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## **SYNTHESIS AND SOLUTION PROPERTIES OF A NOVEL SIDE-CHAIN POLYAMINIMIDE**

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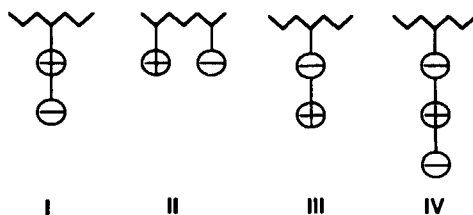
### **ABSTRACT**

The novel polyaminimide polymer, poly[sodium dimethyl(3-propyl)amine-4-methacrylamidobenzimide sulfonate] (PSDM), was prepared. This polymer contains a pendant sulfonate group in addition to the dipolar aminimide moiety. The dilute solution viscosity and light-scattering behavior of PSDM were studied as a function of salt concentration. It was found that the polymer exhibited typical polyelectrolyte behavior in spite of the presence of the dipolar aminimide group.

### **INTRODUCTION**

A number of studies have been published concerning the synthesis and solution properties of ampholytic polymers. These investigations have focused on polymers derived from sulfobetaines I [1–3], in which the positive and negative charges reside on a single pendant group, ion-pair comonomers II [4–9], in which the charges exist on separate pendant groups and either alternate or exist in a random configuration, and aminimides III [10–14], in which the positive and negative charges are on a single pendant group and exist as a dipolar ion.

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The aqueous solution properties of these various classes of polyampholytes are found to vary somewhat with structure; however, some generalizations can be made regarding each class. In the case of polysulfobetaine inner salts, these polymers are generally found to be insoluble in aqueous media due to the intra- and intermolecular attractions of the dipolar units they possess. These interactions are found to be disrupted by the addition of a simple salt, and the viscosity of the resulting solution is found to increase with increasing salt concentration, contrary to what is observed in the case of polyelectrolytes. This unusual increase in the viscosity is accounted for by the expansion of the polymer chains in solution as more intra- and intermolecular ionic interactions are replaced by interactions with the added salt. Similarly, most polymers prepared from ion-pair comonomers are also water insoluble but soluble in aqueous salt solution. In addition, these solutions also show an increase in viscosity with salt concentration. To date, very little work has been published on the solution properties of polyaminimides or the effect of salt on these systems. While these polymers are reported to be soluble in water due to the highly polar nature of the aminimide group [10, 11], additional work has shown that the water solubility of both main-chain and side-chain polyaminimides is highly dependent on the degree of hydrophobic/hydrophilic character of the remaining portion of the polymer [12, 13]. These polymers have also been reported to show interesting rheological behavior in aqueous solution [14].

In this work we report on the synthesis of a novel aminimide polymer which has the ionic configuration depicted as Structure IV, as well as its aqueous solution properties in the presence of added salt.

## EXPERIMENTAL

### Starting Materials

All solvents were dried and distilled prior to use. 4,4'-Azobis(cyanovaleic acid) (ACVA), Aldrich, was recrystallized once from water, 1,3-Propanesultone, Aldrich, was distilled under vacuum.

## Techniques

NMR spectra were recorded using a Bruker-IBM model WP-270-SY spectrometer. Proton spectra were recorded in 270.13 MHz and carbon spectra were obtained at 67.92 MHz. Gated decoupling and additivity parameters were used to identify the carbon resonances where necessary. Light-scattering data were obtained using a Dawn model F laser photometer in the batch mode at room temperature. The refractive index increments were measured with a Brice-Phoenix differential refractometer. Dilute solution viscosity measurements were made using a Ubbelohde dilution viscometer maintained in a water bath at  $25 \pm 0.05^\circ\text{C}$ . Elemental analyses were determined by Atlantic Microlabs Inc., Atlanta, Georgia.

## Syntheses

### 1,1-Dimethyl-2-(4-nitrobenzoyl)hydrazide (V)

This material was prepared by modification of the procedure of Smith et al. [15].

