EXPERIMENT X

POLYMER VISCOSITY (S&G 6th & 7th eds. Expt. 28, 8th ed. Expt. 27)

1. Purpose

The molecular weight of a polymer may be obtained from viscosity measurement of a solution of this polymer. This technique will be applied to a sample of polyvinyl alcohol (a water soluble polymer) before and after treatment with a site selective cleaving agent to eventually obtain information on the regularity of the polymer chain.

2. Safety

None of the reagents used in this experiment present a special hazard, however be constantly aware that you are working in a chemistry laboratory where many hazardous substances and equipment may be present; please wear you safety goggles at all time.

3. Notes about the Experiment

Several batches of hydrolysed polyvinyl alcohol, each having different average molecular weight, are available as powder. The solutions are prepared as described in the text book, although a stock solution may be available. If you prepare you own stock solution, make sure to record the identification number of the batch of polyvinyl alcohol you are using. If the stock solution has already been prepared for you, record in your notebook (and in the report) its ID and its concentration.

The viscosity depends significantly on temperature; make sure that the temperature of the circulating/thermostatting bath is stable at 25 C; check this temperature regularly during the experiment.

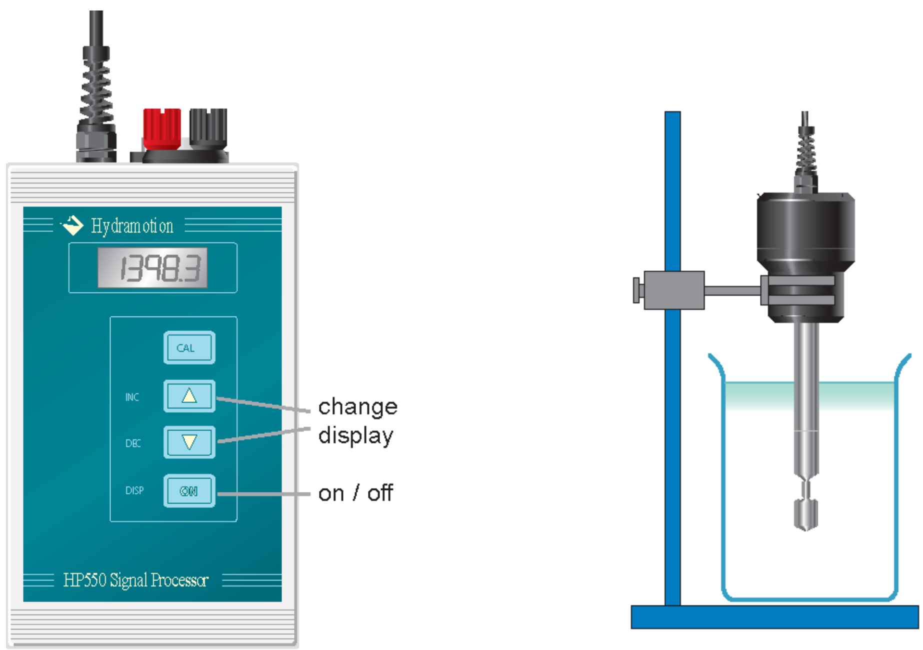
The experiment should be performed with at least *three* different concentrations of the polymer. Three measurements should be taken of each concentration once the viscosity value has stabilized. The measurements should be taken one minute apart.

4. Viscometer

The Viscolite 700HP (see Fig. 2) is a handheld or bench-mounted instrument for the instant measurement of viscosity of fluids by insertion. The actual sensor has a solid construction with no moving parts. It is connected to a bench-top microprocessor (HP550) unit by a flexible cable, and the whole instrument is powered by a transformer/adaptor unit. The Viscolite can be used with any volume of fluid. In the present set-up, the sample to be measured in contained in jacketed beaker kept at a constant temperature (25 C).

Principle of operation.

The sensor element consists of a shaft with an end mass, or bob, which is made to vibrate (also called resonate) at its natural frequency. When vibrating, the moving parts of the sensor shear through the fluid. As this shearing takes place, energy is lost to the drag forces on the sensor caused by the viscosity of the fluid. The loss of energy in each cycle of vibration is measured by the sensor electronics and the microprocessor in the Display Unit. From this energy loss, the actual viscosity of the fluid is determined. The Viscolite is therefore in a class of instruments sometimes called 'resonant' or 'vibrational' viscometers. The response of these devices is not purely with viscosity but with the product of viscosity and density, i.e. viscosity x density. In practice, viscosity changes on a far greater scale than density, and the fluid density can be accommodated by simply entering its nominal value in the Display Unit. Note that this type of instrument responds properly only with Newtonian fluids[1].



(a) (b)

Figure 2. a) HP550 signal processor, b) sensor properly immersed in sample.

