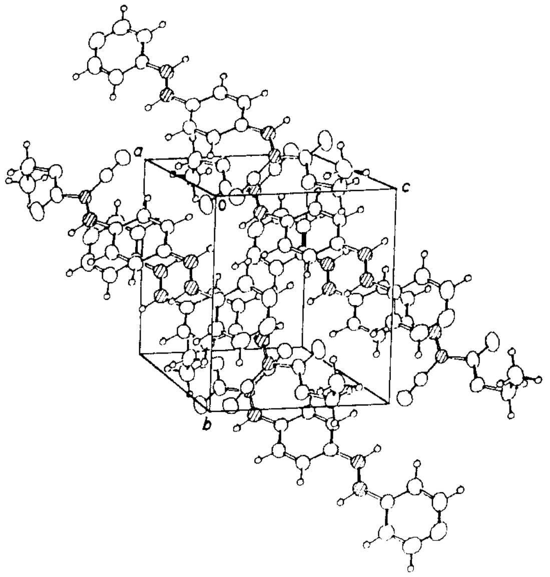

Figure 2. Crystal structure of 2. The atoms comprising the ethylenic groups are hatched for clarity.

chain. On the other hand, 5 consists of a hydrophilic and hydrophobic alternating structure in the polymer chain.

Acknowledgment. We are grateful to Dr. Sadao Sato of Sankyo Co., Ltd., and Dr. Kazuhide Kamiya of Takeda Chemical Industries, Ltd., for crystallographic analysis of 1 and 2.

Registry No. 1 (homopolymer), 100515-61-5; 2 (homopolymer), 100515-63-7; 4, 100515-64-8.

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(9) Dimer 4: mp 189.5–192.0 °C; ^{1}H NMR (CDCl$_3$, 90 MHz) δ 0.95 (t, 6H), 4.03 (q, 4 H), 5.13 (s, 2 H), 6.9–7.7 (m, 16 H), 8.55 (d, 2 H); mass spectrum, m/e 608 (P), 304 (P/2, symmetric cleavage of the cyclobutane ring), but no trace of asymmetric cleavage of the cyclobutane ring.
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Configurational Diffusion in Semidilute Solutions†

The viscoelastic properties of entangled polymeric fluids are inextricably linked to the dynamics of single chains, yet there are relatively few measurements of these single-chain dynamics, since most experimental methods are sensitive to the relatively rapid collective fluctuations of many-chain systems. For this reason, experimental methods which focus on single-chain properties often employ some sort of labeling technique. In this Communication we report measurements of the single-chain dynamics of polystyrene (PS) in a poly(vinyl methyl ether) (PVME)/toluene host. In these experiments the semidilute PVME solutions are index matched by the solvent (and hence give no measurable scattering), whereas the dilute high molecular weight PS scatters very strongly. Using this "optically" labeled-chain technique[1] we were able to observe the diffusion of long-wavelength, single-chain modes in semidilute solutions and thus test current ideas about the semidilute screening of hydrodynamic interactions.

Although detailed theories exist for the internal modes of polymer chains,[2,3] it is convenient here to give a description in terms of scaling ideas.[4] The important parameter in a dynamic light scattering experiment is q, the momentum transfer. In terms of λ, the wavelength in the scattering volume, and θ, the scattering angle, $q = 4\pi \sin(\theta/2)/\lambda$, which has the dimensions of inverse length. A dynamic light scattering experiment measures the relaxation times of fluctuations which occur on a length scale $1/q$. In the simplest experiments these fluctuations may be due to translational diffusion of a narrow fraction polymer, in which case a single relaxation time is measured, $1/D_tq^2$, which is the mean time for a polymer with diffusion constant D_t to diffuse a distance $1/q$. However, when the length scale $1/q$ is smaller than R, the radius of the polymer, internal degrees of freedom contribute to the observed relaxation processes if the polymer is in a fluctuating (nonglobule) state. In this fluctuating state each of the normal modes of a polymer has a relaxation time which is proportional to R^2/D_t times some function of the mode index. So if we consider a transformation of scale, $R \rightarrow R'$, the relaxation time of the pth mode will become $\tau_p(R') = \tau_p(R)(R'/R)^{2+\alpha}$, where α is the diffusion exponent defined by $D_t \sim R^{-\alpha}$. Now a mode of spatial extent R will have fluctuations, δR, of magnitude R, so we may replace R by $1/q$ to obtain $\tau_q \sim 1/q^{2+\alpha}$ for the q dependence of the relaxation time. The Rayleigh line width, or mean relaxation rate, Γ, is just $1/\tau_q \sim q^{2+\alpha}$.