A solution of 12.38 g (0.06 mol) dicyclohexylcarbodiimide and 3.60 g (0.06 mol) 1,1-dimethylhydrazine in 200 mL methylene chloride was cooled to  $0^\circ\text{C}$  in an ice bath. To this solution was added 10.30 g (0.06 mol) *p*-nitrobenzoic acid over a period of 20 min, and the resulting mixture was stirred overnight at room temperature. The precipitated dicyclohexylurea was removed by filtration, and the filtrate was treated with 100 mL of 4 *N* HCl to give a yellow precipitate which was then separated by filtration. The methylene chloride layer was extracted twice more with 4 *N* HCl, the combined HCl extracts added to the precipitate, and the mixture neutralized with sodium carbonate. The neutralized mixture was extracted three times with 200 mL chloroform and the combined extracts were dried with anhydrous sodium sulfate overnight. Evaporation of the solvent afforded a yellow solid which was recrystallized from benzene to give 8.7 g (a 69% yield) of yellow needles which melted at  $148\text{--}150^\circ\text{C}$  (literature,  $151\text{--}153^\circ\text{C}$  [15]).

$^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$ 2.67 (s, 6H),  $-\text{CH}_3$ ; 8.05 (s, 2H), 8.31 (s, 2H) aromatic; 9.70 (s, 1H),  $-\text{NH}$ .

$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$ 47.37 (q),  $-\text{CH}_3$ ; 123.19 (d), 128.59 (d), 139.70 (s), 148.84 (s), aromatic; 162.37 (s),  $\text{C}=\text{O}$ .

Analysis. Calculated for  $\text{C}_9\text{H}_{11}\text{N}_3\text{O}_3$ : C, 51.67%; H, 5.26%; N, 20.02%. Found: C, 51.65%; H, 5.28%; N, 19.97%.

## 1,1-Dimethyl-2-(4-aminobenzoyl)hydrazide (VI)

To 10 g (0.05 mol) of V in 200 mL absolute ethanol was added 0.5 g of 5% palladium on carbon. The mixture was placed on a Parr shaker at 50 psi of hydrogen and reacted until the hydrogen uptake ceased (approximately 8 h). The resulting solution was filtered through Celite and the filtrate evaporated to dryness to give a white solid. After recrystallization from ethyl acetate, 8.06 g (a 90% yield) of white crystals was obtained which melted at 180–182°C (literature, 136–138°C [16]).

$^1\text{H}$  NMR (DMSO- $d_6$ ):  $\delta$ 2.57 (s, 6H),  $-\text{CH}_3$ ; 5.51 (s, 2H),  $-\text{NH}_2$ ; 6.55 (d, 2H,  $J = 7.3$  Hz), 7.54 (d, 2H,  $J = 7.4$  Hz) aromatic; 8.91 (s, 1H)  $-\text{NH}$ .

$^{13}\text{C}$  NMR (DMSO- $d_6$ ):  $\delta$ 46.21 (q),  $-\text{CH}_3$ ; 112.47 (d), 120.68 (d), 128.64 (d), 151.45 (s), aromatic; 164.53 (s),  $\text{C}=\text{O}$ .

Analysis. Calculated for  $\text{C}_9\text{H}_{13}\text{N}_3\text{O}$ : C, 60.34%; H, 7.26%; N, 23.46%. Found: C, 60.43%; H, 7.34%; N, 23.37%.

## 1,1-Dimethyl-1,2-(4-methacrylamidobenzoyl)hydrazide (VII)

This material was prepared by adaptation of the method of Bahr et al. [16].

To a suspension of 17.9 g (0.1 mol) of VI in 50 mL benzene containing a trace of phenothiazine was added 20.0 g (0.13 mol) methacrylic anhydride. The mixture was heated to 60°C to obtain a clear solution. After approximately 20 min a profuse quantity of precipitate was observed, at which time the heat was removed and the reaction cooled to room temperature. The precipitate was collected by filtration and washed with a small amount of benzene. After recrystallization from ethyl acetate, 18.73 g (a 76.0% yield) of off-white crystals was obtained which melted at 172–175°C (literature, 156–158°C [16]).

$^1\text{H}$  NMR (DMSO- $d_6$ ):  $\delta$ 1.99 (s, 3H),  $\text{C}=\text{C}-\text{CH}_3$ ; 2.60 (s, 6H),  $\text{N}-\text{CH}_3$ ; 5.55 (s, 1H),  $\text{H}-\text{C}=\text{C}-\text{CH}_3$  *cis*; 5.89 (s, 1H)  $\text{H}-\text{C}=\text{C}-\text{CH}_3$  *trans*; 7.82 (s, 4H), aromatic; 9.35 (s, 1H),  $-\text{NH}$  amide, 9.99 (s, 1H), hydrazide.

