Extracting fullerenes from fullerene soot

Instructor Preparation and Discussion

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Age: This activity is most appropriate for a high school science class rather than a take home project.

Background: In 1996, Robert F. Curl, Jr., Sir Harold Kroto, and Richard E. Smalley were awarded the Nobel Prize in Chemistry for isolating fullerenes, cage-like molecules made entirely of carbon. In 1990, scientists Donald Huffmann of the University of Arizona and Wolfgang Krätschmer of Max Planck Institute for Nuclear Physics and their co-workers devised a way to produce fullerene soot in sufficient enough quantities to measure using spectroscopy. In this activity students perform similar experiments to those of the Huffmann and Krätschmer to extract the two most abundant fullerenes, C_{60} and C_{70}, from fullerene soot and examine their samples using a spectrophotometer.

Instructor Notes: Preparation for the experiment will take approximately 30 minutes of instructor time, but will require approximately 3 days of resting time before solutions are ready to use. The students will elute their C_{60}/C_{70} filtrate through a mixture of activated charcoal and silica gel for 24 hours. Samples could be collected every 6 hours or every 12 hours and compared for purity using the spectrometer. The C_{60}/C_{70} filtrate recipe is enough for one set of experiments. Scale appropriately for larger class.

Supplies for classroom activity

- Fullerene soot (Sigma-Aldrich, 572497)
- Laboratory-grade light mineral oil (Sigma-Aldrich, 330779)
- Whatman No. 1 or 2 filter paper
- Filter funnel
- Activated charcoal (Sigma-Aldrich, C5510)
- Reagent-grade silica gel pore size 60 Å, 70-230 mesh (Sigma-Aldrich, 288624)
- Scanning Spectrophotometer

One set per experiment:

- 1 24 mL (6 dram) screw cap vial
- 3 short-tip Pasteur pipets (VWR, 14673-010)
- Cotton balls
- 3 Plastic cuvettes (use glass cuvettes if examining peaks in UV range)
- Enough 4 mL (3 dram) vials for collecting the eluate

Preparing the C60/C70 filtrate: Start at least three days before in-class activity

1. Shake a mixture of 0.1 g fullerene soot and 20 mL of laboratory-grade light mineral oil vigorously in a 24 mL screw cap vial for 3 minutes. (Note: for a set of 15 experiments, shake a mixture of 0.1 g fullerene soot and 300 mL of laboratory-grade light mineral oil)
2. Allow mixture to sit at room temperature for approximately two days.
3. The day before the in class activity, filter the slurry through Whatman No. 1 or 2 paper funnel. This can take up to four hours. The filtrate will be a wine-red solution characteristic of a predominantly C_{60} and C_{70} mixture.

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Student Instructions

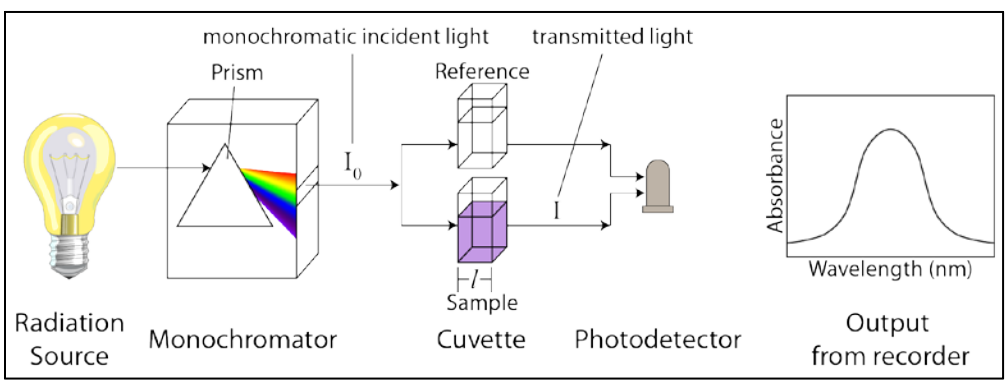
Background

History
The "buckyball" is a cage-like nanoscale structure made entirely of carbon. Discovered in the laboratory in 1985 by Robert F. Curl Jr., Sir Harold Kroto, Richard E. Smalley, each buckyball is composed of 60 carbon atoms (C_{60}). Curl, Kroto and Smalley and their graduate students, directed a laser at graphite (another allotrope of carbon) to produce carbon vapor. The vapor was mixed with an inert gas (helium), and as it cooled, the carbon atoms combined to form clusters. Analysis of these clusters showed that they were very stable, and the most abundant cluster contained 60 carbon atoms. The scientist correctly proposed that the structure of this new carbon species resembled a soccer ball with the bonds between the carbon atoms forming 20 hexagons and 12 pentagons. In 1996, Curl, Kroto, and Smalley won a Nobel Prize in Chemistry for their discovery.