In dilute solution there are two possibilities for the diffusion exponent α, corresponding to strong or weak hydrodynamic interactions. If hydrodynamic interactions are strong the diffusion is Stokes–Einstein, giving $(T/\eta_0)R^{2-d}$ for the diffusion coefficient in terms of the temperature, solvent viscosity, and dimension of space, respectively. In the regime $qR \gg 1$ this gives

$$\Gamma \sim \frac{T}{\eta_0}q^d \qquad (1)$$

which we simply call Zimm behavior.[5] Zimm dynamics have been observed by several investigators.[6-8] On the other hand, if hydrodynamic interactions are weak the diffusion coefficient is T/f, where $f = Nf_0$ is the friction factor for the chain of N monomers and f_0 is the monomeric friction factor. In this case $D_t \sim R^D$, where D is the fractal dimension, and

$$\Gamma \sim \frac{T}{f_0}q^{2+D} \qquad (2)$$

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This we will refer to as Rouse behavior.[9] At the Θ condition $D = 2$ and $\Gamma \sim q^4$, which is markedly different from the Zimm model where $\Gamma \sim q^3$ in three dimensions.

The importance of the Zimm model is clear enough, since hydrodynamic interactions are typically very strong in dilute solutions. The significance of the Rouse model is more subtle, however, because it depends on the screening of hydrodynamic interactions in semidilute soutions. In dilute solutions hydrodynamic interactions are described by the Oseen tensor, T_0, whose trace falls off like $1/\eta_0 r$. In semidilute solutions it has been conjectured that this hydrodynamic interaction is screened by intervening polymer chains, leading to $T = T_0 e^{-r/\xi_h}$, where ξ_h is called the hydrodynamic screening length.[10] The simplest scaling theories assume that the hydrodynamic screening length is proportional to the static correlation length, ξ, which is an inverse power of the concentration. Thus on length scales greater than ξ both excluded volume and hydrodynamic interactions are screened[10] and it is thought that the static properties of a single chain can be described by the Rouse model.

Under these assumptions the full q dependence of Γ can be divided into three regimes; a translation diffusion regime ($qR \ll 1$) where $\Gamma \sim q^2 D_t$, a Rouse regime ($q\xi \ll 1 \ll qR$) where $\Gamma \sim (T/f_0)q^4$, and a Zimm regime ($q\xi \gg 1$) where $\Gamma \sim (T/\eta_0)q^3$. Well into the semidilute regime, the q^4–q^3 crossover will occur at sufficiently small length scales that it will not be observable in a light scattering experiment, and so an extensive Rouse regime should be observed if these hydrodynamic screening ideas are correct. However, it should be pointed out that doubts have been raised as to the validity of the hydrodynamic screening concept.[11]

Experimental Procedure. A total of eight ternary solutions were used in this experiment, with PVME concentrations of 0%, 5%, 10%, 15%, 20%, 25%, 32.5%, and 40%. The PS concentration was fixed at 0.25 mg/mL in all solutions. The PVME was a polydisperse MW = 110 000 sample obtained from GAF (Gantrez M-556), whereas the PS was a narrow fraction ($M_w/M_n = 1.3$) MW = 48 000 000 sample obtained from Pressure Chemical Co. Both the PS and PVME fractions were exhaustively centrifuged (ca. 70 h at 35000*g* for the PVME) to remove dust before mixing to make the final solutions. The toluene was filtered with 0.1-μm Millipore filters. To avoid stray light problems, scattering measurements were made in very large diameter (8 cm) precision scattering cells. Intensity autocorrelation functions were analyzed by the method of cumulants. Viscosity measurements on the semidilute solutions were made with a series of Cannon–Ubbelohde viscometers.