$^{13}\text{C}$  NMR (DMSO- $d_6$ ):  $\delta$ 18.50 (q),  $\text{C}=\text{C}-\text{CH}_3$ ; 46.12 (q),  $\text{N}-\text{CH}_3$ ; 119.19 (d), aromatic; 120.16 (t),  $\text{H}_2\text{C}=\text{C}$ ; 127.77 (d), 128.69 (s), aromatic; 140.20 (s),  $=\text{C}-\text{CH}_3$ ; 141.62 (s), aromatic; 163.88 (s),  $\text{C}=\text{O}$  hydrazide; 166.91 (s),  $\text{C}=\text{O}$  amide.

Analysis: Calculated for  $\text{C}_{13}\text{H}_{17}\text{N}_3\text{O}_2$ : C, 63.16%; H, 6.88%; N, 17.00%. Found: C, 63.05%; H, 6.97%; N, 16.97%.

### 3-[1,1-Dimethyl-2-(4-methacrylamidobenzoyl)-1-hydrazinium]-1-propanesulfonate (VIII)

To a suspension of 10 g (0.04 mol) of VII in 50 mL of dry acetone containing a trace amount of phenothiazine was added 5.37 g (0.044 mol) 1,3-propanesultone. The mixture was heated to reflux and after a short time became thick and difficult to stir. The mixture was held at reflux for 48 h and then allowed to cool to room temperature. The precipitate was collected by filtration and washed with a small amount of cold acetone. After drying at 50°C under vacuum, 13.97 g (a 91% yield) of white powder was obtained which melted at 240–243°C (literature, 230–232°C [16]) after recrystallization from an ethanol–water mixture.

<sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>): δ1.97 (s, 3H), C=C–CH<sub>3</sub>; 2.09 (m, 2H), CH<sub>2</sub>–CH<sub>2</sub>–CH<sub>2</sub>; 2.52 (t, 2H), –CH<sub>2</sub>–SO<sub>3</sub>; 3.70 (s, 6H), N–CH<sub>3</sub>; 4.15 (t, 2H), N–CH<sub>2</sub>; 5.59 (s, 1H)  $\underline{\text{H}}-\text{C}=\text{C}-\text{CH}_3$  *cis*; 5.89 (s, 1H),  $\underline{\text{H}}-\text{C}=\text{C}-\text{CH}_3$  *trans*; 7.88 (m, 4H), aromatic; 10.16 (s, 1H), –NH amide; 11.56 (s, 1H), –NH hydrazinium.

<sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>): δ18.49 (q), C=C–CH<sub>3</sub>; 19.29 (t), CH<sub>2</sub>–CH<sub>2</sub>–CH<sub>2</sub>; 47.27 (t), CH<sub>2</sub>–SO<sub>3</sub>; 54.68 (q), N–CH<sub>3</sub>; 66.16 (t), N–CH<sub>2</sub>; 119.22 (d), aromatic; 120.76 (t),  $\underline{\text{H}}_2\text{C}=\text{}$ ; 125.29 (s), 129.05 (d), aromatic; 139.93 (s) =C–CH<sub>3</sub>; 143.45 (s), aromatic; 165.03 (s), C=O hydrazinium; 167.08 (s), C=O amide.

Analysis. Calculated for C<sub>16</sub>H<sub>23</sub>N<sub>3</sub>O<sub>5</sub>S: C, 52.03%; H, 6.23%; N, 11.38%; S, 8.97%. Calculated for C<sub>16</sub>H<sub>23</sub>N<sub>3</sub>O<sub>5</sub>S(0.1 H<sub>2</sub>O): C, 49.61%; H, 6.46%; N, 10.85%; S, 8.27%. Found: C, 49.67%; H, 6.53%; N, 10.84%; S, 8.33%.

### Sodium Dimethyl(3-propyl)amine-4-methacrylamidobenzimide Sulfonate (IX)

A suspension of 6.0 g (0.016 mol) of VIII in 100 mL of distilled water was titrated with 1 N NaOH to a phenolphthalein endpoint to give a clear pink solution. The resulting solution was freeze dried to obtain 6.44 g (a 98% yield) of white solid which melted at 250–253°C after recrystallization from ethanol.

