

## Experiment 32

### Molecular Weight and Monomer Linkage Properties of Poly(vinyl alcohol)

**Objective** To determine the viscosity-average molecular weight of poly(vinyl alcohol) (PVOH) and the fraction of "head-to-head" monomer linkages in the polymer.

**Introduction** One of the fundamental molecular properties used to characterize a polymer is its molecular weight. Many of the physical characteristics of polymeric materials can be associated with the shape and weight distribution of the polymer. Some experimental techniques used to obtain this information are viscosity, osmotic pressure, and light scattering measurements. These measurements are made not on the polymer itself but on solutions containing the dissolved polymer. Because the viscosity and osmotic pressure of these solutions depend systematically on the concentration of polymer, they are called *colligative properties*.

This experiment deals with viscosity measurements of solutions of poly(vinyl alcohol) in water. This technique is very straightforward and does not require specialized equipment. We begin by reviewing the fundamentals of fluid viscosity. If a liquid undergoes laminar or streamline flow through a cylindrical tube, the differential equation that describes the mass transport in terms of the flow rate through a cylindrical tube is known as the Poiseuille law [named after Poiseuille (1844)], in whose honor the cgs unit of viscosity the poise ( $1 \text{ g cm}^{-1} \text{ s}^{-1}$ ) is named. This equation is

$$\frac{dV}{dt} = \frac{\pi r^4 (P_0 - P')}{8\eta(z' - z_0)}, \quad (1)$$

in which  $dV$  is the volume of fluid (liquid or gas) transported in a time  $dt$  through a straight, cylindrical tube of radius  $r$ . The fluid is under a pressure difference  $(P_0 - P')$  that extends over the length of the tube  $(z_0 - z')$ . If the pressure gradient is produced by the gravitational force acting on the fluid (the fluid flowing, for simplicity, in a downward vertical direction),  $P_0 - P'$  is proportional to the density of the fluid,  $\rho$ . Hence for an arbitrary but constant volume of fluid, equation (1) yields (after integration)

$$V = \frac{t(\pi r^4 g \rho)}{8\eta}, \quad (2)$$

where  $g$  is the gravitational constant, and  $\rho$  is the bulk density of the fluid. We can rearrange equation (2) to give an expression for the fluid viscosity:

$$\eta = \frac{\pi r^4 g}{8V} \rho t = c \rho t \quad (\text{g cm}^{-1} \text{s}^{-1}), \quad (3)$$

where the constant  $c$  depends on the characteristics of the measuring device used, that is, the tube bore radius,  $r$ , and the total volume of fluid flowing in time  $t$ . Equation (3) is the basis for the measurement of liquid viscosities with an apparatus called a *viscometer*.<sup>1</sup> The constant  $c$  is determined by calibrating the viscometer with a liquid of a known viscosity, in this case water. The time  $t$  required for water to flow through the viscometer is measured. Knowledge of the water density (at the particular temperature) then allows  $c$  to be determined. For highly precise determinations of viscosity a kinetic-energy correction term must be subtracted from equation (3). Because this correction is usually less than 1% of the quantity  $cpt$ , it can be ignored.

We seek a relationship between the viscosity of a solution containing a dissolved high molecular weight polymer and some molecular weight property of the polymer itself. A significant contribution to this field was made by Einstein (1906), who showed that the *fractional change* in the viscosity of a solution—relative to the pure solvent—is related to the fraction of the total volume of solution occupied by the solute, in this case the polymer. It is assumed that the solute has simple spherical geometry. Thus the relevant equation is

$$\frac{\eta - \eta_0}{\eta_0} \equiv \eta_{sp} = \frac{Cv}{V}, \quad (4)$$

where  $\eta$  and  $\eta_0$  are the respective viscosities of the solution and the pure solvent. The term  $\eta_{sp}$  [which is dimensionless and positive (because  $\eta > \eta_0$ )] is defined as the *specific viscosity* and is proportional to the ratio of the solute volume,  $v$ , to that of the solution,  $V$ . Thus  $\eta_{sp}$  is a *colligative property* because its value depends on the amount of polymer in solution, i.e., its concentration. The constant  $C$  has a theoretical value of 5/2 (for spherical solutes).