Results. For the purpose of establishing a firm experimental base line, a dilute solution of PS was run without any PVME. In Figure 1 the data are dividied by $q_3 k_B T/\eta_0$ to illustrate the third-power dependence expected for dilute solutions. The asymptotic 0.067 prefactor is smaller than either the preaveraged (0.071) or nonpreaveraged (0.079) hydrodynamic interaction models of Benmouna and Ackasu[12,13] but is in somewhat better agreement with the recent $\mathcal{O}(\epsilon)$ renormalization calculation of Lee et al.,[14] which gives 0.069 for this universal prefactor.

Figure 2 shows the data for the full range of isorefractive samples. The lines through the data are q^3 fits so that small deviations from Zimm behavior are readily discerned. At high concentrations the relaxations became too slow (>10 s) to observe at small q, and for this reason the small-q data cutoff moves to larger q at higher concentrations. It is evident that within experimental error there is no evidence for Rouse behavior (hydrodynamic screening) in any of these data. Instead, the observed q^3 behavior of the data is consistent with the Zimm model, which implies that on these length scales the hydrodynamic interactions are falling off like $1/\eta_p r$, where η_p is the *solution* viscosity.

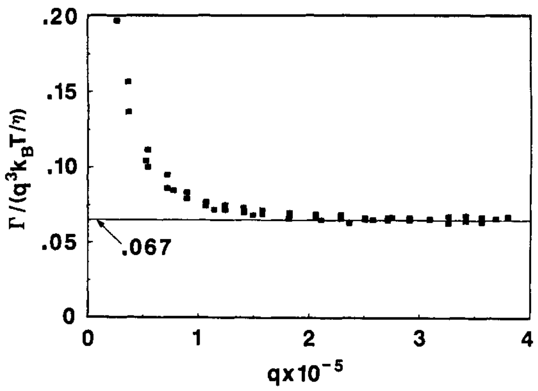

Figure 1. The first cumulant for the 48 000 000 polystyrene sample in toluene is divided by the universal, strong hydrodynamic interactions result, $\Gamma \sim q^3 k_B T/\eta$. At large q good agreement is found with this third-power theory, and the universal prefactor is estimated to be ~0.067.

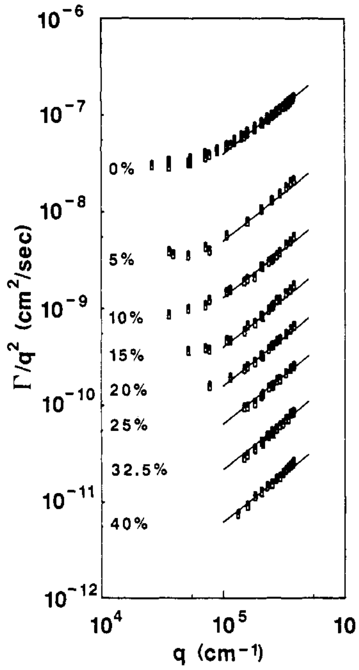

Figure 2. The relaxation rate data for the ternary PS/PVME/toluene solutions is shown for various weight fractions of PVME. The lines are not fits to the data but merely indicate q^3 behavior, so that deviations from this behavior can be discerned.

To substantiate this form of the Oseen interaction, the data were scaled by the temperature and viscosity. The scaled data in Figure 3 fall very nearly on the base-line curve, indicating that the primary effect of the semidilute solution on the dynamics is an increase in viscosity and that the data for PVME concentrations less than 25% are described by

$$\Gamma \sim (T/\eta_p)q^3 \tag{3}$$

The failure to observe Rouse dynamics for the internal modes was foreshadowed by the theoretical work of Oono et al.[11] on mutual diffusion and the complementary experimental work of Wiltzius et al.[15] The absence of screening effects on the long length scales observed here is consistent with these previous results.

