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PERSONAL BENZENE VAPOR DETECTION DEVICE

P. Glatkowski
M. Druy
Foster-Miller, Inc.
350 Second Avenue
Waltham, MA 02154-1196

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16. Abstract (Limit: 200 words) A badge was developed as a personal vapor detector to record the cumulative exposure of an individual to benzene (71432) vapor. The badge consisted of an optical fiber, small sections of which were coated with a membrane specific for the adsorption of benzene vapor. Membranes made of polyethylene and nylon, and filled with zeolite were applied to a silver halide fiber. Following exposure, the fiber was subjected to Fourier Transform Infrared (FTIR) spectrometry. The coating successfully retained the benzene vapor for several days. A concentration of 100,000 parts per million (ppm) could be detected. Concentrations of dichlorobenzene (25321226) of 80,000ppm were also detected. Unique absorbance peaks were used to distinguish the two compounds. It was possible to reuse the sensor. However, the desired detectability range of 1 to 100ppm was not met.

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Significant Findings

Benzene and dichlorobenzene were detected using both silver halide and chalcogenide infrared fiber optic sensors. The sensor portion of the fiber optic system was coated with Silicalite S-115 (Zeolite) to concentrate the benzene vapor into the region, within 5μ of the surface, where the evanescent wave propagates outside the optical fiber. Benzene could be detected at levels of 100,000 ppm and the coating retained the benzene for a period of days. Membranes of polyethylene and nylon filled with zeolite were successfully applied to a silver halide fiber and benzene at 100,000 ppm concentration could be detected.

Abstract

This report describes the Phase I work performed to develop a personal wear badge for the accumulated exposure detection of benzene vapor. Small sections of optical fiber were coated with a membrane specific for the adsorption of benzene vapor. After exposure the fiber was placed in a Fourier Transform Infrared (FTIR) spectrometer and the infrared spectra of the surface bound benzene was obtained. The qualitative exposure levels were determined by peak intensities. Benzene was detected at a concentration of 100,000 ppm. Dichlorobenzene was also detectable (80,000 ppm) and could be differentiated from benzene by unique absorbance peaks. The sensor was reusable and the cumulative exposure was determined as a function of time. The sensor modifications were based on the incorporation of benzene absorbing compounds contained in a thin, high surface area porous membrane coating applied to the fiber surface. The surface coating technology was developed and demonstrated on an active fiber system. A sensor cell was designed to allow handling and direct insertion into the FTIR spectrometer.

1. INTRODUCTION

Benzene is a volatile liquid that is soluble in lipids. It can be absorbed into the body following ingestion, inhalation, and actual contact. Available data from both animal and human studies indicate that after absorption benzene must undergo metabolic transformation to exert its toxic effects. Metabolism of benzene occurs primarily in the liver; however, the enzymes necessary for metabolism are also present in the bone marrow. The lymph system is also a target for benzene toxicity (1).

In humans the toxicity of benzene is characterized by a decrease in the various circulating blood cells. Some persons have developed myelogenous leukemia. Benzene can also cause neurotoxic effects; it is also genotoxic, causing structural changes to the chromosomes. The carcinogenicity of benzene has been demonstrated in rats and mice. Epidemiological studies suggest that long-term, low level exposure to benzene is carcinogenic in humans. The EPA has classified benzene as a human carcinogen (2-6).

Humans can be exposed to benzene in the environment, in consumer products, and in the workplace. The greatest exposures levels are found in the workplace. Most people are exposed to benzene vapor in tobacco smoke and in automobile gasoline. Consumer products containing benzene include glues, adhesives, household cleaning products, paint strippers, art supplies, tobacco, and gasoline. Exposure at the gas pump could be significant as can exposure in the rubber industry, oil refineries, chemical plants, the shoe industry, and gasoline storage facilities.

In order to assess this increased risk in a more quantitative manner it is desirable to have available a personal monitoring device that will allow a person to determine the level of his/her exposure to benzene in various locations, such as the workplace, home gasoline stations, etc. This device would integrate the exposure to benzene over a period of hours, days, weeks or months giving the person direct quantitative information concerning their exposure to benzene. This device, at the end of the designated time period, would be removed, placed in an FTIR spectrometer, and the level of exposure to benzene determined by rapid analysis.

This report discusses the results of a Phase I study to prove the feasibility of developing a personal badge for the personal monitoring of environmental benzene vapor. The badge consists of a modular fiber optic cell. A surface coating was designed for the adsorption of benzene vapor from the atmosphere and applied to the optical fiber. The coating was a high surface area porous coating consisting of micro porous nylon or polyethylene into which a small amount of zeolite was dispersed. The benzene absorbed by the zeolite was detected by means of FTIR infrared spectroscopy using the evanescent wave at the fiber's surface.

The fiber optic sensing system can be used in two ways to monitor benzene in air. It can be placed in a given location and monitored continuously using fiber optic cables as transmission lines. The alternative method is to place the fiber or fibers in a small badge that would be worn in an exposed position on the persons clothing to determine the level of exposure to benzene. In this case, the optical fiber is used both as a sensing element and as a transmission link. After a given length of time, the badge would be removed, placed in an FTIR spectrometer, and specific contaminants identified and the length of exposure determined by rapid analysis. This Phase I effort addressed the second method of application.

The optical fibers were analyzed spectroscopically by attenuated total reflectance (ATR) methods. This method takes advantage of the evanescent electromagnetic field that propagates across the interface of the fiber and sample. ATR is particularly useful when the sample under study is highly absorbing and/or scattering. Two kinds of fiber were employed in this program. The first was silver halide (AgBrCl) that was formed by extrusion into .75 mm diam fiber. In this program 1.25 in. segments of silver halide fiber were used by mounting them into a polyethylene holding cell which could be handled and fixtured into a IR spectrometer. In order to enhance (increase) the low concentration of benzene at the surface of the fiber, a thin (5 μm), high surface area adsorbent coating was placed on the fiber. Two different absorbents were used in this program. The first was a nylon membrane that was infiltrated with either picric acid or activated zeolite. Picric acid was tried as an absorbent/reactant to investigate whether benzene picrate will form during exposure to benzene vapor. The picrate formed should be detectable in the IR spectra. Secondly, zeolite adsorbents were tried, since zeolites should accumulate benzene near the surface and enhance the detection of benzene. Fiber coated in this fashion will continue to absorb provided the absorbent does not become saturated.

The second type of optical fiber used was a chalcogenide (NTEG) glass. This fiber was .360 mm diam and tapered in the region where it functioned as a sensor. Tapering in these fibers, means to draw the fiber down to a diameter of .100 mm, over a 1.5 cm length. This chalcogenide sensor was not dip-coated with polymer but was embedded in pure zeolite adsorbent. The IR signal was transmitted to and from this sensor using two 1.5m fiber optic cables made from the same material.

In this report the technical objectives of the Phase I effort are presented in Section 2. Details of the experimental procedure, apparatus, results and conclusions are presented in Section 3 through 3.3.

2. TECHNICAL OBJECTIVES

The primary technical objective of the program was to develop a personal detection device for the quantitative detection of exposure to benzene. This device is based on (a) the use of optical fibers coated with a membrane specific to benzene and (b) analysis by Fourier Transform Infrared (FTIR) spectroscopy using the evanescent wave at the fiber surface. Specific objectives or milestones of this program include:

- Development of a porous coating that is specific to the absorption of benzene-like molecules.
- Demonstrate that materials other than benzene can be resolved and differentiated from benzene.
- Development of a thin (5 to 10μ) and uniform coating on the optical fiber.
- Determine the saturation capacity of the nylon coating for benzene.
- Detection of low concentrations of benzene vapor with the coated fiber by FTIR attenuated total reflectance.

3. EXPERIMENTAL DESIGN, METHODS, AND CONCLUSIONS

In this section, the series of experiments performed is presented. This section has been divided into three subsections. The first subsection (3.1) details the measurement apparatus used in each of the experiments. In subsection 3.2 each of the four experiments are summarized. Subsection 3.3 summarizes the conclusions of this effort. The series of events that took place, which lead us to these four experiments, is presented next.

Four different fiber coatings were studied. The first coating or membrane, attempted for use as a benzene absorbent, was a porous nylon doped with picric acid. Even though we were able to create coated fibers it was quickly discovered that the picric acid did not readily form benzene-picrate. At this time we consulted with Professor Alfred Viola, Northeastern University Department of Chemistry, to better understand picrate chemistry. Following this meeting and the results obtained in the laboratory, we decided that picric acid will not provide the reactivity and sensitivity required to detect low concentrations of benzene in air. Subsequently, we consulted with Yi Hua Ma, professor and director of the Center for Inorganic Membrane Studies at Worcester Polytechnic Institute, regarding the application of zeolites as gaseous absorbent coatings. Following his recommendations, we planned three more experiments to demonstrate that our fiber optic sensors can function as specific gas detectors. Professor Ma calculated that the amount of benzene absorbed by the chosen zeolite (Silicalite S-115) will be sufficient to allow measurement by our infrared optical fibers. The next three experiments all employed activated zeolites, as the absorbent, coated onto both silver halide and chalcongenide fiber optic sensors. Details of each of these experiments is presented in the following sections.

