filtrates & residues

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Extraction, Isolation, and Characterization of Fullerene C_{60}

A Safe and Reliable Separation Experiment

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The recent discovery (*1*) and characterization (*2, 3*) of the pure-carbon, soccer ball–shaped molecule buckminsterfullerene-C_{60} has spawned intensive research (*4*). Now a favorite study topic of physical and organic chemists, fullerene chemistry has instilled in scientists a variety of application hopes. Although development is slow, many envision a future of fullerene superconductors, large-scale industrial catalysts, and perhaps even antiviral agents (*5*). This academic interest and enthusiasm in fullerene science has prompted us to develop a safe, inexpensive, and practical undergraduate activity for the study of C_{60}, the most popular member of the fullerene family.

Appropriate for an advanced high school or introductory college chemistry laboratory, the experiment described here allows students to safely perform organic chemistry techniques in the absence of a fume hood. The methods we employ are modifications of those described by Ajie (*3*), Scrivens (*6*) and Marecek (*7*). Owing to the nonpolar nature of fullerene molecules, light-viscosity mineral oil is a suitable solvent for recovery of small quantities of C_{60}. Nontoxic and relatively unreactive, mineral oil has the added benefit of not vaporizing at room temperature, unlike typical organic solvents. While no material safety data sheet (MSDS) is currently available for fullerene soot and its properties have not been fully investigated, recent studies involving topical application of fullerene extracts to mouse skin have shown no tumor-promoting activity in either acute or subchronic exposure (*8*). In this activity, students solubilize and extract fullerenes (predominantly C_{60} and C_{70}) from fullerene soot and chromatographically separate C_{60} molecules from the resulting mixture. The C_{60} product may then be characterized spectrophotometrically.

Experimental Procedure

Crude Fullerene Extraction

Commercial fullerene soot may be purchased at a relatively low cost (MER Corp., 7960 Kolb Rd., Tucson, AZ). A mixture of 0.10 g of soot and 20 mL of laboratory-grade light mineral oil (80–90 Saybolt viscosity, 38 °C) is shaken vigorously for 3 min in a small capped glass bottle and allowed to sit overnight (mineral oil extraction of fullerenes is a slow process). The resulting slurry is filtered through Whatman No. 1 qualitative paper under aspiration. Approximately 30 min is needed to achieve sufficient recovery of the filtrate, a wine-red solution characteristic of a predominantly C_{60} and C_{70} mixture. An alternative approach if aspiration is not available is to allow self-filtration of the original shaken mixture for at least 4 hours. Extraction occurs during the filtration process.

Chromatography Column Preparation and C_{60} Separation

A small plug of household cotton (the size of a pea) is inserted into a short-tip Pasteur pipet and lightly forced to the bottom. A uniformly distributed mixture of 0.5 g of activated charcoal (Norit SG) and 5 g of reagent-grade silica gel (Baker analyzed, 60–200 mesh) is poured from a creased weighing paper into the Pasteur pipet column to a level 2 cm above the cotton plug (Fig. 1). Two additional columns are prepared in the same manner. The three columns are then taped together and clamped above a small vial. A Pasteur pipet is used to carefully load the columns to the top with the C_{60}/C_{70} solution. Complete elution of the columns takes about 24 hours. The light purple color indicates a relatively pure C_{60} solution. Additional separations using the same columns may prove unreliable owing to C_{70} elution. The C_{60} solution, as well as the C_{60}/C_{70} mixture, may be spectrophotometrically analyzed against a mineral oil blank in a Spectronic-20. Waste from this experiment should be disposed of in the same way as waste motor oil would be according to local ordinances.

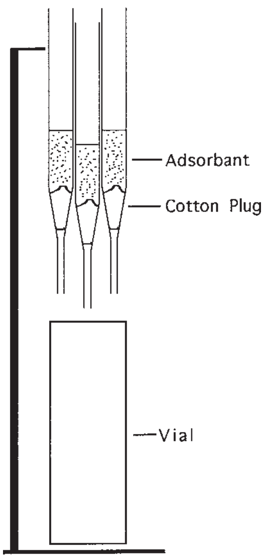

Figure 1. Chromatographic column setup.

Results and Conclusions

Analysis of the relative amounts of C_{60} and C_{70} recovered may be approximated using a Beer's law calculation and the extinction coefficients for C_{60} (ca. 710 $M^{-1}cm^{-1}$ at 540 nm) and C_{70} (ca. 15,000 $M^{-1}cm^{-1}$ at 468 nm) (*9*). It is not unusual to expect a 50% decrease in C_{60} concentration measured at 540 nm after chromatographic separation. However, absorption readings should fall within the reliability range of the Spectronic-20. We typically recover between 2.0

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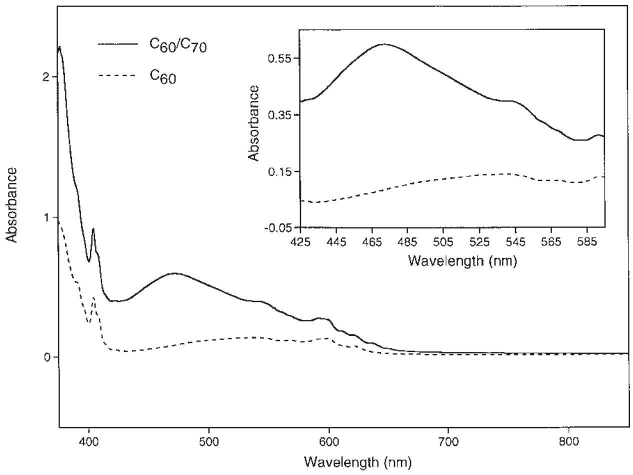

Figure 2. Sample absorption spectra for fullerenes in mineral oil.

and 3.0×10^{-4} M C_{60} in the final column eluates. Sample spectrophotometric results are shown in Figure 2. An expected maximum near 540 nm can be visualized for the C_{60} solution, and the original filtered extract shows the characteristic C_{70} peak near 468 nm.

The value of the experiment described here, while it provides a practical means for studying a currently significant topic, may be viewed as twofold. The techniques outlined (separation and characterization) are fundamental experimental tools for beginning chemistry students. Comparisons can be drawn between the specificity of two-phase filtering extractions and column chromatographic single-phase isolations. The experiment clearly illustrates a chemical separation. Furthermore, characterization of materials based on their optical properties is of universal value in science education, and this experiment provides a practical application for the use of the spectrophotometer. Finally, the dramatic visible color changes in this activity stimulate student interest and excitement and reinforce the intrinsic reward of laboratory success.

Acknowledgments

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Molecular model of C_{60} created using software from CAChe Scientific, Inc.