<sup>1</sup>H NMR (D<sub>2</sub>O): δ1.93 (s, 3H), C=C–CH<sub>3</sub>; 2.19 (m, 2H, *J* = 7.1 Hz), CH<sub>2</sub>–CH<sub>2</sub>–CH<sub>2</sub>; 2.94 (t, 2H, *J* = 7.3 Hz), CH<sub>2</sub>SO<sub>3</sub>; 3.37 (s, 6H), N–CH<sub>3</sub>; 3.87 (t, 2H, *J* = 7.6 Hz), N–CH<sub>2</sub>; 5.51 (s, 1H),  $\underline{\text{H}}-\text{C}=\text{C}-\text{CH}_3$  *cis*; 5.74 (s, 1H),  $\underline{\text{H}}-\text{C}=\text{C}-\text{CH}_3$  *trans*; 7.42 (d, 2H), 7.70 (d, 2H), aromatic.

$^{13}\text{C}$  NMR ( $\text{D}_2\text{O}$ ):  $\delta$ 18.53 (q),  $\text{C}=\text{C}-\underline{\text{C}}\text{H}_3$ ; 19.49 (t),  $\text{CH}_2\underline{\text{C}}\text{H}_2\text{CH}_2$ ; 48.34 (t),  $\text{CH}_2\text{SO}_3$ ; 52.45 (q),  $\text{N}-\underline{\text{C}}\text{H}_3$ ; 64.61 (t),  $\text{N}-\underline{\text{C}}\text{H}_2$ ; 118.62 (d), aromatic; 119.64 (t),  $\text{H}_2\underline{\text{C}}=$ ; 127.41 (d), 134.62 (s), aromatic; 139.37 (s),  $\text{C}=\underline{\text{C}}-\text{CH}_3$ ; 140.27 (s), aromatic; 166.51 (s),  $\text{C}=\text{O}$  hydrazinium; 167.14 (s),  $\text{C}=\text{O}$  amide.

Analysis. Calculated for  $\text{C}_{16}\text{H}_{22}\text{N}_3\text{O}_5\text{SNa}$ : C, 49.10%; H, 5.63%; N, 10.74%; S, 8.18%. Found: C, 49.31%; H, 5.81%; N, 10.51%; S, 8.00%.

### Poly[sodium dimethyl(3-propyl)amide-4-methacrylamidobenzimide sulfonate] (X)

To a 100-mL polymerization ampule were added 2.0 g (5.11 mmol) of IX, 14 mg ( $5.0 \times 10^{-5}$  mol) ACVA, and 40 mL of double distilled water. The contents of the ampule were purged with nitrogen for 20 min and then subjected to three freeze-pump-thaw cycles. The ampule was sealed under vacuum, placed in a water bath, and shaken at  $60^\circ\text{C}$  for 24 h. The resulting viscous solution was transferred to a dialysis membrane with a molecular weight cutoff of 12,000 to 14,000 amu, and exhaustively dialyzed against distilled water. The polymer was isolated by lyophilization to give 1.85 g, a yield of 92.5%.

$^1\text{H}$  NMR ( $\text{D}_2\text{O}$ ):  $\delta$ 1.22,  $\underline{\text{C}}\text{H}_2-\text{C}$ ; 1.94,  $\text{C}-\underline{\text{C}}\text{H}_3$ ; 2.00,  $\text{CH}_2--\underline{\text{C}}\text{H}_2-\text{CH}_2$ ; 2.90,  $-\underline{\text{C}}\text{H}_2\text{SO}_3$ ; 3.43,  $\text{N}-\underline{\text{C}}\text{H}_3$ ; 3.93,  $\text{N}-\underline{\text{C}}\text{H}_2$ ; 7.31, 7.62, aromatic.

$^{13}\text{C}$  NMR ( $\text{D}_2\text{O}$ ):  $\delta$ 19.40,  $\text{CH}_2-\text{C}-\underline{\text{C}}\text{H}_3$  and  $\text{CH}_2\underline{\text{C}}\text{H}_2\text{CH}_2$ ; 46.84,  $\text{CH}_2\text{SO}_3$ ; 54.77,  $\text{N}-\underline{\text{C}}\text{H}_3$ ; 65.45,  $\text{N}-\underline{\text{C}}\text{H}_2$ ; 122.70, 128.99, 132.17, 139.84, aromatic; 170.20,  $\text{C}=\text{O}$  aminimide; 177.93,  $\text{C}=\text{O}$  amide.