For a solution containing  $N$  spherical solute molecules each of radius  $R$ , equation (4) becomes (using  $C = 5/2$  and  $v = 4\pi R^3/3$ )

$$\eta_{sp} = \frac{10\pi R^3 c_m}{3m}. \quad (5)$$

Equation (5) can more conveniently be expressed in terms of the mass concentration of the solute in the solution,  $c_m$  (defined as *grams per milliliter*), and the *molecular mass* of the solute,  $m$ , as (noting that  $c_m = N_A m/V$ )

$$\eta_{sp} = \frac{10\pi R^3 c_m}{3m}. \quad (6)$$

The colligative nature of  $\eta_{sp}$  is explicit in equation (6) because of the  $c_m$  dependence.

<sup>1</sup> See Experiment 17 for a description of the Ostwald viscometer.

Another quantity that will prove to be very useful is the *intrinsic viscosity*,  $[\eta]$  (sometimes referred to as the Staudinger index):

$$[\eta] \equiv \lim_{c_m \rightarrow 0} \left( \frac{\eta_{sp}}{c_m} \right) = \frac{10\pi R^3}{3m} \quad (\text{cm}^3 \text{ g}^{-1}). \quad (7)$$

The limit of infinite dilution ( $c_m \rightarrow 0$ ) is required to define a viscosity property that is intrinsic to the solute (in the given solvent), i.e., independent of the concentration. This approach essentially eliminates problems caused by the fact that the bulk properties of the polymer solution vary with concentration.

The limiting condition defined in equation (7) is needed to define  $[\eta]$  because the reduced specific viscosity (i.e.,  $\eta_{sp}/c_m$ ) depends on the polymer concentration due to shear forces between the dissolved macromolecule and the solvent medium. This concentration dependence is observed to follow the relation

$$\frac{\eta_{sp}}{c_m} = [\eta] + k[\eta]^2 c_m + k' c_m^2, \quad (8)$$

where  $k$ , known as the Huggins constant, has a value of about 2 for rigid, uncharged spheres, and about 0.35 for flexible polymers in a "good" solvent.<sup>1</sup> The higher-order term in  $c_m$  in equation (8) can often be neglected. From equation (6) we can see that the ratio  $\eta_{sp}/c_m$  is not a colligative quantity; it is equal to an intrinsic property of the solute itself, namely,  $10\pi R^3/3m$ . The intrinsic viscosity has dimensions of  $\text{cm}^3 \text{ g}^{-1}$  and is perhaps analogous to a molar volume. Because both  $\eta_{sp}$  and  $c_m$  are experimental quantities, equation (8) can be used to obtain  $[\eta]$  by extrapolation to zero concentration.

It is interesting to note that if  $R$  is known, equation (7) can be used to determine  $m$ , the molecular mass of the solute, from  $[\eta]$ . Furthermore, the molecular weight of the solute divided by its mass provides Avogadro's number, which suggests an experimental approach for obtaining this fundamental constant. Conversely, if  $m$  is known, a value of the solute radius,  $R$ , can be determined from  $[\eta]$ . Following this reasoning, Einstein was able to obtain a satisfactory value of Avogadro's number as well as the molecular radii of carbohydrates. Few other direct experimental methods can be used to measure Avogadro's number.

Intrinsic viscosity measurements can be applied to a high molecular weight polymer to obtain its (spherical) radius, or its molecular weight. Remember that the value of  $R$  in equations (5) to (7) denotes the *effective* radius of the solute, because the solute may not be exactly spherical, and in addition, the solute may be associated with a (possibly large) number of solvent molecules. This effective radius is referred to as the *hydrodynamic radius* and can, in principle, vary for a given solute from solvent to solvent depending on what shape the solute adopts in a particular solvent, that is, the extent to which solvent molecules penetrate or stick to the solute.

Another complication that can be anticipated in applying equation (7) to molecules is that the assumption of spherical geometry may be invalid in some cases. Deviations from spherical geometry would manifest themselves theoretically in that  $C$  [in equation (4)] would have values

other than 5/2. In principle, this can be accounted for if the correct *solvated* molecular shape (oblate or prolate spheroid, oblong, etc.) is known.

