Use of Viscosity in Qualitative Analysis

Michael D. Mosher* and Chris Saw

Department of Chemistry, University of Nebraska at Kearney, Kearney, NE 68849-1150, mosherm@unk.edu

Abstract: There are many examples of undergraduate laboratory experiments that illustrate physical properties (i.e., melting point, boiling point, refractive index, density). There are relatively few examples of the use of viscometry in the freshman and sophomore laboratories. Moreover, the use of viscometry is usually treated in the same fashion as melting point determinations (comparison of the experimental viscosity to the literature value.)

The experiment reported here describes a laboratory exercise that uses viscosity to illustrate the principles of an analytical method. Students prepare a standard curve comparing viscosity to the length of the carbon chain in a series of alcohols or alkanes. Students then measure the viscosity of an unknown straight-chained compound to determine its identity.

The experiment is suitable for students in high school or in freshman- or sophomore-level college chemistry classes. The experiment gives students a greater understanding of both viscosity and the use of analytical methods to qualitatively explore an unknown compound.

Introduction

The typical undergraduate laboratory courses, especially those in general and organic Chemistry, devote considerable time to experiments that illustrate the use of physical properties to identify an unknown compound. These experiments allow students to gain experience with many typical experimental devices. For example, students typically gain experience with a melting point apparatus by determining the melting point and mixed melting point of an unknown compound. The unknown is identified by comparison to a table of literature melting point values. Several experiments have been published over the years relating the usefulness of this type of experiment [1].

Experiments that utilize physical properties are often open-ended assignments-students decide which steps they should take in order to solve the identity of the unknown compound. This type of experiment requires a great deal of flexibility in scheduling. In many cases, the experiments are designed to last two or three laboratory periods in order to provide enough time for the majority of the students to complete the exercise. Time spent working on the same laboratory experiment could be better used learning a different laboratory apparatus. Many experiments that illustrate the use of physical properties require considerable amounts of time to complete.

Those experiments that do a good job of relating physical properties in a relatively short amount of time are focused on one or two properties and have little readily apparent application to structure analysis or to the development of the analytical method. Experiments involving the measurement of viscosity, refractive index, melting point, boiling point, and density are examples. These experiments focus on either the identification of an unknown compound by comparison to a table of data found in external sources or on the determination of the effects caused by changing the temperature, concentration, pressure, or other parameters.

We wish to report an experiment that utilizes viscosity measurements to assist the preparation of an analytical method and to identify an unknown compound without consulting the external literature. By adjusting the level of prelaboratory discussion and/or in-laboratory exercises, the experiment can be made suitable for science students at any stage of their education. The supplementary material for this experiment includes student handouts for high school chemistry and college general chemistry courses.

Experimental

This laboratory experiment is designed to use an Ostwald viscometer; however, other varieties of viscometers are acceptable. Also, instructions for making an inexpensive viscometer have been described in the literature [2]. During the prelaboratory lecture, the use of the viscometer, viscosity theory, and the relationship between intermolecular forces and viscosity are discussed [3].

Students are supplied with a set of handouts [4] (instructions and a data sheet), a viscometer, a stopwatch, and a series of known alcohols or alkanes (the compounds used in the experiment are shown in Table 1). Alternatively, the class can be divided such that some students perform the experiment on the alcohols while others work on the alkanes. Two of the compounds from each group are reserved as unknowns.