3.1 Measurement Apparatus

The apparatus and techniques common to each of the four experiments is explained and depicted in this subsection. Spectral data was acquired using a Bomem MB155 FTIR spectrometer. This spectrometer can be fitted with either of two optical tables. One of these optical tables is used to guide the IR light, using parabolic cones, into the fiber optic sensor and also to collect the emitted light as it exits the sensor (Figure 1). The second table is configured to launch the light into fiber optic cables (each 1.5m long), these cables lead to the chalcogenide sensor and back to the table, and collect the returning light back into the spectrometer's detector. This arrangement is discussed in further detail in Section 3.2.

The fiber optic sensors used in the first three experiments were 1.25 in. segments of .75 mm diam silver halide (AgBrCl) fiber. This fiber was prepared for used by shaving both ends to form a cone, similar to a pencil sharpened at both ends. A micrograph of one end of a sensor used in this program is shown in Figure 2. The fiber is held in a sensor cell during handling and mounting in the optical table (Figure 3). This sensor cell was fabricated from high molecular weight Polyethylene (PE) and two O-rings. Polyethylene was chosen because is has only a few weak absorption peaks in the IR spectral range used in these experiments and therefore will not interfere in the measurements.

The absorbent coatings were applied to the silver halide fibers by dipping them into a 1 cc solution of the polymer. Small boats were made to hold the solutions and allow dipping and coating of only the center portion of the 1 in. fibers. The ends of the fibers must remain clean to facilitate the launch and retrieval of the IR

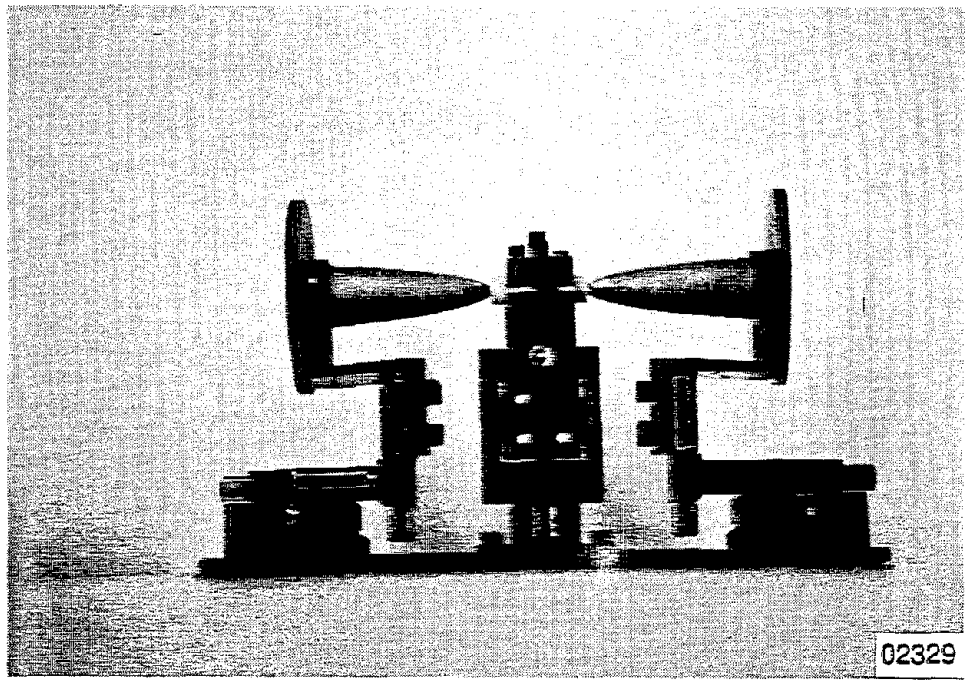


Figure 1. Optical Table Used with Silver Halide Optical Fibers

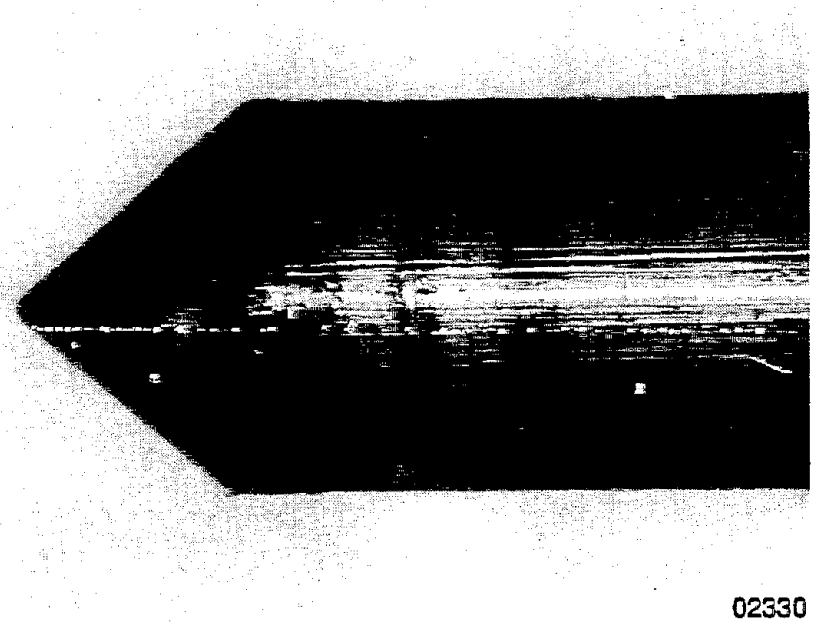


Figure 2. Optical Micrograph of the end of a Silver Halide Sensor Fiber, 75X

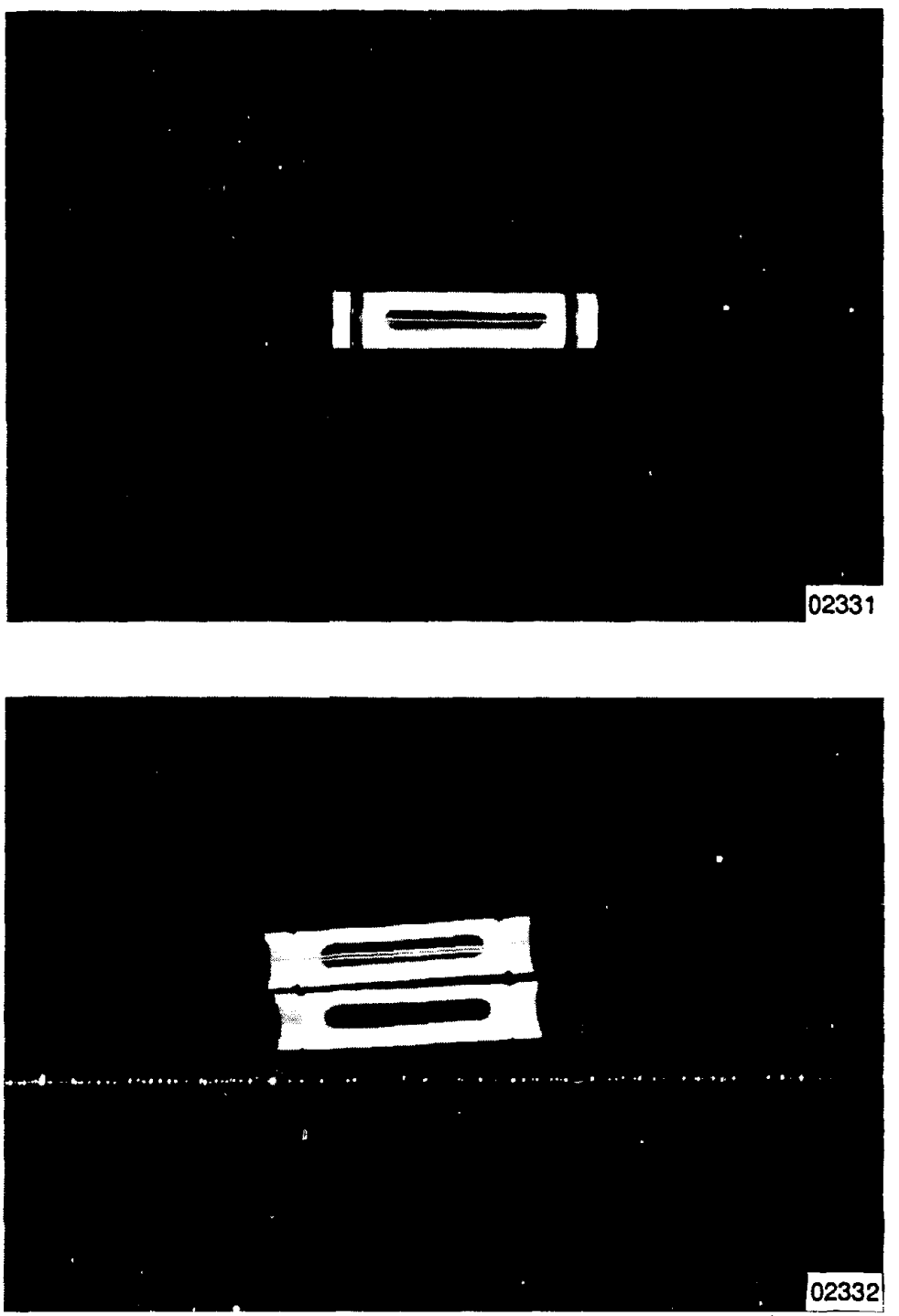


Figure 3. PE Sensor Cell used to Hold Silver Halide Fiber during Testing, Showing Assembled and Disassembled Views

light. The fibers were held, during coating, using stainless steel tubing which was pinched to fit the diameter of the fiber. Once the fiber was coated and dried it was placed into the sensor cell for the remainder of the experiment.