A more serious problem encountered in dealing with high polymers is the fact that these systems consist of polymers of various chain lengths. Thus these are not homogeneous solutes (monodisperse) but have a distribution of molecular weights and are called *polydisperse*. The degree to which a polymer is polydisperse depends on the conditions under which it is synthesized. Although in theory a polydisperse polymer can be fractionated into groups that have a more narrow molecular weight distribution (nearly monodisperse), this is often a long and tedious process. Measurements on raw, polydisperse polymer solutions provide information that is averaged over the molecular weights (and sizes) of the polymer. Such is the case in viscosity measurements.

In order to apply equation (7) to a system that is composed of a high molecular weight polydisperse polymer, a statistically averaged form of the molecular "radius" has to be used. In this experiment, the polymer studied—like many others—is a *linear* chain system, which means that the macromolecule is formed from a large number of monomer units in such a way that monomers add to the developing polymer chain without branching. Although the molecule is called a "linear" chain, its geometry surely does not resemble a straight-line assembly of monomer units. Rather, it is coiled up and adopts an overall shape that is approximately spherical. It may be thought to resemble a loosely tangled ball of yarn. See Figure 1.

This so-called random coil is not a static structure but continually undergoes contortional motion as the different segments go through various conformational transitions. On a time-averaged basis, however, we can define a statistical radius called the *radius of gyration*,  $R_g$ . This characteristic property is expressed quantitatively as

$$R_g = \left( \frac{I}{m} \right)^{1/2}, \quad (9)$$

where  $m$  is the molecular mass and  $I$  is the moment of inertia, a second-order moment defined as

$$I = \sum_i m_i r_i^2, \quad (10)$$

in which the sum is over all the point masses,  $m_i$  (atoms), of the molecule, and  $r_i$  is the distance to the  $i$ th atom from the center of mass. We can calculate  $I$  only if we adopt some (static) structural model of the polymer. If we substitute the radius of gyration for the unique-valued radius  $R$  in equation (7), we have

$$[\eta] = \frac{N_A 10 \pi R_g^3}{3 M} \quad (\text{cm}^3 \text{g}^{-1}), \quad (11)$$

where  $M$ , the molecular weight (along with  $N_A$ , Avogadro's number), replaces the molecular mass,  $m$ .

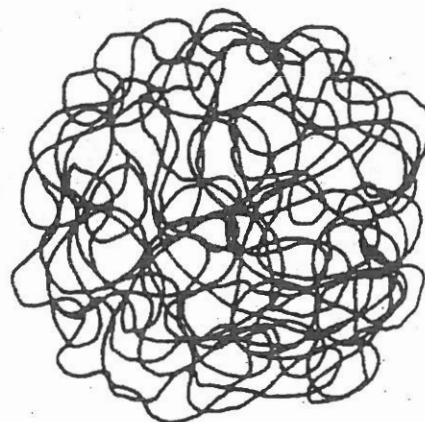


Figure 1. Schematic diagram of a linear chain polymer in a random-coil configuration.

If we now assume that the linear chain polymer is constructed of  $N$  monomer units linked together in such a way that *each* link is rotationally flexible, or unhindered (freely jointed chain); it can be demonstrated that the radius of gyration is proportional to the square root of the number of links in the polymer chain; thus  $R_g \propto N^{1/2}$ . This important conclusion is developed through the application of statistical mechanics to polymers.<sup>2</sup> Thus

$$[\eta] = K'M^{1/2}, \quad (12)$$

where  $K'$  is a proportionality constant that contains the conversion factor between  $R_g$  and  $M^{1/2}$ . Equation (12) is of particular importance in this experiment. It relates a measurable quantity, the intrinsic viscosity, to the desired molecular weight of the polymer. Although the square root dependence of  $[\eta]$  on molecular weight is actually observed for some monodisperse polymer solutions, many others deviate from equation (12). The reasons for these discrepancies have to do with the nature of solvation and the effects brought about by solvent association on the structure of the polymer solute. Thus the molecules of a "good" solvent enter into the polymer coils to maximize solvolytic associations, and the polymer expands. Looked at another way, the polymer "swells" out into the solvent. In a "poor" solvent, on the other hand, the polymer knots up into itself and avoids the interactions with the solvent molecules. As you might expect, the solubility of a given polymer is larger in a good solvent than in a poor one. See Figure 2.

Another important consideration we have ignored thus far in the discussion is called the "excluded volume." This is the effective space that is inaccessible to a random coil or freely jointed chain. This volume cannot be occupied because the segments of the polymer avoid each other as they become too close during the twisting and turning that the polymer undergoes.

