Microscale experiments on the purification of organic compounds

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Two inexpensive set-ups for purifying organic compounds are improvised: one for solid, the other for liquid samples. The materials used are accessible and affordable, and the construction of the set-up itself is quite simple and easy. The students can, in fact, prepare one for their own use, and because of its being so handy, the actual experiment may be done outside the traditional laboratory room. Moreover, the efficiency of purification is comparable to that of the conventional ones.

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One of the basic experiments in a laboratory course in organic chemistry is the purification of organic compounds. Among the techniques often taken up in these experiments are the crystallization of a solid and the distillation of a liquid. The conventional experiments involve either macro (about 1-10 g of solid or 50 mL of liquid) or semi-micro amounts of reagents (about 0.5 to 1.0 g of solid or 10 to 25 mL of liquid).

Interest in the use of small-scale methods in the chemistry laboratory dates back to a period well before World War II [1]. However, it was not until the mid 80's that the concept of microscale experiments was formally introduced to the academic community [2]. Since then, microscale experiments have been a part of chemistry laboratory classes in many colleges and universities.

The move to down-scale experiments in the teaching organic chemistry laboratory provides a solution to several problems inherent in a laboratory course: high cost of chemicals, laboratory safety, waste disposal, and completion time of experiments. Studies [3,4] have demonstrated that the reduction in the scale does not affect the development of laboratory skills in the student, but rather enhances understanding of the principles and techniques illustrated, thus resulting in higher level of performance.

In this paper, we present microscale experiments for crystallization and for distillation. Several papers and publications have previously described how to filter [4-10] and distill [9, 10, 11] micro amounts of organic samples. Most of these methods employ Pasteur pipets and glass tubes manipulated and put together in different ways and designs.

In the experiments described here, some other new materials which are very economical and readily available are utilized.

Microscale crystallization of a solid

In small-scale organic chemistry experiments, crystallization may be considered to be the most rapid and convenient method of purifying the solid products of a reaction. Based on differences in solubility of the components to be separated, the method involves dissolving the material to be purified in an appropriate solvent at an elevated temperature. As the solution cools, it becomes saturated with respect to the substance being purified and crystals of the pure substance separate.

Microscale equipment can be employed for this experiment. A 10-mL autoclavable vial is used as the vessel, and a 5-mL disposable plastic syringe is employed for the filtration step. Fig. 1 shows the filtration set-up for this experiment.

Procedure: About 100 mg of an impure sample of benzoic acid is weighed into an autoclavable vial. Two mL of distilled water and a small boiling chip are added to the sample and the mixture is heated to boiling over an alcohol lamp. A pinch of activated carbon is added and the mixture is reheated to boiling. (Caution: Boiling chips and carbon pieces are not added to a hot solution).

The hot solution is filtered into a filter syringe and the liquid is forced down the syringe through the plunger. The hot filtrate is allowed to cool to room temperature, then placed in an ice bath. The crystals are collected by filtering through paper.
This procedure gave a recovery of 60-80%, and the isolated crystals had a melting point of 120-124°C. With the small amount of mixture dealt with, the error due to premature crystallization during the initial filtration is minimized as the process is over while the mixture is still very hot. The use of the plunger as an aid hastens the process even more.

MICROSCALE DISTILLATION OF A LIQUID

Distillation is a method of purifying volatile liquids based on differences in boiling points. Fig. 2 shows an improvised set-up for microdistillation.

The innovated set-up consists of a 25 mm x 4.5 in tube as the distillation vessel and a 10-mL disposable plastic syringe as the condenser. Crushed ice that cools the vapors is introduced into the condenser through a hole bored at the upper end of the syringe. The distillate is collected in a 3.0-mL disposable plastic syringe one end of which has been plugged.

In purifying a sample, the crude liquid is placed in the distillation tube and heated to boiling. Vapors of the lowest-boiling liquid rise up the column to the condenser where they are condensed and collected in a receiver to yield a distillate. Increasing the heat causes the next highest-boiling liquid to boil; its vapors follow the same path and are collected in a different receiver. Thus the liquids distill in order of their boiling points with the lowest-boiling liquid distilling first.

If the liquids to be purified have a large difference in boiling point (50°C or higher), simple distillation will suffice; otherwise, fractional distillation is necessary.

In this experiment, commercial rubbing alcohol is subjected to distillation. From a plot of temperature versus volume of distillate collected, the number of components, the respective approximate boiling points and volume % of each component are determined.

Procedure. Around 3 mL of the sample (commercial rubbing alcohol) and a small boiling chip are placed in the distillation tube and heated over a small flame of an alcohol lamp until vapors are observed to rise up the tube. The rate of heating is then adjusted by raising or lowering the alcohol lamp such that the rate of distillation is no faster than 2 drops per minute. The temperature is recorded initially and for every 0.1 mL of distillate collected. The process is discontinued before the sample dries up.
Figure 3 shows the results obtained by distilling 25 mL of rubbing alcohol. Fractions rich in isopropyl alcohol distilled at around 82°C (boiling point of isopropyl alcohol is 82.5°C). These fractions comprise close to 70% of the sample. It will be noted that the distillation curve obtained approximates that for a fractional distillation, an indication of good degree of resolution for a simple distillation. Moreover, the running-water component of the conventional set-up is done away with and replaced by crushed ice, thus simplifying the set-up considerably without sacrificing efficiency.

CONCLUSION

Purification of organic compounds can be done much faster, more economically, and with comparable efficiency as the conventional ones, on a microscale. Simple, economical, and easy-to-assemble devices can be improvised which can be used to demonstrate purification techniques without sacrificing quality of instruction. With the handy feature of the set-ups, the experiment can be done in a "nontraditional laboratory", i.e., anywhere conducive and convenient to the experimenter.

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REFERENCES: