Cigarette Smoke Analysis Using an Inexpensive Gas-Phase IR Cell

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Background

In 1992 Amey reported the development of relatively simple and very elegant experiments using FTIR spectroscopy for general chemistry lab courses (*1*). The experiments used balloons to trap different gases, the balloons serving as IR gas cells. These experiments allowed students to identify some of the components of air and smog. Some of the gases trapped in the balloons had to be investigated quickly because they caused damage (typically rupture) to the balloon. We wish to report the development of a more robust gas cell that also gives spectra with higher resolution, increasing the versatility of experiments in the freshman chemistry lab. The new cell is constructed from PVC pipe fittings available in most hardware stores and NaCl windows (*2*). It can tolerate more corrosive gases such as NO_x and HCl. It can be dismantled, cleaned, and reused. It also can hold vacuum (100 mTorr) for a reasonable amount of time, allowing students to sample such things as car exhaust and cigarette smoke, two new applications that are also included in this communication.

As Amey described in his article, FTIR experiments are often considered to be more interesting than the typical experiments presented in freshman chemistry courses, partially because they are more instrument intensive (*1*). FTIR experiments also allow for the introduction of infrared spectroscopy into the curriculum prior to organic and physical chemistry laboratory work. The overall goal of our project is to present IR spectroscopy in increasing levels of complexity throughout the curriculum and in the context of many types of chemical problems. Students would be introduced to IR spectroscopy in freshman chemistry, where they would use the technique to identify gases. These experiments would give them a first look into detection limits and the effect of concentration on band intensities. In organic chemistry they would see how useful IR spectroscopy is in identifying organic functional groups. Experiments developed for an instrumental analysis lab could be directed towards IR spectroscopy as a method of determining concentrations (such as the concentration of CO in car exhaust). In physical chemistry labs the technique is used to obtain bond lengths and vibrational and rotational constants, as in the typical HCl rovibrational experiment. It can also be used to follow reaction kinetics and assist in the determination of a reaction mechanism (*3*). In inorganic chemistry lab it can be used to follow the catalytic ability of a new material (*4*). Thus throughout a student's education one spectroscopic technique can be used to solve a large number of different and difficult chemical problems.

Experiments and Results

A $\frac{3}{4}$-in. PVC compression tee with a body length of approximately 5 in. serves as the cell body (Fig. 1). A PVC bushing connects a ball valve to the tee. A suitable adapter is then attached to the valve. We selected an adapter that allows us to connect the cell to a rubber hose. The compression fittings securely position two NaCl windows 2.5 cm in diameter and 0.5 cm thick.[1] Details on the cell construction, including a photograph, are available online.W

Once constructed and sealed tightly, the cell can be evacuated. The cell will not hold vacuum as well or as long as a typical IR glass gas cell. However, it is an excellent cell for less rigorous experiments. The cell is less likely to break than a glass cell and can be dismantled, cleaned, and reassembled for future use. (This is essential when analyzing cigarette smoke.) If the time in lab permits, the students can assemble the cell as part of the experiment. For a typical freshman chemistry lab of 24 students one might want to have available 12 cells, so that each pair of students can do their own set of experiments.

The cell can be evacuated by one of three methods which are outlined in the supplemental material.W The cell's ability to hold vacuum should be checked. Once the cell is evacuated a background spectrum needs to be collected and stored. The same background spectrum is used for all cells. The FTIR spectroscopic conditions used were 25 scans at 1 cm^{-1} resolution. A resolution of 2 cm^{-1} reduced the scanning time by almost half, allowing more students to use the instrument in a given time without losing information (with the exception of the cigarette smoke sample; we strongly suggest using 1 cm^{-1} resolution for smoke analysis).

How experiments are performed is dependent on the time available and the number of students in class. The best approach for a large class would be to assign known gas samples to some pairs of students and an unknown environmental sample to others. The goal of the experiment is for the class to determine whether IR spectroscopy is useful in the

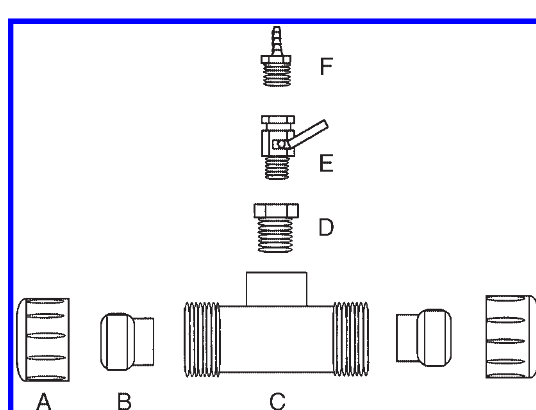

Figure 1. Schematic of PVC cell. Parts A, B, and C comprise the body of the cell. Two NaCl windows are securely positioned in the compression fittings (B) using a short segment of PVC tubing. A $\frac{1}{2}$- to $\frac{1}{4}$-in. PVC bushing (D) serves as the adapter to connect a ball valve (E) to the tee. A suitable adapter (F) is connected to the valve.