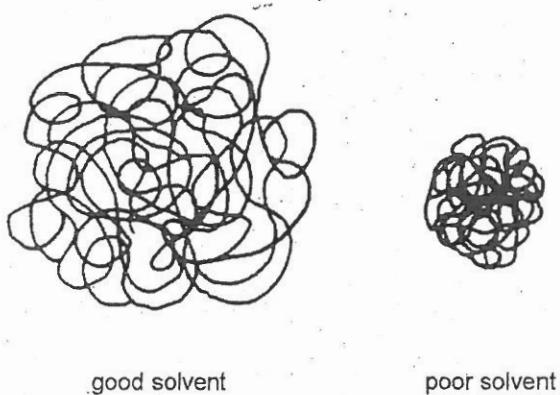


Figure 2. Schematic diagrams of a random-coil polymer in a "good" solvent (left) and a "poor" solvent (right).

A more general relationship between the intrinsic viscosity and molecular weight is provided by the Mark-Houwink equation:

$$[\eta] = KM^a \quad (\text{cm}^3 \text{ g}^{-1}), \quad (13)$$

where  $K$  and  $a$  are parameters that depend on the particular polymer, the solvent medium, and the temperature. Typically,  $a$  ranges between 0.5 and 0.8 [see equation (12)]. The Mark-Houwink parameters are obtained from log-log plots of  $[\eta]$  vs.  $M$  for a series of monodisperse polymers. Agreement between  $K$  and  $a$  values obtained by different workers is often apparently poor.<sup>3</sup> For example, for PVOH in water at 25°C, the following parameters shown in Table 1 have been reported:

Table 1. Mark-Houwink parameters for PVOH in water at 25°C.

$K$ ( $\text{cm}^3 \text{ g}^{-1}$ )	$a$	Molecular Weight Range	Note*
0.020	0.76	$(0.6 - 2.1) \times 10^4$	4
0.30	0.50	$(0.9 - 17) \times 10^4$	5
0.14	0.60	$(1 - 7) \times 10^4$	6

\* Notes appear at end of experiment.

We can obtain the molecular weight in explicit form from equation (13):

$$M_v = \left( \frac{1}{K} \right)^{1/a} [\eta]^{1/a} . \quad (14)$$

This expression (in which  $[\eta]$  has dimensions of  $\text{cm}^3 \text{ g}^{-1}$ ) is the computational basis of the experiment; it can be applied to polydisperse PVOH (which is used in this experiment), but the molecular weight obtained from equation (14) is a *viscosity-average* molecular weight,  $M_v$ . Statistically this quantity is different from the *number-average* molecular weight,  $M_n$ , which is obtained, for example, from osmotic pressure measurements.  $M_n$  is defined as

$$M_n = \sum_i f_i M_i , \quad (15)$$

where  $f_i$  is the fraction of polymers having molecular weight  $M_i$ ; the sum in equation (15) extends (in theory) from 0 to  $\infty$ .

Flory and others<sup>7</sup> have shown that the relationship between  $M_v$  and  $M_n$  is

$$\frac{M_v}{M_n} = [(1 + a)\Gamma(1 + a)]^{1/a} , \quad (16)$$

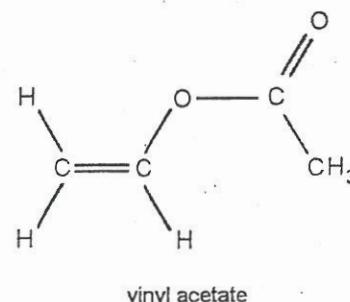
where  $a$  is the same parameter used in equation (13), and  $\Gamma$  denotes the *gamma function*, whose value (for a given  $a$ ) can be obtained from mathematical tables. For example, for  $a = 0.76$  (for PVOH in water at 25°C),<sup>4</sup> the ratio in equation (16) is

$$\frac{M_v}{M_n} \equiv S = 1.89 . \quad (17)$$

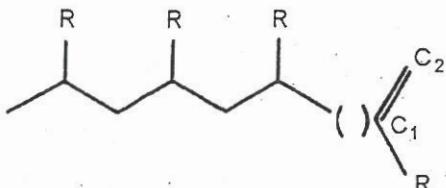
For  $a = 0.50$  and  $0.60$  (see Table 1 on p. 498),  $S$  is 1.77 and 1.81, respectively.