Appendix A contains FTIR spectra of pure benzene, xylene, polyethylene, and picric acid. Appendix A also contains some of the spectra collected using the silver halide optical fibers, not shown in the experiments below.

3.2 Summary of Experiments

Table 1 lists a summary of the experiments conducted during the Phase I effort.

3.2.1 Experiment 1 - Surface Modification of AgBrCl Fiber Using Nylon 6,6 and Picric Acid

The purpose of this experiment was to demonstrate that membranes doped with picric acid will capture and react with gaseous benzene. Furthermore, that this reactive coating could be deposited on an optical fiber that would sense the presence of benzene molecules concentrated or reacted near the surface of the fiber.

In order to quantitatively determine the exposure to benzene vapor, the coated fiber optic sensor must sense if benzene picrate is formed on its surface. The hypothesis is that as gaseous benzene molecules react with the picric acid to form benzene picrate, more benzene molecules will be able to continue to react at the surface, effectively concentrating benzene near the fiber. The presence and quantification of benzene picrate or benzene is determined by taking difference spectra between the optical fiber or plate, coated with Nylon-6,6/picric acid, and treated with and without benzene. Work was first done using silver chloride windows (flate crystals) and then with silver halide (AgBrCl) optical fibers. Flate crystals of silver chloride provided us a way to demonstrate the reactivity of the picric acid with benzene without the added complication of handling the optical fibers.

The first test performed was the dip coating of the flate crystal with picric acid. A spectra of this

Table 1. Phase I Experiments

Experiment No.	Coating(s)	Sensor Type	Vapor Sensed
1a	Nylon 66 (Vydyne) with Picic Acid	AgBrCl Fiber	Benzene
1b	Nylon 66 (Vydyne) with Picic Acid	AgCl Plate	Benzene
2	Polyethylene with Zeolite	AgBrCl Fiber	Benzene, o,m,p-Xylenes, Dichlorobenzene
2b	Polyethylene with Zeolite	AgBrCl Plate	Benzene, o,m,p-Xylenes, Dichlorobenzene
3	Nylon 6 (Elvamide) with Zeolite	AgBrCl Fiber	Benzene, Dichlorobenzene
4	Zeolite Bed	Chalcogenide	Benzene

sample was collected and compared to the spectra of the same sample after it had been dipped in pure benzene. The picric acid spectra obtained after dipping was identical to pure picric acid, therefore no reaction was detected. It is possible that the benzene picrate dissolved away into the benzene liquid as it was formed. To avoid this from happening and to generate a quality spectra of benzene picrate for comparisons later on, we altered the experiment. In the next experiment, a solution of 50 percent benzene in methanol and 5 percent picric acid in methanol was combined. The reaction mixture was heated to 73°C for 30 min, cooled and dripped on to the flate chloride crystal. Immediately crystals formed and dried on the surface. Subsequently, IR spectra where taken and compared to spectra of pure picric acid (see Figure 4). The result of the experiment was a spectra of pure picric acid, evidently no reaction occurred.

In light of these results we contacted and met with Professor Viola to discuss these findings. In summation, it does not seem feasible to adapt picrate complex formation for the purpose of concentrating any particular aromatic species onto the sensors surface. The main reasons for this are:

- The formation of picrates are nonspecific to benzene.
- The electronic nature of benzene suggest an unfavorable association equilibrium (forms unstable complex).

Therefore we concluded that a different absorbent or reactive material must be tried. In the following experiments we used a zeolite as the absorbent.

3.2.2 Experiment 2 - Surface Modification of AgBrCl Fiber Using Polyethylene Filled with Zeolite

Recent research using polyethylene coatings to absorb very dilute halogenated hydrocarbons from water (7) led us to our next experiment. The use of low molecular weight polyethylene as a porous coating has advantages over the use of Nylon. As mentioned earlier, PE has only a few very weak absorbences in the mid-IR,

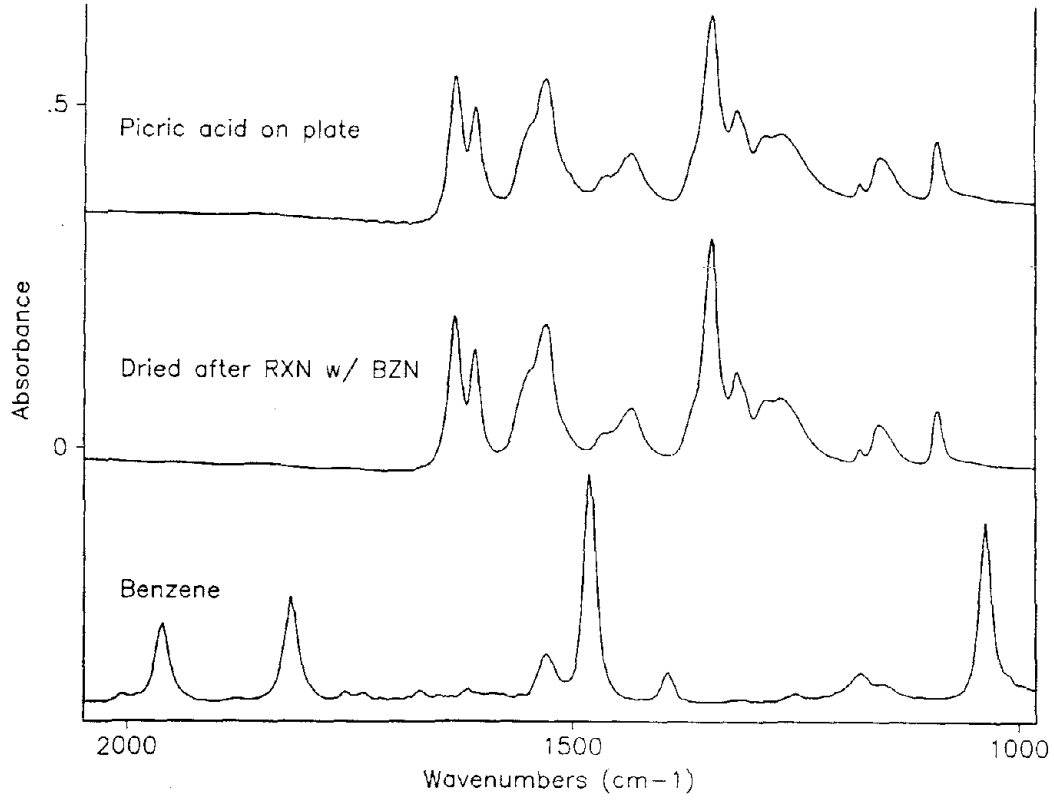


Figure 4. Spectra of Picric Acid Before and After Reaction with Benzene

therefore it will not interfere with the measurements. Furthermore, PE is hydrophobic, due to the purely hydrocarbon structure, which excludes water from contaminating the measurements. To form porous films from PE a solution of low molecular weight (~50,000 MW) PE was dissolved in Xylene at 50°C. At room temperature the polymer precipitates from solution, therefore casting was done with warm solutions held at the cloud point. The absorbent, zeolite, is added to this solution just prior to casting.

The absorbent chosen for this experiment is Silicalite S-115, a zeolite from Union Carbide. Silicalite was chosen because it is designed for absorbing hydrophobic molecules of 6Å kinetic diameter or smaller. Benzene has a kinetic diameter, or radious of rotation, of 5.85Å. For silicalite to function as an absorbent it must be activated. Activation was accomplished by heating to 350°F in an inert atmosphere or vacuum overnight to remove any water or adsorbed molecules from the pores.

The experimental procedure, using for both the flat plate and the AgBrCl fiber as IR substrates, is listed below. This is the procedure used in both experiment No. 2 and No. 3.

- Background spectra collected of the clean plate or fiber.
- Plate or fiber dipped into PE dissolved in Xylene.
- Collected spectra.
- Exposed to Benzene, or Dichlorobenzene.
- Collect spectra - Used as control experiment to measure the amount absorbed by the polymer alone.
- The fiber or plate is cleaned and dipped in PE+Zeolite in solution and dried.
- Collected spectra.
- Exposed sample to benzene of dichlorobenzene for 1 to 5 min at a vapor pressure of 95 torr.
- Collected spectra.

Initially we tried the above procedure using the flat plate of silver chloride. However, we did not detect the absorption of Benzene or dichlorobenzene. Our second attempt was done using the silver halide optical fibers. We were able to generate excellent coatings on the fibers and measure the characteristic absorptions of PE and zeolite (Figure 5). Unfortunately, when these coated fibers were exposed to benzene, the benzene did not absorb onto the coating. This was determined by the resultant IR spectra. We did notice that the characteristic xylene peaks, solvent used to dissolve the PE, was still present in the sample even after prolonged drying steps were taken.

We concluded that the xylene solvent contaminated the zeolite pores. The xylene can only be removed from the zeolite by heating to temperatures well beyond those that the PE can tolerate. These results lead us to replacing the PE+xylene solution with a methanol soluble Nylon-6,6 called Elvamide.

3.2.3 Experiment 3 - Surface Modification of AgBrCl Fiber Using Nylon 6,6 and Zeolite Membranes

In this experiment a coating that consists of Nylon-6,6 and zeolite is deposited onto the surface of silver halide optical fibers. The nylon used in this experiment is from Dupont and is a methanol soluble polymer called Elvamide™. It was selected because it readily forms membranes from methanol solution and it is the least absorptive in the IR spectra of all the nylons considered. This polymer was much easier to work with than the Vydyne used in the first experiments, mainly because it doesn't involve using acid as a solvent.