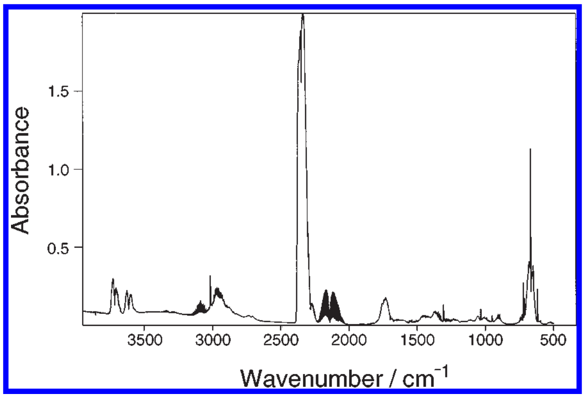

Figure 2. FTIR spectrum of cigarette smoke.

identification of gases present in car exhaust and cigarette smoke (*5*). Other gas mixtures of environmental concern can also be incorporated into this project.

The known gases included in the instructor notes are HCl, CO, H_2O vapor, CO_2, NO_x, CH_4 and NH_3.[W] Preparation of some of these samples is outlined in Amey's paper (*1*) and mentioned again in our supplemental material.[W] Depending on what is available and the amount of time set aside for the experiment, the instructor can incorporate other gases into the experiment such as ethylene, acetylene, CFCs, formaldehyde, propane, butane, and isobutane (components in aerosol cans) (*1*) and gasoline vapors (for comparison to car exhaust). We elected to only use gases that could be easily produced or collected from known sources like car exhaust. Two pairs of students are assigned car exhaust as their sample. One pair collects its sample in the first 30 seconds of cold-starting a car, and the second pair waits until the car has warmed up (15 minutes). These samples are usually different. The cold-start sample will contain detectable amounts of CO because the catalytic converter has yet to completely eliminate CO from the exhaust. After a few minutes the catalytic converter is fully operational and CO is not detected. If an old, polluting car is available it might be interesting to repeat the experiment with it to determine whether its catalytic converter is working.

Two pairs of students are assigned cigarette smoke. It is instructive to select several brands of cigarettes, particularly one without a filter and one with a filter, to determine if there is any difference in the gaseous components of their smoke (*6*). Details on how to get the cell to "smoke" a cigarette are given online.[W] Figure 2 shows a spectrum obtained at 1 cm^{-1} resolution for smoke from an unfiltered cigarette. This spectrum was collected using a 14-cm glass cell with KBr windows. The cell was evacuated to 1 mTorr. The valve for the cell was opened slowly so as to allow the cell to smoke $\frac{1}{5}$th of the unfiltered cigarette. Water vapor is subtracted to more easily visualize other features. This spectrum is almost identical to the one obtained by the students using the PVC cell (after water is subtracted). The difference resides in the visibility of the HCN band feature at 3300 cm^{-1}. This HCN feature is very weak when we use a PVC cell. Another feature of HCN, a narrow band at 712 cm^{-1}, is detected in both the glass cell and the PVC cell. Since the students should not be obtaining the spectrum of HCN in lab, the missing HCN feature in the PVC spectrum is not important for the experiment. The spectrum in Figure 2 is provided so that the instructor can indicate to the students that HCN, a very dangerous gas, is also produced in cigarette smoke. More detailed spectra of cigarette smoke are available online.[W]

Students will be able to identify water, carbon monoxide, carbon dioxide, and methane. Whereas carbon dioxide and water are the products of complete combustion of organic materials, the presence of carbon monoxide and methane indicates that there is incomplete combustion of tobacco in cigarettes. Not all the gases in cigarette smoke can be identified by the students (*7*). The wide band in the 1700s cm^{-1} region is most likely the combination of acetaldehyde, acetone, and acrolein, all molecules containing strong carbonyl stretches. Other narrow peaks in the spectrum identify other trace gases in smoke. These are methanol at 1033 cm^{-1}, ethylene at 949 cm^{-1}, and isoprene at 893 cm^{-1}.

Acknowledgments

This is one of several FTIR experiments being developed under the auspices of NSF ILI grant DUE-9750554. R. Fan, T. Furch, and K. A. Singmaster thank NSF grant CHE-9625628 (San Jose State/IBM Almaden Research Center Integrated Research and Education Program); A. Macias thanks the McNair Scholars program at SJSU; and N. Garizi thanks the College of Science at SJSU for financial support. K. A. Singmaster would like to thank the Petroleum Research Fund for partial support. P. Wagenknecht thanks the Research Corporation and The Camille and Henry Dreyfus Foundation.

[W]Supplemental Material

The complete description of this experiment, including cell construction and other supplemental materials, is available in this issue of *JCE Online*.

Note

1. Although unpolished NaCl windows are less expensive than polished ones, purchasing polished ones still keeps the cost of the cell below $40.

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