### Chemical Bonding in PVOH

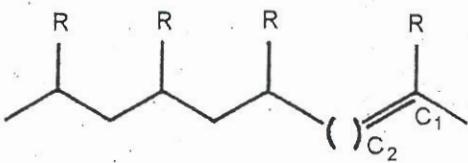
Poly(vinyl alcohol) is obtained from the hydrolysis of poly(vinyl acetate). The latter is synthesized from vinyl acetate monomers. Let us consider the question of how the vinyl acetate monomers link up to form the poly(vinyl acetate) polymer. The structure of vinyl acetate is shown.



It is an unsymmetrically substituted ethylene molecule, since only one end of the molecule is derivatized. Thus when a monomer is about to bond to the "growing" end of a polymer chain, it can do so in two ways with respect to the previously bonded monomer unit: Either the carbon atom containing the functional group, X, ( $C_1$ ) can bond to the terminal end of the polymer chain or the unfunctionalized carbon ( $C_2$ ) can form the bond. These alternatives are illustrated.



head-to-head

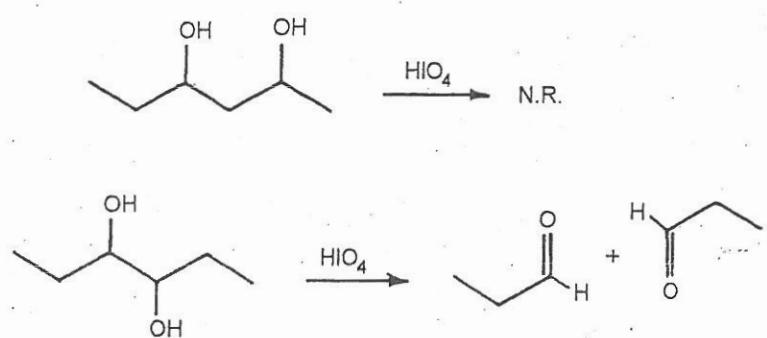


head-to-tail

An attachment in which the functional groups alternate is called "head-to-tail," and the one in which they are attached on adjacent carbon atoms is referred to as a "head-to-head" linkage. Because a head-to-head linkage involves considerable steric repulsion between the functional groups (they will have been added to *adjacent* carbon atoms on the polymer chain to form 1,2-substituents), it proceeds more slowly than a head-to-tail linkage. Therefore, a polymer will have a predominance of the latter arrangements, and the functional groups will, for the most part, alternate in their positions on the polymer backbone (i.e., repetitive 1,3-substituents).

Note also that if, after a head-to-head linkage has taken place, the next monomer attaches in a tail-to-tail fashion, the resulting arrangement between the substituents is a 1,4-disubstituted configuration. Considering the way this experiment is to be carried out (see the procedure), only the presence of head-to-head attachments is chemically significant. These result in the formation of 1,2-disubstituted structures on the polymer chain. The number of such events that occur in polymerization relative to the more common head-to-tail linkages (and thus 1,3-disubstituted structures) is of interest because the physical and chemical properties of the polymer depend on the fraction of such head-to-head linkages. Thus some analytical method for establishing this information is desirable.

In the case of the polymerization of vinyl acetate and the subsequent hydrolysis of the polymer to form PVOH, the consequence of a head-to-head linkage is the formation of a 1,2-diol (or vicinal glycol). The more common head-to-tail linkages result in the formation of 1,3-diols. The presence of 1,2-diol structures in the polymer can be conveniently distinguished from the 1,3-diols by a chemical means. The 1,2-diol is specifically cleaved (and oxidized) using periodic acid,  $HIO_4$ . Actually, the reagent used is the periodate anion (from  $KIO_4$ ), which hydrolyzes in water to form  $HIO_4$ . The reaction is as follows:



Hence treatment of a PVOH sample with  $\text{KIO}_4$  will split the polymer wherever a 1,2-diol structure exists. This results in a decrease in the (average) molecular weight of the polymer, and this change can be detected via viscosity measurements.