A solution of Elvamide and methanol, with Silicalite suspended in it, was prepared such that the total solids content in methanol was 24 percent by weight (solids were 50 percent Elvamide and 50 percent Silicalite by weight). The fiber was dipped and dried, to yield a uniform dull white coating. We also prepared control samples. The control fibers were coated with only the nylon. Appendix A contains FTIR spectra of these Elvamide and zeolite coated fibers. The measurement procedure was the same as in experiment No. 2. The coated fiber

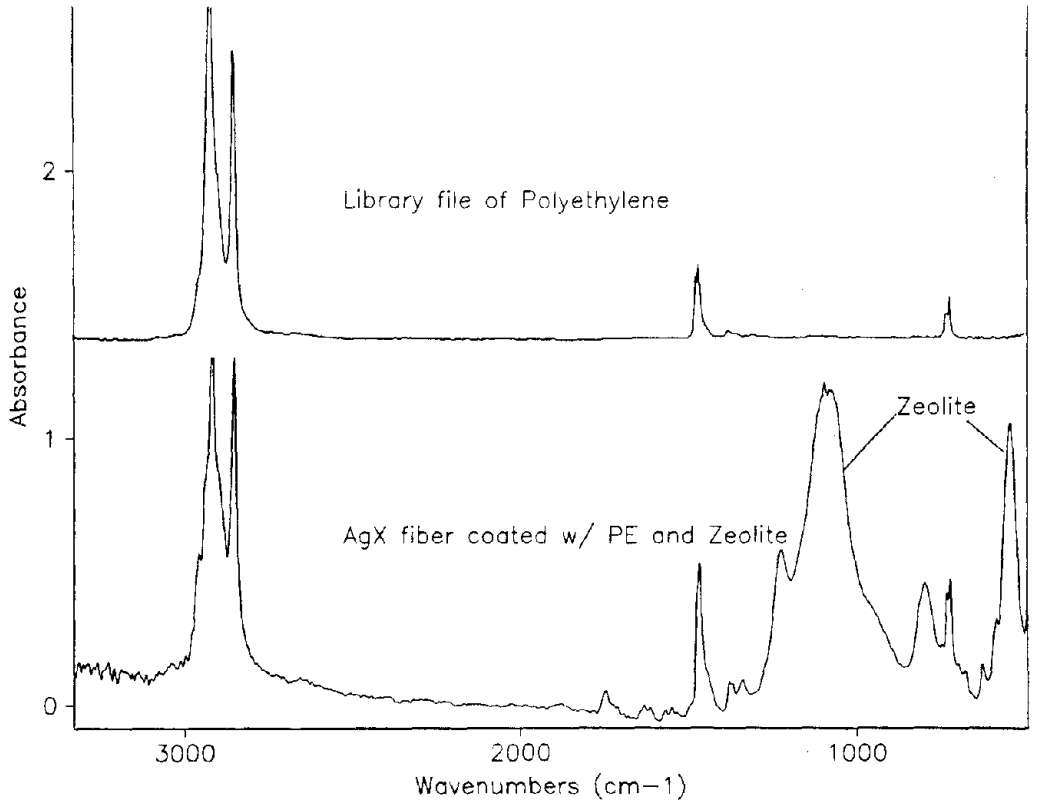


Figure 5. Spectral Plot of a Polyethyene/Zeolite Coated Silver Halide Fiber

was exposed to benzene by placing it into a sealed vial that contained liquid benzene trapped in glass wool. The benzene soaked wool was packed on the bottom of the vial and the sensor fiber was placed on top of the wool separated by a dry barrier. The vial was kept at 75°F, which corresponds to a benzene vapor pressure of 95 torr. The samples prepared in this way were expected to have the maximum amount of vapor the coating will absorb. Following exposure to benzene or dichlorobenzene, the fiber was mounted in the fixture and spectra were collected immediately and at subsequent time intervals. In Figure 6 a series of scans from the time of exposure to days later is shown for the control sample. Figure 7 shows a trend plot for the absorbance data in Figure 6. Figure 8 shows a series of scans from the time of exposure to days later for the zeolite containing sample exposed to benzene. The peaks shown are those which are characteristic of benzene. The next (Figure 9) shows the trendline plot of these peaks verses time. A similar series of graphs for dichlorobenzene are in Figure 10 and Figure 11. These samples were exposed to saturated vapor pressures for each benzene (95 torr @ 75°F) and dichlorobenzene (75 torr @ 75°F). These vapor pressures translate into ~100,000 ppm, which are relatively high concentrations. Take note that both benzene and dichlorobenzene could be detected and differentiated using the same sensor. This is possible because nearly all organic compounds have unique absorbance peaks or combinations of peaks.

To examine the sensitivity of this sensor design to lower concentrations an apparatus composed of a bubbler and mixing tubes was fabricated. This device allowed the mixture of dry air with benzene saturated air, to afford a range of concentrations from 10 ppm to 100,000 ppm. Figure 12 shows a schematic and photograph of the entire measurement setup we used to perform the low concentration experiments.

Benzene vapor (1,000 ppm) in air was passed over a Elvamide+ Silicalite coated silver halide fiber for 13 hr. Spectral data were collected every hour and compared to a reference spectra. The spectral data did not show the absorption of benzene with time. Evidently, this particular coating did not concentrate enough benzene

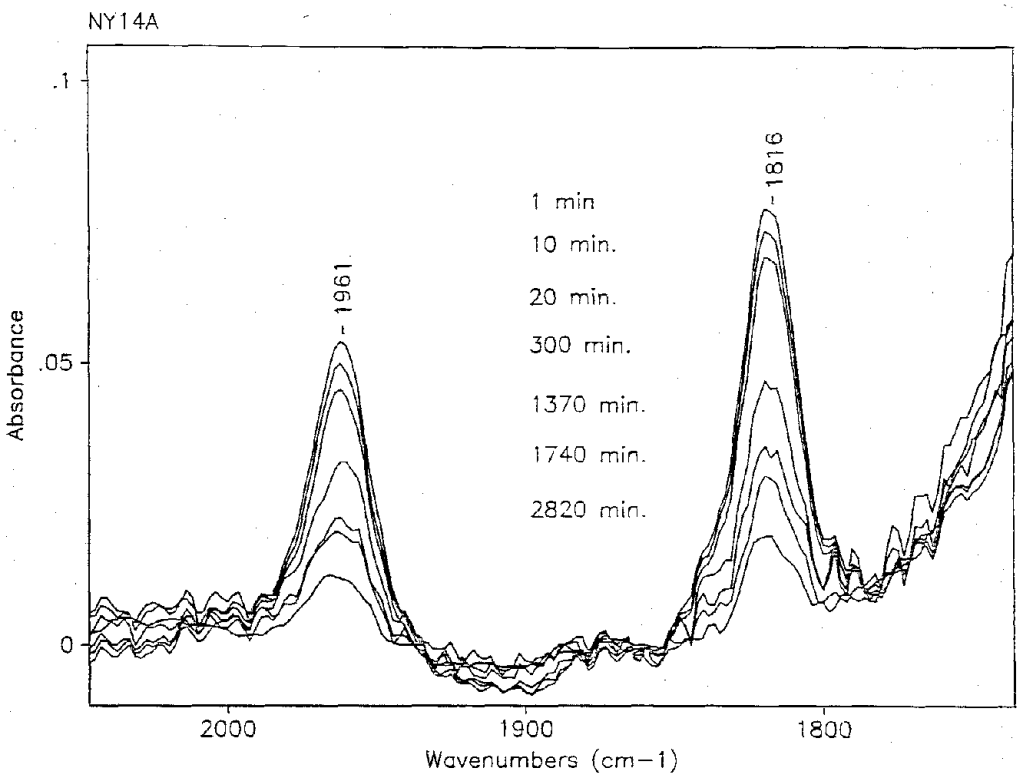


Figure 6. IR Spectra Showing Benzene Absorbance Peaks as a Function of the Time Passed Since Exposure (control)

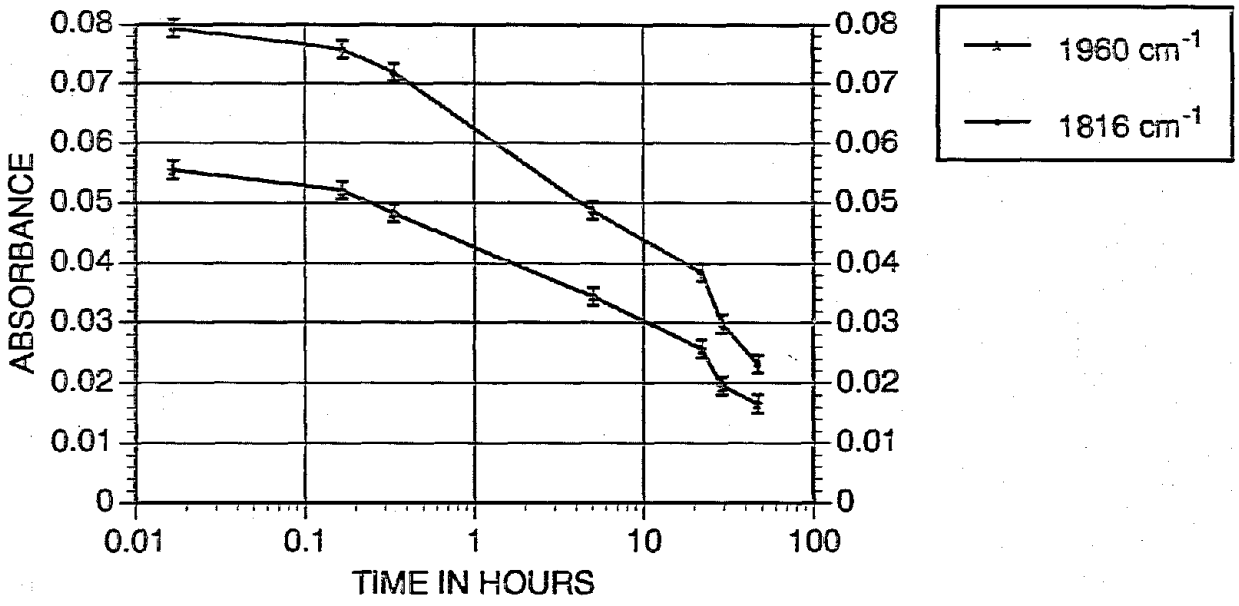


Figure 7. Trend Plots for Two Benzene Peaks versus Time (control)