Using the Viscolite 700HP

Plug the transformer into the mains and turn on. The microprocessor of the unit goes through a start-up routine, running the display through all its digits and settling on the display of viscosity, with the letters ' **VL** ' shown briefly, in units of 0.01 centipoise (cP ≡ mPa·s). This is called Normal Mode. The HP550 signal processor/display unit has a 5-digit display and 4 keys.

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[1] A Newtonian fluid is a fluid whose stress at each point is linearly proportional to its strain rate at that point. The constant of proportionality is the viscosity.

- For measurement purposes only the ON key and the ▼ and ▲ keys are of concern (the CAL key is used for setting up the instrument). Each time a key is pressed a beeper sounds.
- The ▼ and ▲ keys are used to cycle from one measured value display to the other.
- The ON key is not used for power-on in transformer powered units, but is used to access various parameters within each display mode.
- Each time the ▼ and ▲ keys are pressed the display briefly shows a symbol for a particular measurement, followed by its value, namely:
    - **VL** Live viscosity reading
    - **VC** Viscosity corrected to reference temperature
    - **t** Fluid temperature

The live viscosity value, **VL**, is the actual instantaneous viscosity of the fluid at its current temperature. The corrected viscosity value is calculated from the fluid temperature and keypad entered correction constants (see Section 3). Following power-up the Display Unit will display the dynamic viscosity, **VL**, of the fluid surrounding the sensor.

Measuring Viscosity

IMPORTANT NOTE. Please be extremely careful not to drop the probe; this might result in deformation of the resonant head, thus affecting the performance of the instrument.

If the sensor is in air, *and perfectly clean*, the instrument should read zero. Measurement of viscosity is straightforward. Pour 45 ml of the sample to be measured into the thermostated beaker provided, insert the sensor into the fluid to be measured, *to the depth shown in Fig. 2* and the reading of viscosity in 0.01 centipoise (ie, 1 cP reads 100) will appear on the **VL** display. It may be necessary to wait for the sample temperature (and the reading) to stabilize. To change sample, slide the sensor out of the solution, empty the jacketed beaker into a waste container, rinse with some distilled water, then dry with a tissue. Reposition and refill the beaker with the new solution and insert the probe.

5. Data analysis and Discussion

The units of "intrinsic viscosity" are $(\text{concentration})^{-1}$. It is not really a viscosity at all and alternate names are used in newer texts. The correlations represented by Eq. 7 are given for $[\eta]$ in units of $(\text{g}/100\ \text{ml})^{-1}$, which is not very clearly stated in S&G. As indicated in the text book, assume that all your solutions have the density of water (*this approximation introduces an insignificant error in the results*). To calculate the apparatus constant, make sure to use the value of the density and the viscosity of water at the temperature at which the experiment was run (may need to interpolate this value from the data provided in Table 1).

To analyse properly the data and to get meaningful results, it is essential to have a good estimate of the errors associated with each derived quantity and to perform weighted fits.

Table 1. Density and intrinsic viscosity of water around 25 C.

t/C	**d_{water} /g cm^{-3}**	**$\eta_0(H_2O)$/cp**
23.00	0.99757	0.9317
25.00	0.99708	0.8909
27.00	0.99654	0.8525

Answer the questions in the text book. In the results section of your report be sure to include you results tabulated in the format shown below; also provide an electronic copy of this data (Excel template may be found on the course web site)

Sample ID: [PvOH] = *xxxxxx* (units) Flow time units[2]:

	Uncleaved solutions			Cleaved solutions		
water	Soln 1	Soln 2	Soln 3	Soln 1	Soln 2	Soln 3
...	...	...	...	...	...	...
...	...	...	...	...	...	...
...	...	...	...	...	...	...
...	...	...	...	...	...	...

Can you think of other very common applications for which viscosity is an important parameter to know and why?

[2] If using a viscometer requiring flow time measurement.

Chem 366W report check list

A report will not be accepted without all the items of this list checked. If a checked item is found missing in the report, the report will be automatically down-graded.

Student Name:____

Report: Polymer Viscosity

Title page.

Correct title of the experiment .... ☐
Student Name & student ID .... ☐
Partner name (*if applicable*)____
Date of performance of experiment .... ☐
Abstract.. ☐
Introduction and theory .... ☐
Experimental
Changes from text description mentioned (*if applicable*)...................................... ☐
Sample ID, ser no, stock solution …etc recorded (*if applicable*) .... ☐
Results
Results as Tables .... ☐
Graphs
Size, at least ½ page .... ☐
Axis labelled .... ☐
Axis labels have units .... ☐
Axis scales are sensible .... ☐
Only significant figures .... ☐
Uncertainties quoted .... ☐
Raw data provided (*electronic form, if applicable*) .... ☐
Calculations
Sample calculation provided .... ☐
Error analysis .... ☐
Sample error calculation provided .... ☐
Discussion
Comments on results .... ☐
Questions in text book and in manual answered .... ☐
Comparison with literature value(s) .... ☐
Conclusion.. ☐

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