If we assume that the cleavage reaction is 100% effective (so that all the 1,2-diol linkages are cleaved), the increase in the number of solute molecules in the solution after treatment with  $\text{KIO}_4$  divided by the total number of monomer units represented in the polymer sample is equal to the ratio of 1,2-diol structures to the total 1,2-diol and 1,3-diol arrangements in the system. This ratio, which is defined here as  $f$ , can be expressed as

$$f = \frac{\frac{1}{M'_n} - \frac{1}{M^0}}{\frac{1}{M^0}}, \quad (18)$$

where  $M_n^0$  and  $M_n'$  are the number-average molecular weights of the polymer in the sample before and after the  $\text{KIO}_4$  cleavage respectively.  $M^0$  is the molecular weight of the monomer unit ( $\text{CH}_2\text{CHOH}$ ), which is 44 u. Using the relationship between the number average and viscosity-average molecular weight for PVOH [see equation (17)], we get the following expression for the head-to-head fraction:

$$f = SM^0 \left\{ \frac{1}{M'_v} - \frac{1}{M_v^0} \right\}. \quad (19)$$

This equation provides the final result used in this experiment. It permits  $f$  to be determined from  $[\eta]$  measurements of the PVOH sample before and after cleavage by  $\text{KIO}_4$ . The value of  $S$  depends on the choice of  $\alpha$  used in equation (16) (see Table 1).

## Safety Precautions

- $\text{KIO}_4$  is an oxidant and should be handled cautiously.
- Always wear safety glasses in the laboratory.
- Make sure you have been shown how to use proper pipetting techniques. *Never* pipet by mouth.
- Handle the viscometer carefully to avoid breakage.

**Procedure** You will be provided with a clean viscometer. If the viscometer appears to be dirty, do not proceed with the experiment. Either obtain a clean one, or take the time to clean your viscometer thoroughly. Consult your instructor. If you do not remember how to use the viscometer, review the material in Experiment 16, or ask your instructor for this information. A stock solution of PVOH in water ( $c_m$  about  $0.016 \text{ g mL}^{-1}$ ) should be available; if it is not, you will be told how to prepare this solution. This PVOH *must* have been appropriately filtered before use; undissolved particles will clog the viscometer. You will measure the viscosities of PVOH solutions of three or four different concentrations for both the cleaved and uncleaved polymers. You will calibrate the viscometer using pure water. Temperature control for each of these measurements is important.

1. Prepare 100 mL of a 50% solution of the PVOH stock solution. You will use this to prepare the diluted samples of the uncleaved polymer for measurement. Use purified (DI, RO, or distilled) water for the dilution in the volumetric flask. Cap and invert it several times gently. Do not agitate; this causes foaming. Because the polymer tends to adhere to the surface of glassware, rinse the pipet with water, then acetone, immediately after use. Aspirate dry. Follow this procedure whenever using glassware with PVOH solutions.
2. To prepare the *cleaved* polymer, pipet 50 mL of the stock solution into a 100-mL volumetric flask. Add 0.25 g of  $\text{KIO}_4$  and about 20 mL of deionized water. Cap and swirl the liquid a few times. Place the mixture in a hot-water bath ( $\sim 70^\circ\text{C}$ ) for several minutes until the solid dissolves; periodically mix the solution if necessary. After the solid has dissolved, remove the flask and let it cool to room temperature, then fill to the mark with deionized water. Gently invert it several times. Label the flask.
3. There are now two PVOH solutions (one cleaved and one uncleaved), each with  $c_m$  of about  $0.0080 \text{ g/mL}$ . From each of these prepare 50 mL of solutions corresponding to 80, 60, and 40% of that concentration. Make up the 40% solution from the 80% one. Label these new samples *immediately* after preparation. Place them all in a  $25^\circ\text{C}$  bath.
4. Calibrate the viscometer with purified water. Pipet 5 mL of water into the viscometer, which is clamped and held in a  $25^\circ\text{C}$  bath. Using a pipet bulb, carefully “push” the water up through the capillary tube until it is above the upper fiducial mark. Remove the bulb and allow the water to drain. Start the timer or stopwatch exactly as the water meniscus passes the upper fiducial mark; stop the timer just as the meniscus passes the lower mark. Record this time. Repeat this measurement at *least* three times. Use the pipet bulb to “reset” the liquid. Alternatively, use a

flexible tube connected to an aspirator. The timings should be within 0.2 to 0.5 s. After the calibration is complete, remove the viscometer from the bath.