Analysis. Calculated for  $\text{C}_{16}\text{H}_{22}\text{N}_3\text{O}_5\text{SNa}$ : C, 49.10%; H, 5.63%; N, 10.74%; S, 8.18%. Calculated for  $\text{C}_{16}\text{H}_{22}\text{N}_3\text{O}_5\text{SNa}(0.8 \text{H}_2\text{O})$ : C, 47.36%; H, 5.82%; N, 10.36%; S, 7.89%. Found: C, 47.32%; H, 6.36%; N, 10.48%; S, 7.81%.

## RESULTS AND DISCUSSION

### Monomer and Polymer Syntheses

The preparation of sodium dimethyl(3-propyl)amine-4-methacrylamidobenzimide sulfonate (IX, SDM) involved the 5-step process shown in Fig. 1. Compound V was prepared by modification of the procedure of Smith et al. [15]. This route was preferable to acylation through the

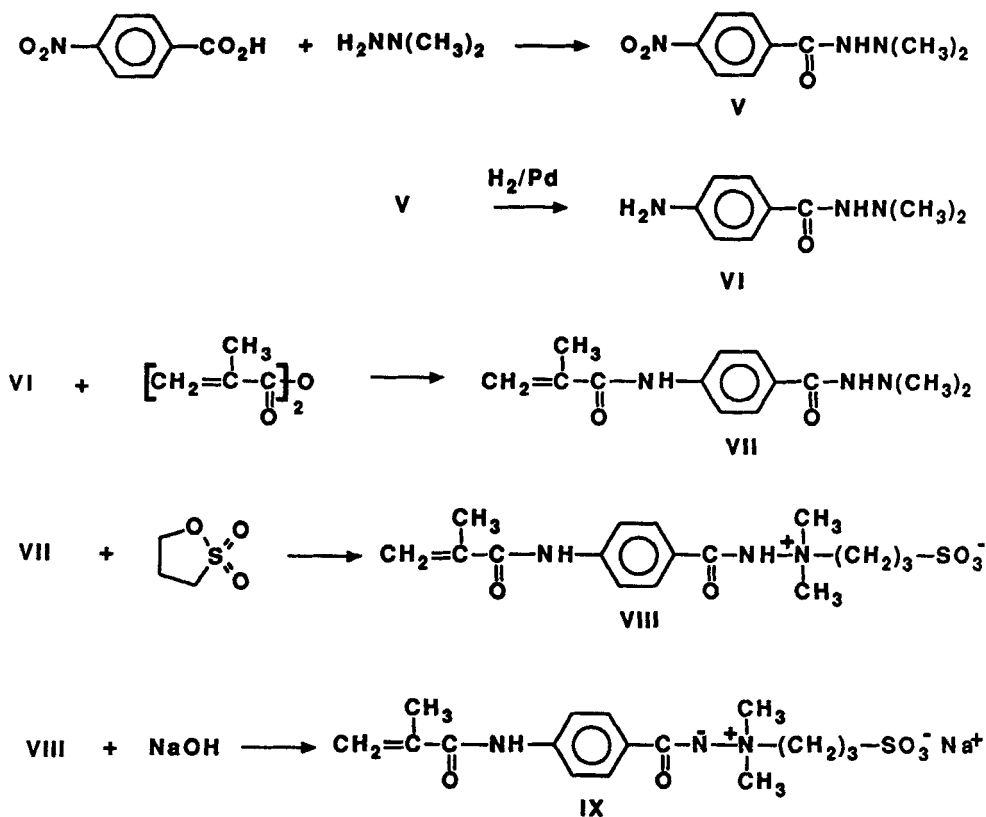


FIG. 1. Synthesis of sodium dimethyl(3-propyl)amine-4-methacryl-amido-benzamide sulfonate (SDM).

acid chloride since it was a 1-step process from the acid and was less likely to give a diacylated hydrazine [15]. This nitro-containing hydrazide was readily reduced to give VI at room temperature using Pd catalyst. Following the procedure of Bahr et al. [16], this material was treated with methacrylic anhydride to give VII, followed by treatment with 1,3-propanesultone to give the hydrazinium salt VIII. The final product, SDM, was obtained by titration of VIII with NaOH to a phenolphthalein endpoint. The polymerization of SDM in water using ACVA as initiator resulted in a highly viscous polymer solution. Following purification by dialysis, a hygroscopic white solid, PSDM, was obtained.