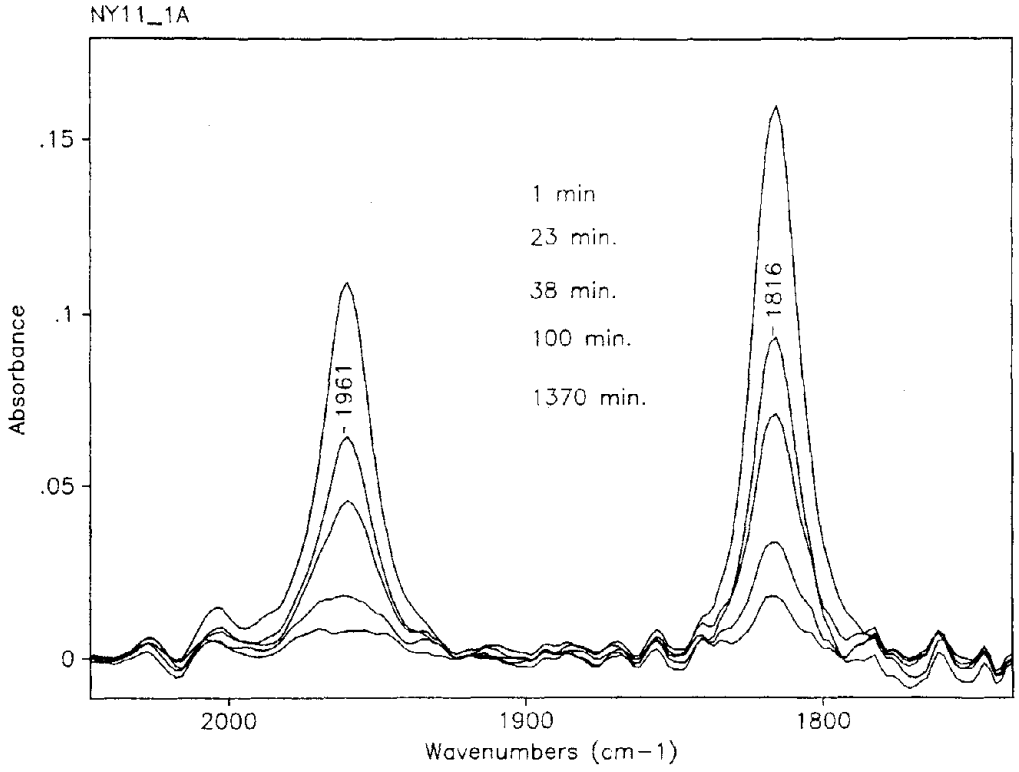


Figure 8. IR Spectra Showing Benzene Absorbance Peaks as a Function of the Time Passed Since Exposure for the Zeolite Containing Sample

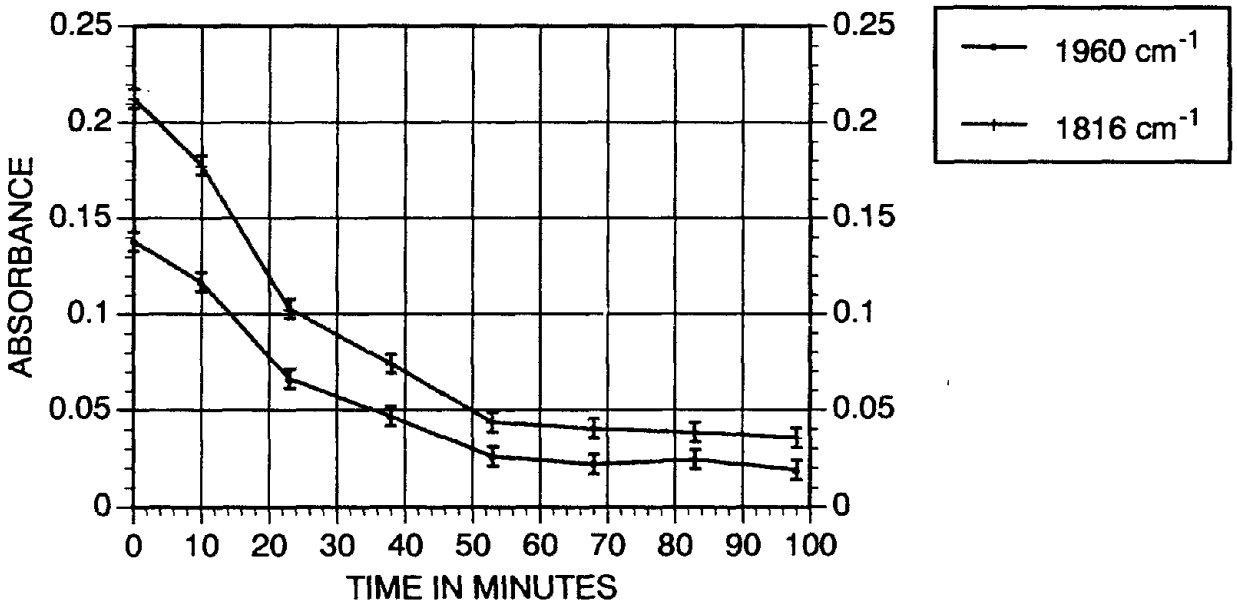


Figure 9. Trend Plots for Two Benzene Peaks versus Time for the Zeolite Containing Sample

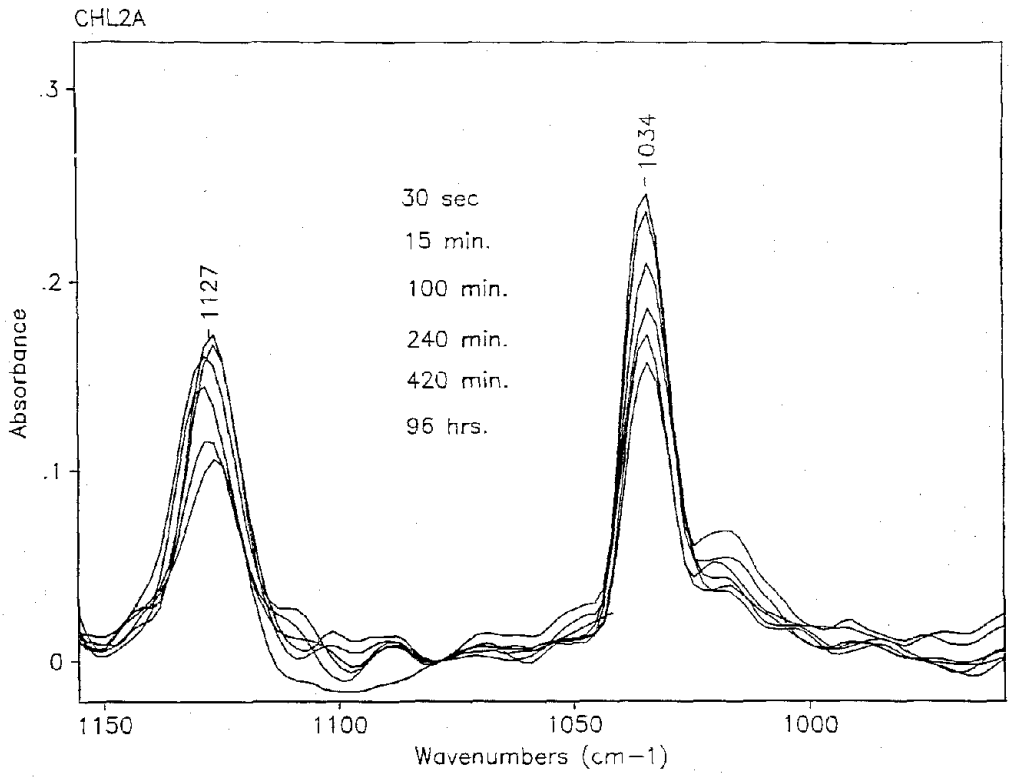


Figure 10. IR Spectra Showing Chlorobenzene Absorbance Peaks as a Function of the Time Passed Since Exposure