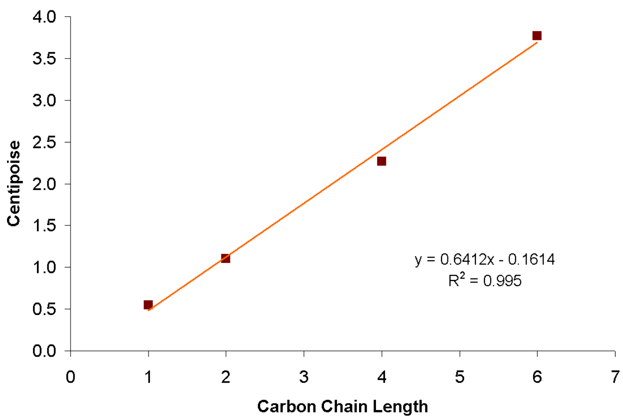
The viscometers can be calibrated by the students using the typical standards or used as is. The students are then told to prepare a standard curve of the series of four known compounds by measuring the viscosity and plotting it versus the number of carbon atoms in the compound. The resulting plot is fit to a straight line using the linear-least-squares method. Then, the students measure the viscosity and determine the identity of an unknown compound (one of the compounds not used from the list in Table 1). Students record the viscosity readings, the standard curve, and a discussion of the results in the laboratory notebook or on a laboratory data sheet [4].

Results and Discussion

The laboratory class is introduced to viscosity theory and its measurement using the Ostwald viscometer or a similar instrument. The viscosities of the compounds vary enough that small measurement errors do not produce major problems in the identification of the unknown compound. The standard curve can be constructed using simple spreadsheet or graphing programs, such as Microsoft Excel, Cricket Graph,

Table 1. Compounds used in the experiment

Alcohols	Alkanes
Methanol	pentane
Ethanol	hexane
1-propanol	heptane
1-butanol	octane
1-pentanol	nonane
1-hexanol	decane



Alcohol chain length	Relative viscosity at 25°C	Centipoise
1	1.00	0.55
2	2.01	1.10
4	4.15	2.27
6	6.89	3.77

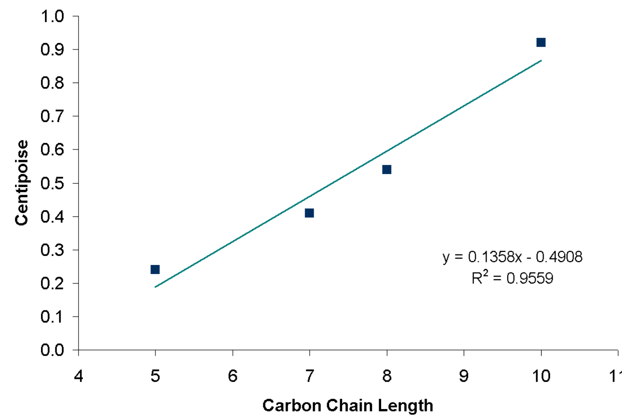
Figure 1. Sample student data and least-squares plot for the alcohol series.

Kaleidograph, or Delta Graph. The best-fit line through the experimental data can also be approximated with the use of graph paper and a ruler. Alternate methods for determining the best-fit line include manual calculation of the equation of the line using the linear least squares method [5]. If the equation of the line is to be determined, the instructor should present the linear-least-squares method to the student. Sample student curves are shown in Figures 1 and 2.

When presented with the unknown compound, students are quickly able to tell the identity of the compound based on the results of the standard curve. The difficulty of the unknown compound assigned can be adjusted based on the level of the student. For example, a student in high school chemistry could be presented with an unknown from Table 1, while a student in college-level general chemistry could be presented with a linear compound not listed in Table 1 or with a nonlinear compound (such as isopropanol). The identification of a nonlinear compound provides a chance to discuss the standard curve in greater detail. Students are able to quickly determine the nonlinearity of the compound.

The data that is obtained can be utilized in two ways. For the less advanced student, the raw data from the viscometer (in seconds) can be plotted versus the chain length of the compound to provide the standard curve. The very nature of the standard curve makes this treatment of the data acceptable.

For the more advanced student, the raw data is first converted into centipoise by the standard method before the standard curve is prepared.



Alkane chain length	Relative viscosity at 25°C	Centipoise
5	1.00	0.24
7	1.70	0.41
8	2.25	0.54
10	3.83	0.92

Figure 2. Sample student data and least-squares plot for the alkane series.

