

Bulk Drug Analysis Using IR Spectrophotometry

References:

- Saferstein, Richard. *Criminalistics: An Introduction to Forensic Science*. Prentice Hall: Englewood Cliffs, NJ, 1998; pp 147-157, 253-288.
- Pouchert, Charles J. *The Aldrich Library of FT-IR Spectra*; Aldrich Chemical Company: Milwaukee, 1985; Volumes 1 and 2.
- The website of the National Criminal Justice Information Center has information on street drugs, including links to other government resources. The address of this page is <http://www.ncjrs.org/DrugsandCrime.asp>.

Goal

To determine the composition of a white solid, suspected drugs, found at the crime scene or in a suspect's possession.

Suggested Method of Analysis

Fourier Transform infrared spectrophotometry ; KBr pellet method of sample preparation {Alternatively, one can employ FT-Raman spectrophotometry with similar results. The sample preparation is much easier – the solid is probed directly.}

Reference spectra

For comparison purposes, students should look up the spectra of the “drugs” and cutting agents in an external source (such as *The Aldrich Library of FT-IR Spectra*, compiled by Charles J. Pouchert, published in 1985 by the Aldrich Chemical Company). This is to avoid having them spend inordinate lengths of time making pellets. At a minimum, students should experimentally measure the spectra of the presumed “drug” and cutting agent, and they may show that the spectral addition is nearly the same as the measured spectrum of the unknown.

Making the KBr pellets

Weigh 0.8 mg of the drug unknown or standard onto a piece of weighing paper. Transfer the solid into a small agate mortar; use the pestle to grind the sample hard enough to smear it over the bottom of the mortar. Weigh 0.15 g of ground, dry KBr onto another piece of weighing paper. With a small spatula, add about 0.05 g of the KBr to the solid sample in the mortar. Grind the two together. Use a spatula to scrape the mixture into a neat pile in the mortar; also scrape any solid off the pestle into the pile. Use the pestle to make a thin layer of the powder over the bottom of the mortar. Add the remainder of the KBr and grind lightly to mix the powders together. Scrape the mixture into a pile once more and grind it lightly again. The KBr should not be ground hard, but needs to be thoroughly mixed with the sample.

After grinding and mixing, the powder mixture should be transferred to a piece of weighing paper. Screw one bolt most of the way into the pellet die; about 1 mm of the threads should still be visible from the outside. Pour the powder mixture into the other hole in the die, avoiding as much as possible getting powder in the threads of the interior. Shake or tap the die to make the powder level in the hole. Screw the other bolt into the die as well, making it finger-tight. Use two wrenches to slowly tighten the two bolts. Tighten the bolts as much as you can, let the die sit for 15 seconds, and try again. Once you can no longer tighten the bolts the pellet fabrication is complete. Now use the wrenches to loosen the bolts. Remove the bolts and gently tap the die on the countertop to shake out any loose powder. Hold the die up to the light and inspect your pellet. It should be translucent and

without cracks. If it is not, do not attempt to retighten the bolts and press it more. You must start again from scratch. If the pellet is acceptable, proceed to record the IR spectrum.

Recording the Infrared Spectrum

Place the die on the V-shaped holder in the sample compartment of the spectrophotometer. Make sure that the platform is in the center of the chamber; in this position the pellet coincides with the laser and IR beam focus. Close the lid to the chamber. Record the infrared spectrum against an air background from 4000 to 450 cm^{-1} at a resolution of 1 cm^{-1} . Nine scans provides adequate signal-to-noise.

Cleanup

If the pellet is defective, or if the run is complete, poke the pellet out of the die with a spatula. Clean all of the pellet-making tools by rinsing with water, heating in a drying oven ($100\text{ }^{\circ}\text{C}$) for 30 minutes, and storing in a desiccator.

Identifying the Drug and Cutting Agent

The IR spectrum of the unknown drug mixture will resemble the spectrum of the pure drug fairly closely. It is harder to identify the cutting agent using just the pure spectra for comparison. If spectra of the three possible mixtures are available, students may be able to deduce which is which.

Observing the solubility of the unknown can also distinguish between cutting agents. Glucose is very soluble in water and will dissolve immediately. Starch is not very soluble in water but is distinct in appearance. It is a very fine powder that settles to the bottom of the test tube fairly quickly. Quinine tends to float on top of the water when placed in a test tube; it also clings to the sides. It does not dissolve very readily, either. Caffeine settles to the bottom and will eventually dissolve. It has larger particles than starch.

Observing the mixture and standards through a microscope may also help determine which drug and cutting agent are present.