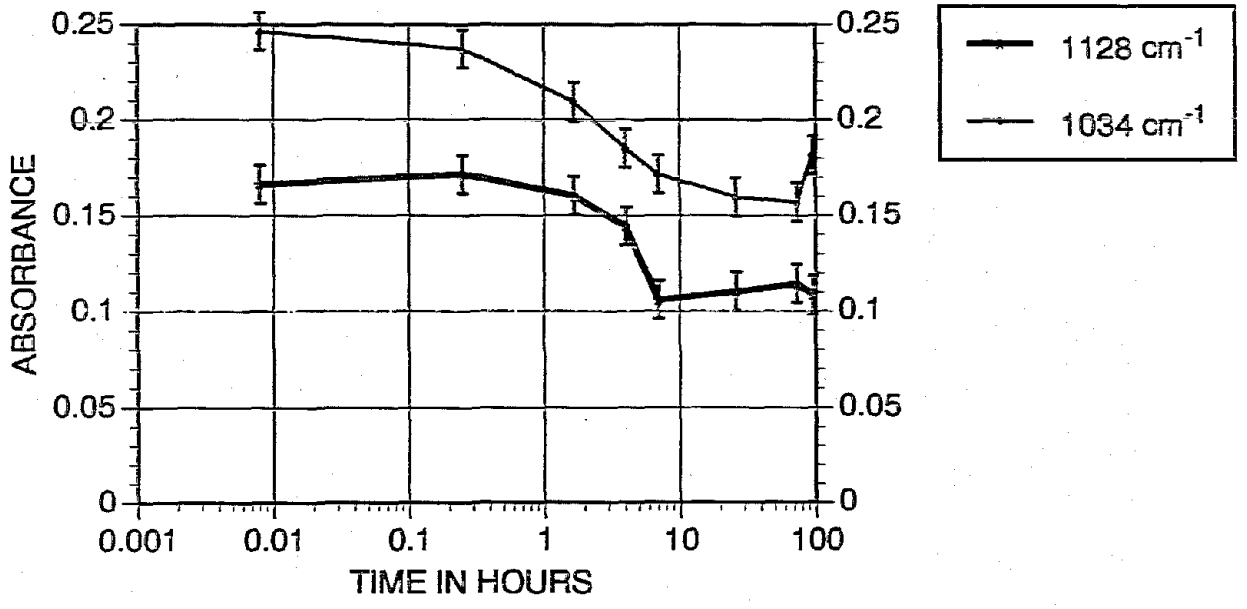
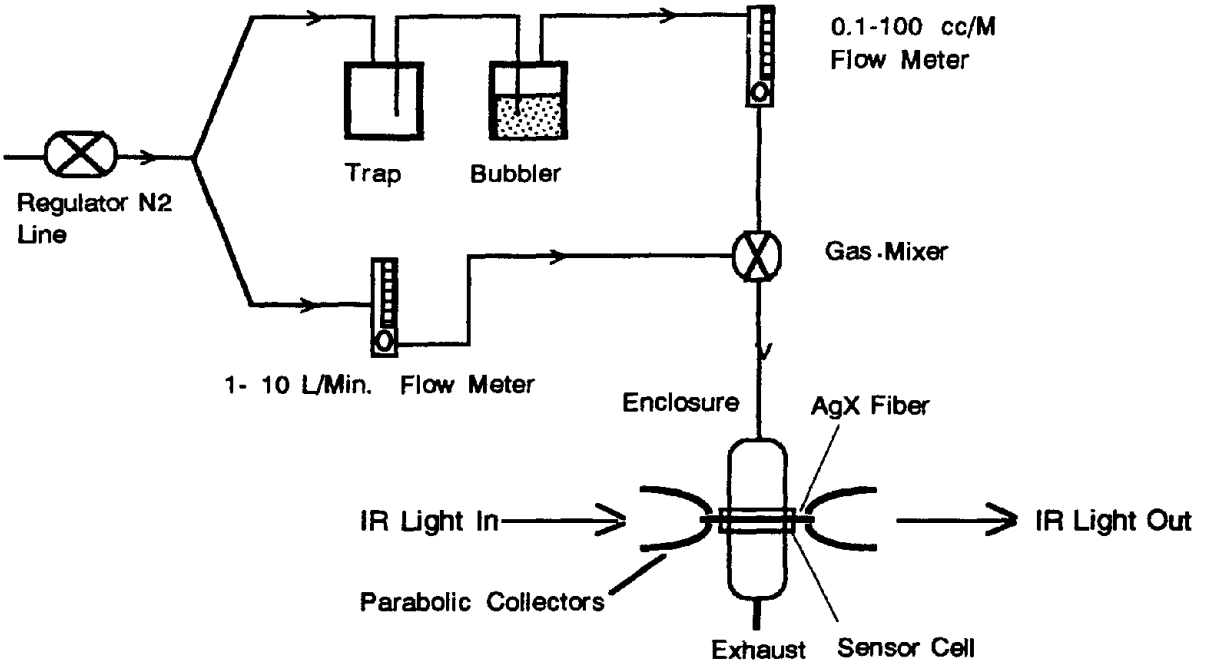


Figure 11. Trend Plots for Two Chlorobenzene Peaks versus Time



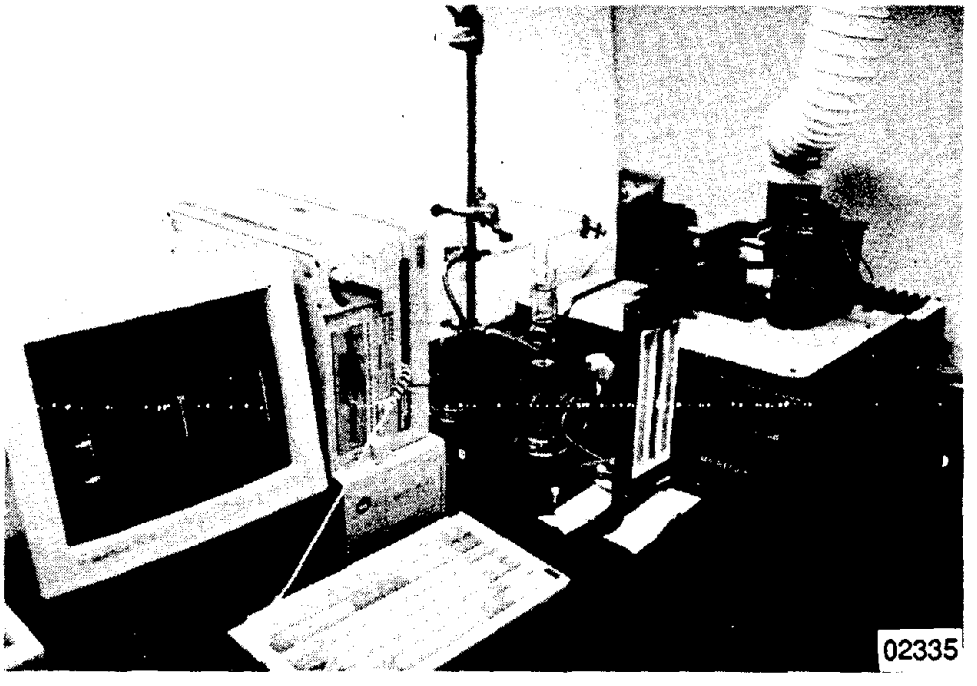


Figure 12. Schematic and Photograph of Apparatus Used to Generate Low Concentrations of Benzene in Air

near the surface of the fiber at 1,000 ppm for it to be detected. Since in experiment No. 2 we observed that benzene is absorbed by the zeolite, a fourth experiment was designed that used only pure Silicalite zeolite as the coating. This experiment No. 4 is discussed in subsection 3.2.4.

3.2.4 Experiment 4 - Surface Modification of Chalcogenide Sensor with Zeolite Packed Bed

In this experiment, the same Silicalite was activated and packed around a tapered chalcogenide sensor. The sensor is housed in an aluminum boat that has a wide tube crossing over it. Figure 13 and Figure 14 show a schematic and photograph of the apparatus, respectively. This tube was used to contain the benzene-air atmosphere, so that the gases could pass slowly over the bed of silicalite. The tapered fiber laid partly embedded in the silicalite. This was done because the depth of penetration of the benzene into the silicalite bed was unknown.

The experiment was performed by first collecting background spectra of the clean fiber and the fiber embedded in clean silicalite. Then the gas flow was started, at first a concentration of 1,000 PPM benzene in air was passed over the sample. Spectra were collected hourly for 16 hr. However, no benzene was detected so we increased the concentration to 100,000 ppm or 10 percent by volume. This time benzene was detected and the absorbance increased with exposure time, a spectral plot and trendline plot is presented in Figures 15 and 16.

3.3 Conclusions

The use of activated zeolite (Silicalite S-115) demonstrated that concentrations of 100,000 ppm benzene in air could be detected with IR fibers. However, the benzene content in the zeolite would gradually decrease once the sensor was removed from the benzene atmosphere.