TABLE 1. Dilute Solution Viscosity Data for PSDM as a Function of Salt Concentration

NaCl concentration, mol/L	$[\eta]$ , dL/g	$k'$	$k' - k''$
0.1	1.61	0.22	0.43
0.5	1.01	0.32	0.51
1.0	0.77	0.34	0.51
2.0	0.63	0.32	0.53

### Dilute Solution Viscosity Behavior

Although the dilute solution viscosity behavior of many polyampholytes has been reported, no studies involving the dilute solution properties of polyaminimides have appeared in the literature. It has been reported that the aminimide functionality is a powerful water-solubilizing group when present in polymeric systems [10, 11]. However, recent data has shown that this is not the case for polymers with a significant degree of hydrophobicity [12, 13]. Furthermore, the limited data available on the effect of ionic strength on such systems suggest that these polymers can be extremely sensitive to salt solutions [14].

It was found that although PSDM was soluble in aqueous media, its intrinsic viscosity could not be determined due to the nonreproducibility of the capillary flow time observed in dilute solution. Although the reason for this is not clear, the shear sensitivity of more concentrated aqueous polyaminimide solutions has been reported previously [13, 14]. When PSDM was dissolved in aqueous salt solutions containing as little as 0.1 *N* NaCl, reproducible flow times were observed which allowed viscosity measurements to be made.

The dilute solution viscosity behavior of PSDM as a function of salt concentration was examined to observe how it would compare with similar studies on polyampholytic systems.

Table 1 shows the effect of ionic strength on the dilute solution viscosity behavior of PSDM. It is seen that the results are the same as would be expected for a polyelectrolyte, with the intrinsic viscosity decreasing with increasing salt concentration. The Huggins constants and  $k' - k''$  values are also reported in Table 1, and these reflect the random coil nature of this polymer in a good solvent system. These data suggest that

the dilute solution viscosity behavior of this polymer is dominated by the sulfonate group, leading to polyelectrolyte behavior, and that the aminimide group does not behave as a classical polyampholyte in this case.

### Light Scattering

To supplement the viscosity data, light scattering was used to study the molecular characteristics of PSDM as a function of salt concentration. The Zimm plots obtained for the material were typical, and a sample plot produced from light-scattering measurements on PSDM solutions in 0.5 *N* NaCl is shown in Fig. 2. From these data the molecular parameters for the polymer were determined and are shown in Table 2.

It is seen that although the weight-average molecular weight appears nearly constant, the radius of gyration decreases with increasing salt concentration. Again, this can be attributed to the contraction of the polymer chains which results from the screening effect of the added electrolyte. In addition, the behavior of the second virial coefficient corroborates these findings by showing a decrease with increasing salt concentration, reflecting an increase in polymer-polymer interactions as a result of chain contraction.

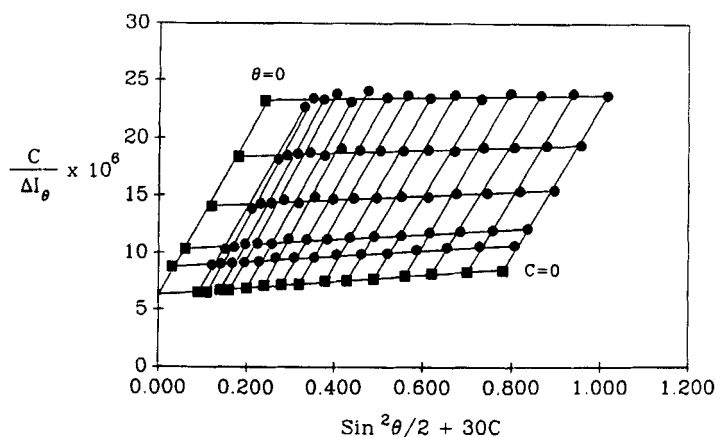


FIG. 2. Zimm plot for PSDM in 0.5 *N* NaCl.

TABLE 2. Molecular Parameters for PSDM in Aqueous NaCl

NaCl concentration, mol/L	$\bar{M}_w \times 10^{-6}$ g/mol	$A_2 \times 10^4$ mL·mol/g <sup>2</sup>	$R_g$ , nm
0.1	1.42	2.6	64.4
0.5	1.46	1.2	53.4
1.0	1.59	1.5	49.7
2.0	1.47	0.7	43.5

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