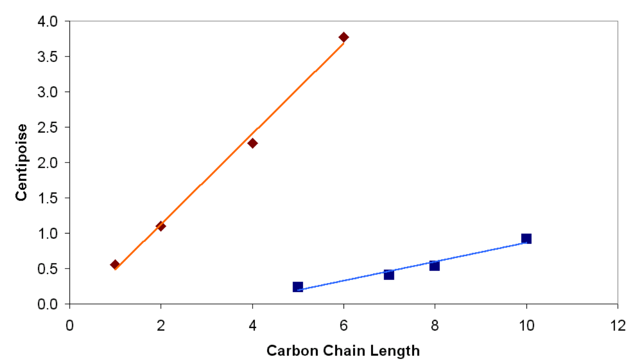


Figure 3. Comparison of alcohol (red line) and alkane (blue line) viscosities.

Both treatments of the data provide linear relationships between viscosity and carbon chain length (see Figure 1 and Figure 2). When the compounds used in the experiment are extended to longer carbon chains, a slight deviation from linearity exists (see Figure 2). This is particularly noticeable in the alkane series as seen by the lower correlation coefficient;. however, the deviation from linearity does not affect the results of the experiment.

In order to discuss the dramatic effect of hydrogen bonding on the viscosity of a molecule, the data from the alcohol experiment can be plotted on the same graph as the data from the alkane experiment (Figure 3).

Conclusion

Student response to this experiment indicates that the basic method behind an analytical and qualitative analysis has been learned. Students are able to use analytical methodology (preparation of a standard curve, calibration of an instrument, graphical analysis of the data) and to apply the method to the solution of an unknown. The experiment clearly demonstrates the features of an analytical method, the theory behind a physical property, and its application to the elucidation of compound structure using an inexpensive viscometer. Students are made keenly aware of the need to use good technique for data measurement and of the existence of trends in the properties of related molecules. Moreover, the experiment clearly demonstrates these principles in the span of one laboratory period.

Acknowledgment. The authors would like to thank the Department of Chemistry at the University of Nebraska at Kearney for financial support to develop of this experiment.

References and Notes

1. See, for example, Wedvik, J. C.; Mcmanaman, C.; Anderson, J. S.; Carroll, M. K. J. Chem. Educ. 1998, 75, 885–888; Ault, A. Techniques and Experiments for Organic Chemistry, 4th ed.; Allyn and Bacon: Boston, MA, 1983; Chapters 16–19; Mayo, D. W.; Pike, R. M.; Trumper, P. K. Microscale Organic Laboratory with Multistep and Multiscale Syntheses, 3rd ed.; Wiley: New York, 1994; Chapter 4.
2. McCullough, T. J. Chem. Educ. 1984, 61, 68–69; Seckin, T.; Kormali, S. M. J. Chem. Educ. 1996, 73, 193–194; Giguere, J.; Arseneault, E.; Dumont, H. J. Chem. Educ. 71, 121–124; Khurma, J. R. J. Chem. Educ. 1991, 68, 63; Daignault, L. G.; Jackman, D. C.; Rillema, D. P. J. Chem. Educ. 1990, 67, 81–82; Bramwell, F .B.; Bramwell, F. J. Chem. Educ, 1982, 59, 680; Bhattacharyya, B., Majumdar, D. K. J. Chem. Educ. 1973, 50, 194.
3. Excellent sources for the theory of viscosity and intermolecular forces can be found in most general chemistry textbooks. See, for example, Zumdahl, S. S.; Zumdahl, S. A. Chemistry, 5th ed.; Houghton Mifflin: New York, 2000; pp 451–458; Hill, J. W.; Petrucci, R. H. General Chemistry, An Integrated Approach, 2nd ed.; Prentice Hall: Upper Saddle River, NJ, 1999; pp 487–490.
4. Instructions and data sheets for both high school and college-level general chemistry students, as well as instructor notes, are included in the supplementary material.
5. An excellent discussion on the manual determination of the linear least squares method can be found in: Popham, W.J., Sirotnik, K.A. "Educational Statistics: Use and Interpretation", 2nd Ed., (Harper & Row, NY: 1973), pp 96-123.