Most of the technical objectives were accomplished, however, the primary objective to detect low concentration (1 to 100 ppm) benzene in air was not successfully demonstrated. Attempts at measuring 1,000 ppm did not succeed using the fiber coatings investigated in this effort. The application of picric acid as a benzene gas absorbent/reactant was unsuccessful. We have found that picric acid does not readily form benzene-picrate during gas phase reactions. In

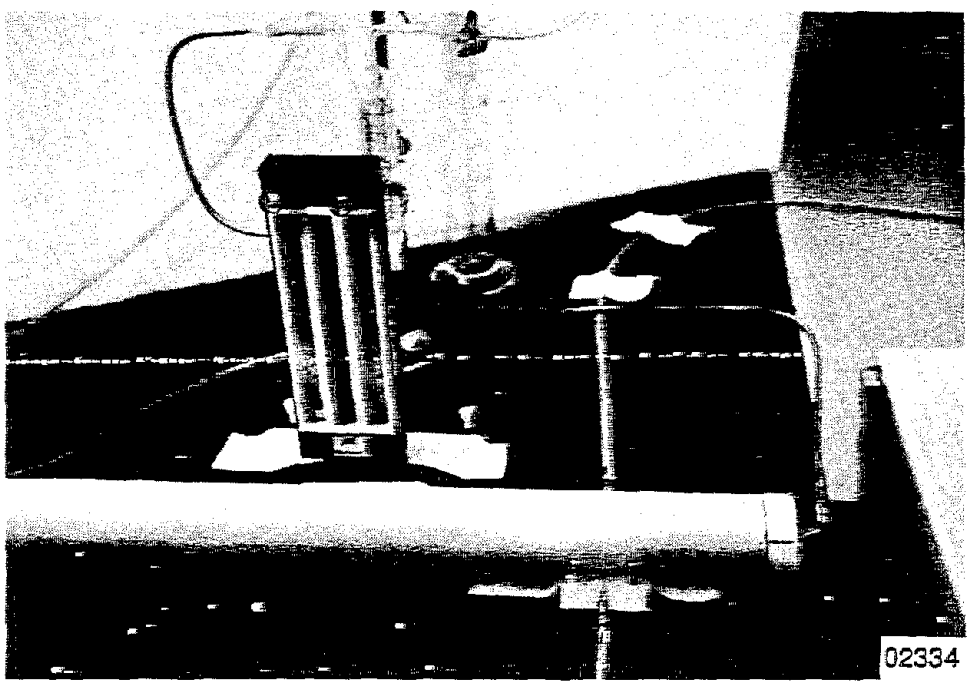


Figure 13. Photograph of Apparatus Used in Experiment No. 4

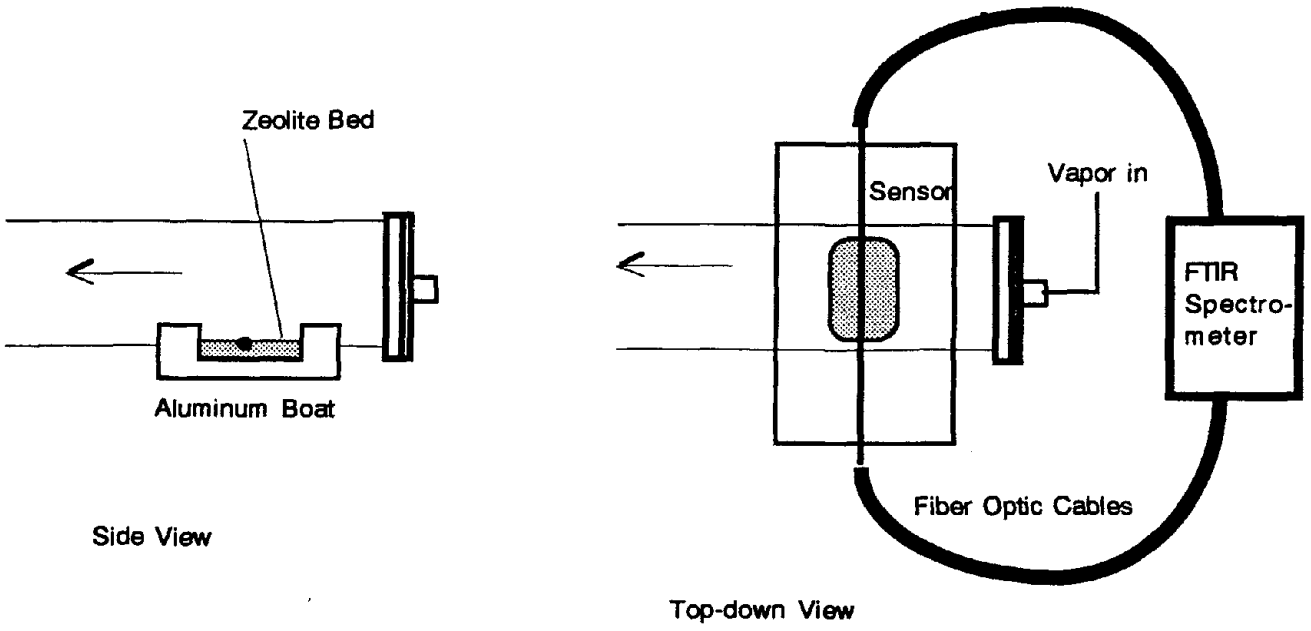


Figure 14. Schematic of Apparatus Used in Experiment No. 4

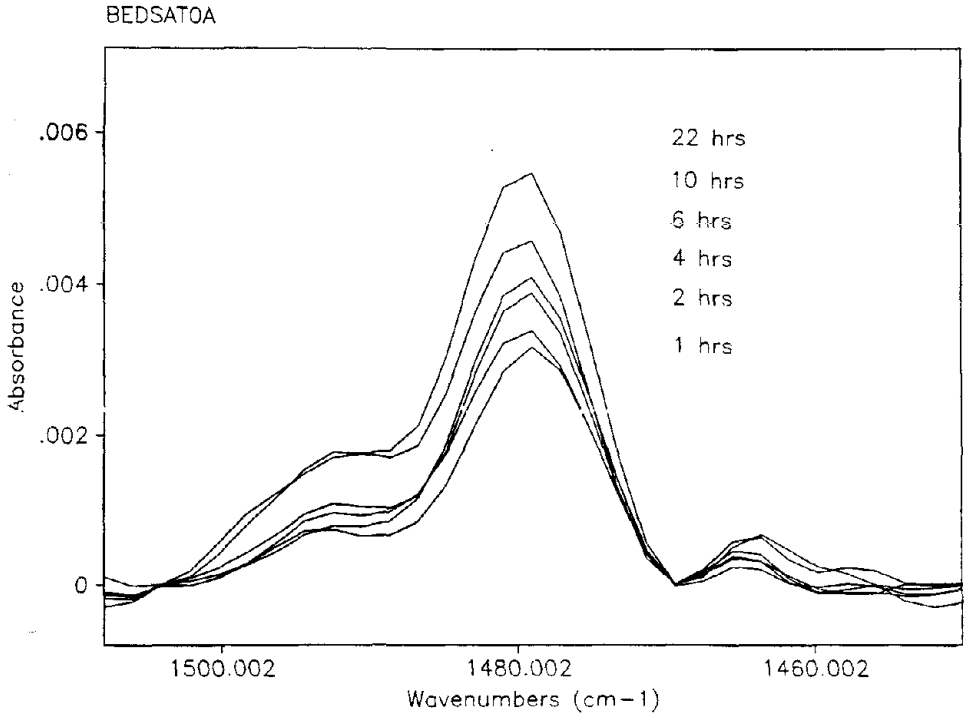


Figure 15. Spectral Plot of Benzene Absorption Peak as a Function of Exposure Time

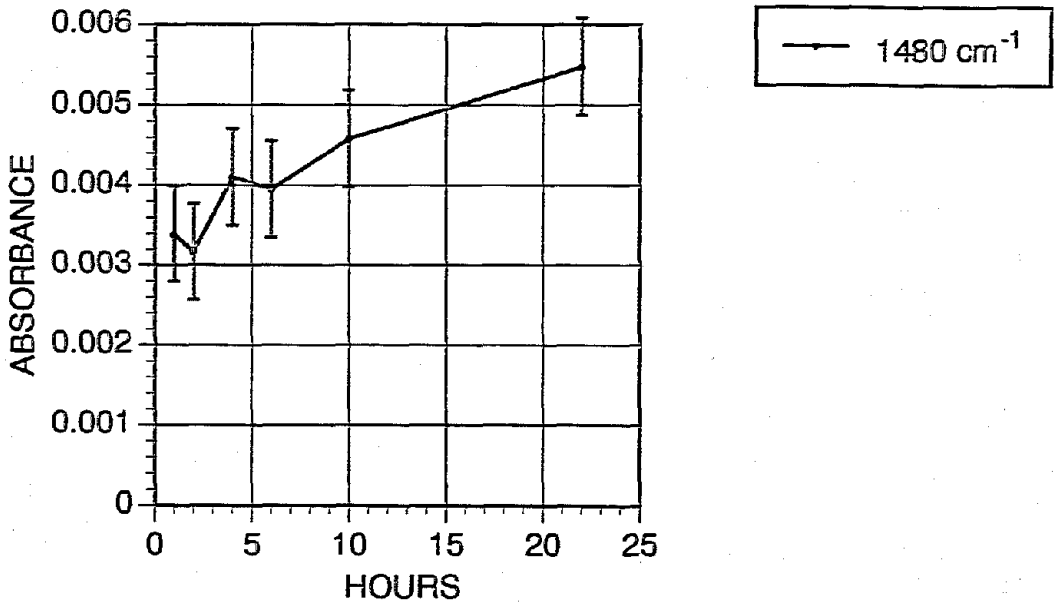


Figure 16. Trendline Plot of Benzene Peak Absorbance as a Function of Time

some cases it took weeks for all the benzene to evolve from the sensor surface. Below are listed the technical objectives that were met.

- Demonstrated that benzene (100,000 ppm) and dichlorobenzene (80,000 ppm) could be detected using Silicalite S-115 as an absorbent in a nylon membrane.
- Demonstrated uniform membranes of nylon and polyethylene could be cast onto silver halide optical fibers.
- Determined the relative saturation capacity of zeolite coated fibers.
- Demonstrated the use of tapered chalcogenide fiber optic sensors as gas sensors.
- Demonstrated that dichlorobenzene can be detected and differentiated from benzene by unique absorbance peaks.
- Picric acid is not reactive toward benzene in the gas phase.
- Developed a small PE holder, that allows handling, that does not interfere with measurements in the mid-IR.