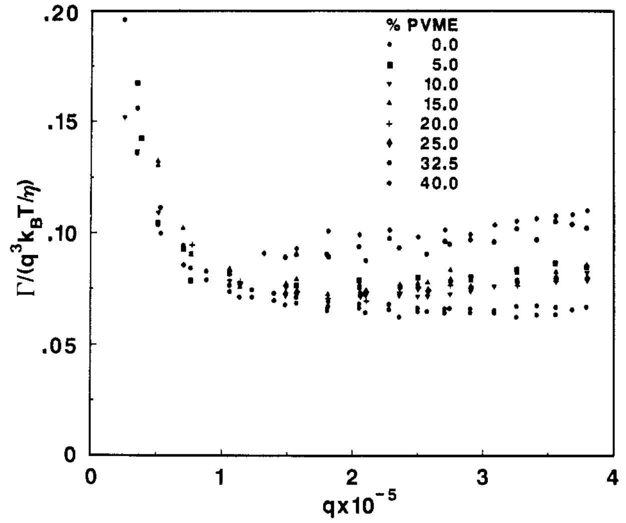

Figure 3. The data from Figure 2 are scaled by Zimm result, modified by replacing the solvent viscosity by the solution viscosity. Up to 25% the data collapse is within experimental error, but at 32.5% and 40% PVME significant deviations from this mean field behavior are observed.

Now it is quite reasonable that on very short length scales, where $q\xi \gg 1$, Zimm behavior will again be observed, but with hydrodynamic interactions moderated by the solvent viscosity: $\Gamma \sim (T/\eta_0)q^3$. Since the solution viscosity can quite easily be 10^6 times the solvent viscosity, it is clear that Γ will have to increase more steeply than q^3 in some intermediate length scale regime. This implies an additional length scale in semidilute solutions, λ, which defines a transition region $\xi \ll q^{-1} \ll \lambda$ in which Γ/q^3 is an increasing function of q such that $\Gamma(\xi^{-}1)/\Gamma(\lambda^{-1}) = \eta_p/\eta_0$. Two obvious questions are what is the length scale λ and how does Γ depend on q in this transition regime?

Zimm/Reptation Crossover. Recent experimental work on self-diffusion has demonstrated an extensive Stokes–Einstein diffusion (Zimm) regime in bimodal semidilute solutions if the labeled species (PS) is much larger than the isorefractive species.[16] And of course if the translational mode is described by Zimm dynamics then the longest wavelength modes must also be described by the Zimm model so $\Gamma \sim (T/\eta_p)q^3$. From this it is clear that the observation of a new Zimm regime in semidilute solutions was adumbrated by previous self-diffusion data. Furthermore, we can use the concept of a Stokes–Einstein (SE)/reptation crossover, postulated for self-diffusion data,[1] to create a logically consistent scaling theory for the configurational dynamics in semidilute solution.

This SE/reptation crossover is couched in terms of a dynamical crossover concentration ϕ_r, where $\phi_r > \phi^*$, the semidilute crossover. For $\phi < \phi_r$ the labeled chain is assumed to Stokes–Einstein diffuse; for $\phi > \phi_r$ the labeled chain will reptate. For example, in terms of the matrix molecular weight, P, and the labeled chain molecular weight, N, the good-solvent crossover[1] is

$$\phi_r = (N^{1/2}/P)^{8/5} \qquad (4)$$

where $N > P$. It is observed that in the unimodal limit ($N = P$), $\phi_r \sim N^{-4/5} \sim \phi^*$ and reptation behavior is expected throughout the semidilute regime. The scaling behavior of the self-diffusion coefficient is then described by $D_t = D_{SE}f(\phi/\phi_r)$, where D_{SE} is the Stokes–Einstein diffusion coefficient, $T/6\pi\eta_pR$, and $f(x)$ is a scaling function with the property $f(x \ll 1) = 1$. Now if $\phi \gg \phi_r$ we require that the diffusion coefficient be independent of the matrix molecular weight P. This constraint gives $f(x \gg 1) \sim x^{15/8}$ and

$$D_t = D_{SE}(\phi/\phi_r)^{15/8} \sim N^{-2}\phi^{-7/4} \qquad \phi \gg \phi_r \qquad (5)$$

which is the familiar result for the reptation diffusion coefficient in good-solvent semidilute solutions.