5. Following the same procedure, measure the viscosities of the cleaved and uncleaved samples, starting with the most dilute samples. Make each measurement in triplicate.

### Calculations and Data Analysis

1. Calibrate the viscometer. Determine the constant  $c$  in equation (3) from the mean flow time of water and its density and viscosity at 25°C.
2. Determine and tabulate the viscosities and specific viscosities of the PVOH samples. Assume they have densities equal to that of pure water. In the same table list the bulk  $c_m$  values (g mL<sup>-1</sup>) of the PVOH samples.
3. For each sample determine  $[\eta]$  for the uncleaved and cleaved polymer. Also determine the Huggins constant. See equation (8).
4. Choose a set of  $K$  and  $a$  values, and using the appropriate relations, determine values of  $M_v$  for the uncleaved and cleaved PVOH, as well as the head-to-head ratio,  $f$ . Comment on the magnitudes of your results.
5. Determine  $M_v$  and  $f$  for another set of  $K$  and  $a$  values and compare them with the results in step 4.

### Questions and Further Thoughts

1. Consult a table of mathematical functions, and using the appropriate gamma functions and equation (16), verify the values of  $S$  cited in and after equation (17).
2. Using the definition of  $f$  [see the discussion before equation (18)], derive equation (18) and use the result in (17) to obtain equation (19).
3. Viscosity measurements are often performed on proteins and other biopolymers. What experiments could be carried out on such molecules to examine the effect of solution conditions (e.g., ionic strength, or pH) on the extent of denaturation (breakdown of the biologically active structure)?
4. Specific and intrinsic viscosities provide structural information about macromolecules under the assumption that the solute is spherical [see equation (4)]. Is this a good assumption for a polymer such as PVOH? For what sort of macromolecule would you expect this assumption to be poor?
5. How would the structure of a polymer such as PVOH in the vapor phase differ from that in aqueous solution?
6. The intrinsic viscosity of a sample of polydisperse PVOH in water was determined to be 86 cm<sup>3</sup> g<sup>-1</sup> at 298 K. Using the information in Table 1 and the Mark-Houwink equation, what range of molecular weights can be reported for the polymer?

**Notes**

1. C. Tanford, *Physical Chemistry of Macromolecules*, pp. 390-392, Wiley (New York), 1961.
2. Ibid, pp. 138-168.
3. See J. Bandrup and E. H. Immergut, eds., *Polymer Handbook*, vol. 2, IV-14, Wiley (New York), 1975.
4. P. J. Flory and F. S. Leutner, *J. Poly. Sci.*, 3:880 (1948); 5:267 (1950).
5. K. Dialer, K. Vogler, and F. Patat, *Helv. Chim. Acta*, 35:869 (1952).
6. H. A. Dieu, *J. Poly. Sci.*, 12:417 (1955).
7. J. R. Schaeffgen and P. J. Flory, *J. Am. Chem. Soc.*, 70:2709 (1948).

**Further Readings**

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## Experiment 32

*Data Sheet A*

### Intrinsic Viscosity

NAME \_\_\_\_\_

DATE \_\_\_\_\_

I. Viscometer calibration: Bath temperature \_\_\_\_\_

Stock solution concentration \_\_\_\_\_

Flow times, s \_\_\_\_\_  
\_\_\_\_\_

\_\_\_\_\_

II. Cleaved samples: Bath temperature \_\_\_\_\_

Flow Times, s

Sample 1

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Sample 2

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Sample 3

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

III. Uncleaved samples: Bath temperature \_\_\_\_\_

Flow Times, s

Sample 1

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Sample 2

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Sample 3

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

## Experiment 32

*Data Sheet B*

### Intrinsic Viscosity

NAME \_\_\_\_\_ DATE \_\_\_\_\_

II. Viscometer calibration: Bath temperature \_\_\_\_\_

Stock solution concentration: \_\_\_\_\_

Flow times, s: \_\_\_\_\_  
\_\_\_\_\_

II. Cleaved samples: Bath temperature \_\_\_\_\_

Flow times, s

Sample 1	Sample 2	Sample 3
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

III. Uncleaved samples: Bath temperature \_\_\_\_\_

Flow Times, s

Sample 1	Sample 2	Sample 3
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____