During the fractionation of the PBEMA sample with the highest molecular weight (A), it was found that the first fraction (A-1) was not completely soluble and contained a gel. This indicates that the high-molecular weight fractions of PBEMA may contain branched structures. Therefore, a straight line was plotted successively, each time through ten adjacent points of the  $[\eta] - M_w$  dependence, and the value of the slope was checked; if any of the points corresponding to the three fractions with highest molecular weight were included, a marked decrease of the slope could be seen. These three fractions were suspected to contain branched structures and, therefore, were not considered in further calculations. No such effect was observed with PEEMA.

From the values of the exponents in the Mark-Houwink equation (Table IV), one can see that, for the polymers investigated here, MEK is a thermodynamically good solvent; *n*-butanol at 25 °C is almost a  $\Theta$ -solvent for PBEMA; the low positive values of the second virial coefficient  $A_2$  (Table III) confirm that the  $\Theta$ -temperature is lower than 25 °C.

### Conformation Characteristics

One of the basic characteristics describing the conformation of a polymer chain in solution is the characteristic ratio,

$$C_\infty = \frac{\langle r^2 \rangle_0}{nl^2} \quad (1)$$

where  $\langle r^2 \rangle_0$  is the unperturbed mean square end-to-end distance, *n* is the number of bonds in the main chain, and *l* is the bond length (for the C—C bond, *l* = 0.154 nm). The characteristic ratio can be obtained experimentally from viscosity measurements using the relation

$$C_\infty = \left( \frac{K_0}{\Phi_0} \right)^{2/3} \frac{M_b}{l^2} \quad (2)$$

in which  $K_0$  is the constant of the Mark-Houwink equation for a polymer in the  $\Theta$ -solvent and  $M_b$  is molecular weight per skeletal bond. The recommended value of the viscosity function is<sup>6</sup>  $\Phi_0 = 2.5 \times 10^{23} \text{ g}^{-1}$ .

Another conformation characteristic frequently used in the literature is the steric factor defined as the ratio of actual unperturbed dimensions to those which would have a freely rotating chain with a fixed bond angle,  $\sigma = \langle r^2 \rangle_0^{1/2} / \langle r^2 \rangle_{\text{fr}}^{1/2}$ . For polymers of the vinyl type, this factor is related to the characteristic ratio by  $\sigma = (C_\infty/2)^{1/2}$ .

The viscometric constant  $K_0$  for PBEMA was estimated from the intrinsic viscosities determined in thermodynamically good solvents using a plot according to Burchard,<sup>7</sup> and Stockmayer and Fixman<sup>8</sup> (Figure 3),

$$\frac{[\eta]}{M^{1/2}} = C_0 K_0 + C_1 M^{1/2} \quad (3)$$

where  $C_0$ ,  $C_1$  are parameters. To estimate  $K_0$  by linear extrapolation according to (3), the determined intercept must be corrected<sup>9</sup> by the empirical factor  $C_0$ . Its value for the range of the expansion coefficients  $1.2 < \alpha_n^3 < 2.3$  is

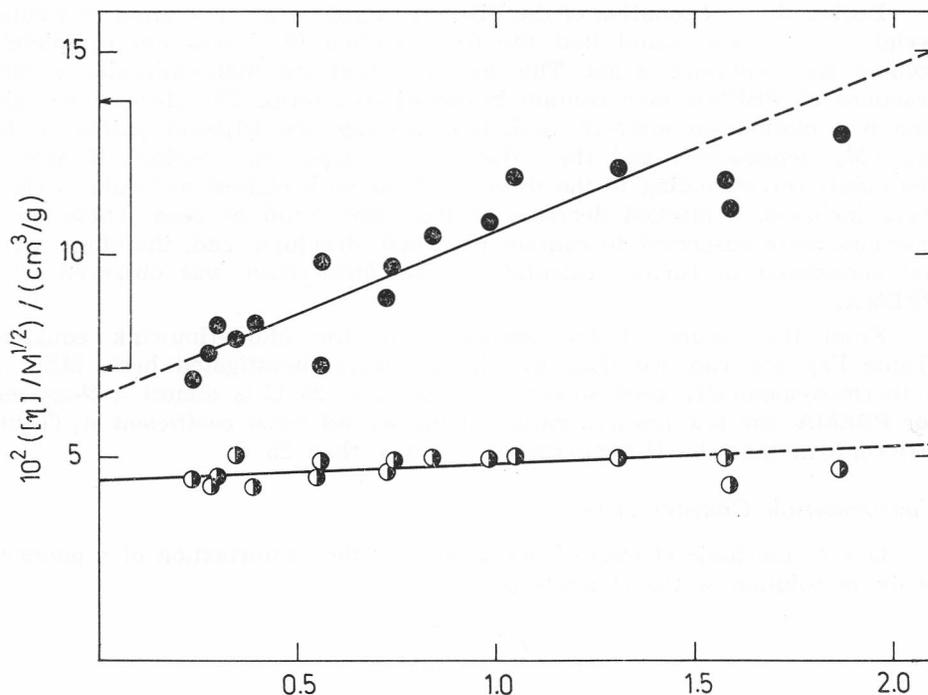


Figure 3. Data plot for poly(2-butoxyethyl methacrylate) in MEK (●) and *n*-butanol (◐) according to Burchard-Stockmayer-Fixman. Arrows indicate approximately the range of  $[\eta]/M^{1/2}$  values for which  $1.2 < \alpha_n^3 < 2.3$ .