The Stokes–Einstein/reptation crossover implies a Zimm/reptation crossover for the internal modes. To see this, imagine a very high molecular weight labeled chain ($N \gg P$) in a semidilute solution at concentration $\phi^* \ll \phi \ll \phi_r$. This chain will undergo SE diffusion and its longest wavelength internal modes will be Zimm modes. Now suppose that the molecular weight of the labeled chain is decreased, at constant matrix concentration, until $\phi_r = \phi$. At this point the labeled chain will begin to reptate, and the long-wavelength internal modes will cross over from being solution viscosity moderated to being independent of the matrix molecular weight. And at just this point the radius of the labeled chain will be equal to the length scale, λ, where the internal modes cross over from Zimm to reptation dynamics. Thus

$$\lambda(\phi,P)_{\phi=\phi_r} = R(\phi,N)_{\phi=\phi_r} \qquad (6)$$

where R is a function of N and ϕ alone and λ is a function of ϕ and P alone. In good solvents $R(\phi,N) \sim N^{1/2}\phi^{-1/8}$, so using eq 4 for ϕ_r gives $\lambda \sim \phi^{1/2}P$. In Θ solvents $\phi_r \sim N^{1/2}/P$ and $R \sim N^{1/2}$, so $\lambda \sim \phi P$. In terms of the static correlation length and the solvent and solution viscosities it is observed that we can write a very simple, universal (good and Θ solvent) relation for the Zimm/reptation crossover length:

$$\lambda = \xi(\eta_p/\eta_0)^{1/3} \qquad (7)$$

From this crossover length the scaling behavior is easily determined. We write

$$\Gamma = \Gamma_{\text{Zimm}}h(q\lambda) \qquad (8)$$

where $\Gamma_{\text{Zimm}} \sim (T/\eta_p)q^3$ and $h(x \ll 1) = 1$. For $q\lambda \gg 1$ we require the relaxation rate to become independent of the matrix molecular weight. Using eq 7 then gives $h(x \gg 1) \sim x^3$ and

$$\Gamma \sim (\mathrm{T}/\eta_0)q^6\xi^3 \qquad q\lambda \gg 1 \gg q\xi \qquad (9)$$

a result which is valid for both good and Θ solvents, since these reptation modes depend on the correlation length alone. This q^6 result, which comes from very simple scaling considerations, has already been identified by de Gennes[17] in an explicit calculation for the reptating chain in a melt—eq 9 simply extends this result into the semidilute regime.

At this point we must be certain that eq 9 is consistent with the appearance of a second Zimm regime at length scales small compared to the hydrodynamic screening length. To see this we multiply eq 8 by a scaling function ($k(q\xi)$ and require that for $q\xi \gg 1$ Γ is independent of the correlation length ξ. This is only ture if $k(x \gg 1) \sim x^{-3}$, which gives

$$\Gamma \sim (T/\eta_0)q^3 \qquad q\xi \gg 1 \qquad (10)$$

Thus we have a scaling theory of the internal modes in semidilute soltuion which smoothly and consistently connects the observed small-q *solution* viscosity moderated Zimm regime with the large-q *solvent* viscosity moderated Zimm regime. These results are diagramed in Figure 4.

A comprehesive scaling theory for the relaxation rate can be given by a product of crossover functions for the regimes we have described:

$$\Gamma = q^2D_{SE}f(\phi/\phi_r)g(qR)h(q\lambda)k(q\xi) \qquad (11)$$

where for values of their argument much smaller than

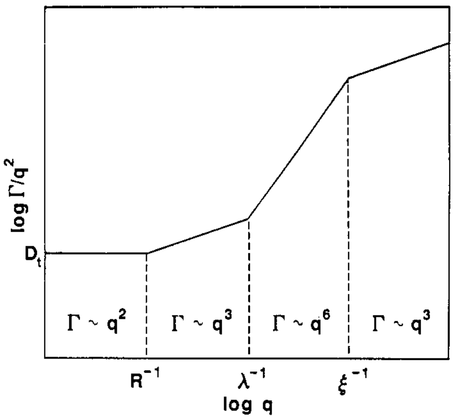

Figure 4. The scaling prediction for the relaxation rate in bimodal solutions gives three distinct regimes for $qR > 1$. The first is a third-power mean field Zimm behavior, where the solution viscosity moderates the relaxation times. The second is a sixth-power reptation regime, and the third is the small length scale Zimm regime, where relaxation processes are moderated by the solvent viscosity.