In 1990, scientists Donald Huffmann of the University of Arizona and Wolfgang Krätschmer of Max Planck Institute for Nuclear Physics and their co-workers devised a way to produce fullerene soot in sufficient enough quantities to measure using spectroscopy. They evaporated graphite rods in inert gas, producing carbon smoke that they collected and dissolved in benzene then examined the infrared and ultraviolet absorption spectra of their sample.

Chromatography
Chromatography is a technique used to separate mixtures. The mixture is dissolved in a solvent (*mobile phase*) and allowed to flow through or pass over an adsorbent material (*stationary phase*) often in a chromatography column or plane (paper chromatography or thin layer chromatography). Compounds in the mixture will adsorb to the stationary phase to different degrees, and thus some compounds will move through the stationary phase faster than others. In column chromatography, the separated mixture is collected as it comes out of the column or *elutes*. These collected fractions are called *eluates*. This experiment uses column chromatography to separate two allotropes of carbon, C_{60} and C_{70}.

Spectroscopy and the Beer-Lambert law
Spectroscopy is the study of the interaction between electromagnetic radiation and the atoms and molecules that make up matter. A spectrometer is a laboratory instrument that measures the amount of light absorbed by a sample solution. A schematic of a spectrometer can be seen below.



The concentration (c) of the sample in moles per liter can be calculated based on the absorbance (A) of the sample at a specific wavelength according to the Beer-Lambert Law:

$$A = \log(I_0/I) = \varepsilon\, l\, c$$

The intensity of the incident light entering the sample is I_0 while the intensity of the light transmitted through the sample is I. The constant ε is the extinction coefficient (L mol^{-1} cm^{-1} or M^{-1} cm^{-1}) at a specific wavelength, and l is the length of the path of light through the sample in the cuvette (cm).

Purpose

In this activity, we will separate sample mixture of C_{60} and C_{70} dissolved in a solvent, mineral oil, using column chromatography so that we can observe the absorption spectra of the nearly pure C_{60} sample.

Procedure

Chromatography column - cotton plugged columns

1. Plug a short-tip Pasteur pipet with a pea size cotton ball and lightly force into the bottom of the pipet.
2. Uniformly mix 0.5 g of activated charcoal and 5 g of reagent-grade silica gel.
3. Pour from creased weighing paper into the Pasteur pipet column to a level 2 cm above the cotton plug.
4. Repeat above for a total of 3 columns.
5. Tape all three columns together and clamp above a 3-dram vial.

Eluting

1. Use a Pasteur pipet to carefully load the columns to the top with the C_{60}/C_{70} filtrate.
2. Collect eluate in 3-dram vials every 12 hours.
3. Complete elution takes 24 hours.

Determining the amount of C_{60} and C_{70} in C_{60}/C_{70} filtrate and the eluates

1. The samples may be spectrophotometrically analyzed using mineral oil as a reference (blank)
   a. Day old C_{60}/C_{70} filtrate
   b. 12 hour eluate
   c. 24 hour eluate
2. Measure the absorbance at each of the following wavelengths and using the corresponding extinction coefficient (ε) to determine how much C_{60} and C_{70} are in each sample.

	Wavelength (nm)	ε (M^{-1} cm^{-1})
C_{60}	540	710
C_{70}	468	15,000

Discussion

1. How does the color of the C_{60}/C_{70} filtrate compare to the color of the eluates?
2. Which of your three samples was the purest C_{60} sample?
3. Did you elute a sample with a high concentration of C_{70}?
4. What could you do to extract more C_{70}?

References

Based on lab described in West, S. P.; Poon, T.; Anderson, J. L.; West, M. A.; Foote, C. S., Extraction, isolation, and characterization of fullerene C-60 - A safe and reliable separation experiment. *Journal of Chemical Education* **1997**, 74 (3), 311-312.

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