usually taken<sup>10</sup> as  $C_0 = 1.05$  (data in MEK, Figure 3). If for all points the condition  $\alpha_n^3 < 1.6$  is fulfilled (data in *n*-butanol, Figure 3), then<sup>8</sup>  $C_0 = 1$ . The  $K_0$  values thus obtained are summarized in Table IV. The data plot suggested by Tanaka<sup>11</sup>

$$\left( \frac{[\eta]}{M^{1/2}} \right)^{5/3} = K_0^{5/3} + C_2 M^{1/2} \quad (4)$$

where  $C_2$  is a parameter, gave for PBEMA results in very good agreement with the preceding method. Let it be recalled that three fractions with the highest molecular weight, for which the presence of branched structures cannot be ruled out, have not been included into the extrapolation procedures.

The data for PEEMA (Table II) were treated in a similar way. As, however, their scatter is larger than in the case of PBEMA data (cf. Figure 2), the determined characteristics of the unperturbed dimensions of PEEMA (Table IV) should be regarded only as a rough estimate.

All the extrapolation procedures used lead to the same conclusion, namely, that for the polymers under investigation  $K_0$  increases in the series PMEMA  $<$  PEEMA  $<$  PBEMA. The calculated values of the steric factor increase with the increasing length of the side chain (Table IV) in agreement with the expected increasing hindrance to free rotation within the polymer chain. The

values of the steric factor are higher by 0.3—0.4, on the average, compared with poly(*n*-alkyl methacrylates) having a similar length of the side chain.<sup>12</sup> For instance, for polymers of ethyl, butyl, hexyl and octyl methacrylates, Chinai et al.<sup>5</sup> found the respective values  $\sigma = 1.94, 2.09, 2.44$  and  $2.32$ . In this respect, the presence of the ether group in the side chain of poly(2-alkoxyethyl methacrylates) has a more pronounced effect.

The results also show that the conformation parameters of PBEMA are different in *n*-butanol and MEK (Table IV). This observation is in agreement with the results reported in the literature<sup>13</sup> from which it can be seen that in self-associating alcohols the expected  $K_o$  values, and thus also the values of the steric factor, are systematically lower compared with other solvents. For instance, for poly(*n*-octyl itaconate) in *n*-butanol and tetrahydrofuran, Gargallo and Radić<sup>14</sup> found  $\sigma = 2.50$  and  $3.03$ , respectively. Also, Bohdanecký and Berek<sup>15</sup> observed for polystyrene that its unperturbed dimensions are higher in nonpolar solvents than in the polar ones.

To supplement the data in Table II, let it be mentioned that the  $\sigma$  values reported in the literature for the steric factor of poly(2-hydroxyethyl methacrylate) in polar solvents<sup>16</sup> range between 2.33 and 2.55; similarly, for stereoregular forms of this polymer in alcohols and their mixtures with water, the respective values are<sup>17</sup> 2.05—2.88. The higher values of the steric factor for poly(2-hydroxyethyl methacrylate), which in fact is the first member of the polymer series studied, are a consequence of the ability of the hydroxyl group to form hydrogen bonds with the solvent.

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## SAŽETAK

## Svojstva razrijeđenih otopina i dimenzije lanca poli(2-alkoksietil-metakrilata)

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Uspoređena su svojstva otopina poli(2-metoksietilmetakrilata) (PMEMA), poli(2-etoksietilmetakrilata) (PEEMA) i poli(2-*n*-butoksietilmetakrilata) (PBEMA). Odgovarajuće  $\Theta$ -temperature u *n*-butanolu iznose 64, 38 i 17 °C. Iz odnosa intrinzične viskoznosti i molekulske mase u termodinamički dobrom otapalu metil-etil-ketonu (MEK) izračunati su karakteristični omjeri  $C_{\infty} = 11,1, 12,6$  i 15,5. Nepromijenjene dimenzije PBEMA ovise o prirodi otapala; manje su u *n*-butanolu (12,3) nego u MEK. Konformacijska svojstva poli(2-alkoksietilmetakrilata) slična su svojstvima poli(*n*-alkilmetakrilata) s usporedivom duljinom pobočnog lanca.