Table I

ϕ	R	λ	ξ	γ	β
$N^{1/2}/P$	$N^{1/2}$	$\xi(\eta_p/\eta_0)^{1/3}$	ϕ^{-1}	3	3
$(N^{1/2}/P)^{8/5}$	$N^{1/2}\phi^{-1/8}$	$\xi(\eta_P/\eta_0)^{1/3}$	$\phi^{-3/4}$	15/8	3
$(N^{2/5}/P)^{4/3}$	$N^{3/5}$	$\xi(\eta_p/\eta_0)^{1/2}$	$\phi^{-3/4}$	9/4	2

unity each of these functions is a simple constant.The Stokes–Einstein/reptation crossover is described by the function $f(x \gg 1) \sim x^{\gamma}$. The self-diffusion/configurational diffusion crossover is given by $g(x \gg 1) \sim x$, a well-known scaling result. The Zimm/reptation crossover is described by $h(x \gg 1) \sim x^{\beta}$, and the reptation/Zimm crossover is given by $k(x \gg 1) \sim x^{-\beta}$. The exponents and crossovers are in Table I for good and Θ solvents and for good-solvent ternary systems where excluded volume interactions are not necessarily screened.

The concept of a second Zimm regime is not new but was originally suggested by Richter et al.[18] to interpret inelastic neutron scattering data from unimodal polymer solutions. The "incomplete" hydrodynamic screening model they proposed, however, predicts a Zimm/Rouse/Zimm crossover, whereas the current model, which more fully accounts for entaglement effects, is Zimm/reptation/Zimm. Further experimental work is needed to distinguish between these possibilities.

In conclusion, dynamic light scattering measurements of configurational diffusion of optically labeled chains in semidilute solution are shown to be described by a modified Zimm model were the solution viscosity replaces the solvent viscosity. Based on this observation, a scaling theory is given for the internal dynamics in semidilute soltuion.

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Registry No. PS, 9003-53-6; PVME, 9003-09-2; toluene, 108-88-3.

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Thermal Decomposition of Poly(γ-methyl L-glutamate)

Recently our research efforts have focused on the thermal decomposition behavior of the side chains of polymers.[1] The side chain of poly(γ-methyl L-glutamate) (PMLG) is expected to influence the thermal decomposition more than the skeletal chain stabilized by hydrogen bonds.[2–5] There are a few studies[6–8] on the thermal decomposition of poly(glutamic acid) esters, but the reaction mechanism is hardly discussed. In the present communication, we propose a thermal decomposition mechanism for PMLG and give some supporting evidence for it.

The PMLG samples were cast from 1.2-dichloroethane. The solution of PMLG (Ajicoat A-2000) was kindly supplied by Ajinomoto Inc. Methyl *n*-butyrate and γ-methyl L-glutamate (MLG), used to elucidate the thermal decomposition mechanism of PMLG, were synthesized in the usual way.[9] The investigation of the thermal decomposition in a flow of helium was carried out by pyrolysis gas chromatography (GC)–mass spectrometry (MS). Details of pyrolysis GC–MS are given elsewhere.[10]

Figure 1 and Table I show results of pyrolysis GC–MS on PMLG at 570 and 770 K. Products 1, 4, 5, and 6 are identified as carbon dioxide,[11] methanol, methyl acetate, and methyl acrylate, respectively. Product 2 is perhaps a mixture of light gases such as methane (m/z = 16), ethene (m/z = 28), ethane (m/z = 30), propene (m/z = 42), and propane (m/z = 44). Water appears above 58 min in pyrogram I; the GC column filler used in the present experiment, DC-550, may have little effect on water. Products 7–10 are unknown, but we regard them as decomposition products due to radical scission of the skeletal chain, since they evolve at 770 K but do not evolve at 570 K. Product 3 was identified as 1,2-dichloroethane, the solvent for Ajicoat A-2000.

Generally, esters undergo nucleophilic substitution at their acyl carbons by reacting with primary or secondary amide.[12,13] Pyrolysis GC–MS data on MLG are shown in Figure 2. The major decomposition products are carbon dioxide, methanol, and 2-pyrrolidone. Liberation of

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