4. PUBLICATIONS

"Detection of Aromatic Hydrocarbons with IR Evanescent Wave Fiber-Optic Sensors," M.A. Druy, P.J. Glatkowski, P. Marinaccio, and W.A. Stevenson, SPIE Symposium on Chemical, Biochemical, and Environmental Fiber Sensors, September 1992.

5. ACKNOWLEDGMENTS

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7. "A New IR Fiber Optic Physico-chemical Sensor for Chlorinated Hydrocarbons in Water," R. Krsha, K. Taga and R. Keller, Institute for Analytical Chemistry, Technical University of Vienna.

APPENDIX A

REFERENCE SPECTRA OF DICHLOROBENZENE, BENZENE, XYLENES, LOW DENSITY POLYETHYLENE, NYLON 6,6; AND NYLON 6,6 CAST ON AgBrCl FIBER

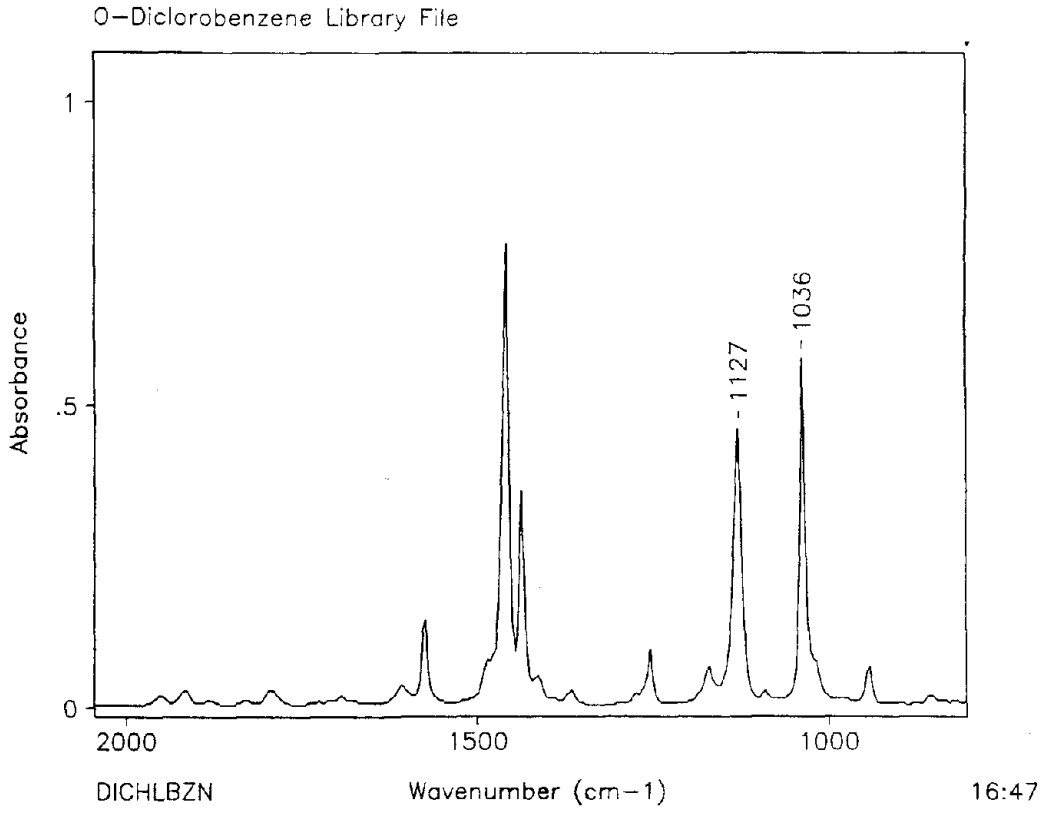

Figure A-1.

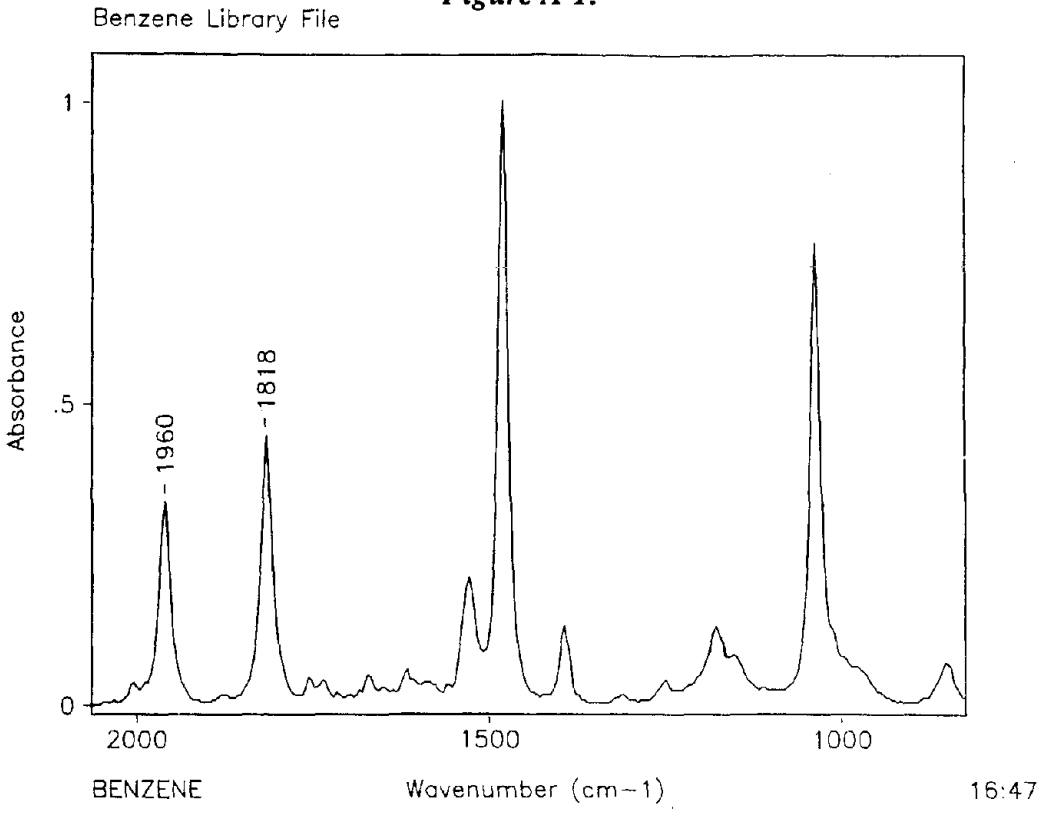

Figure A-2.

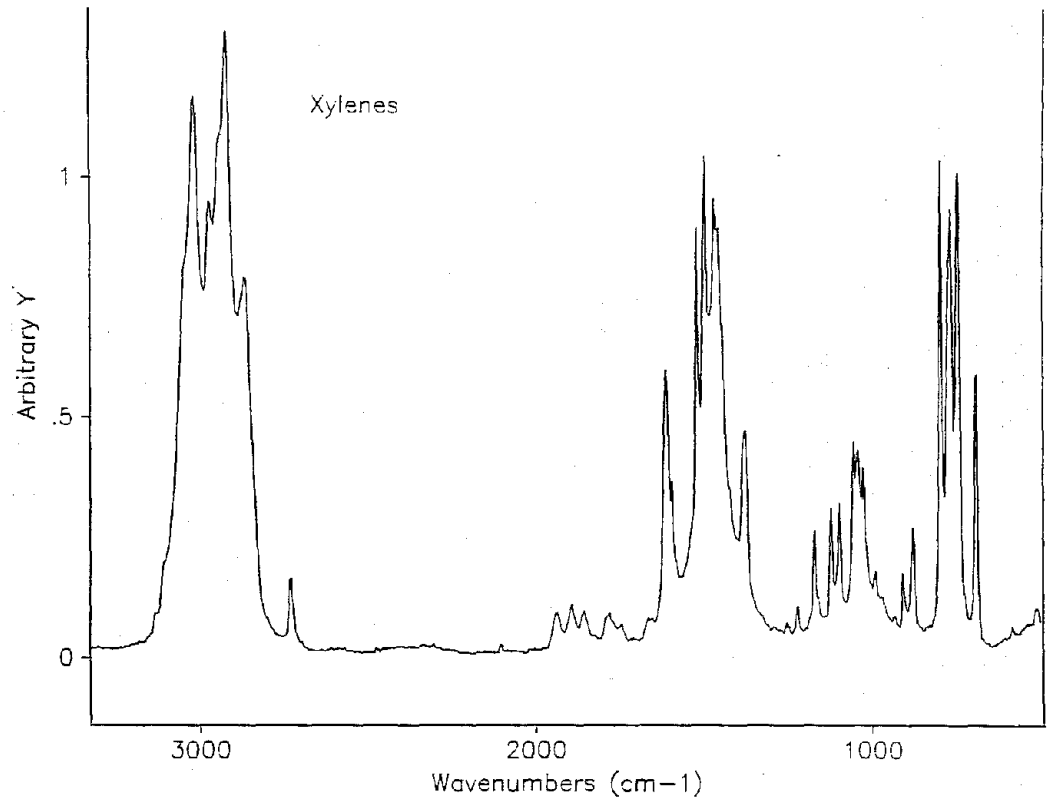

Figure A-3.

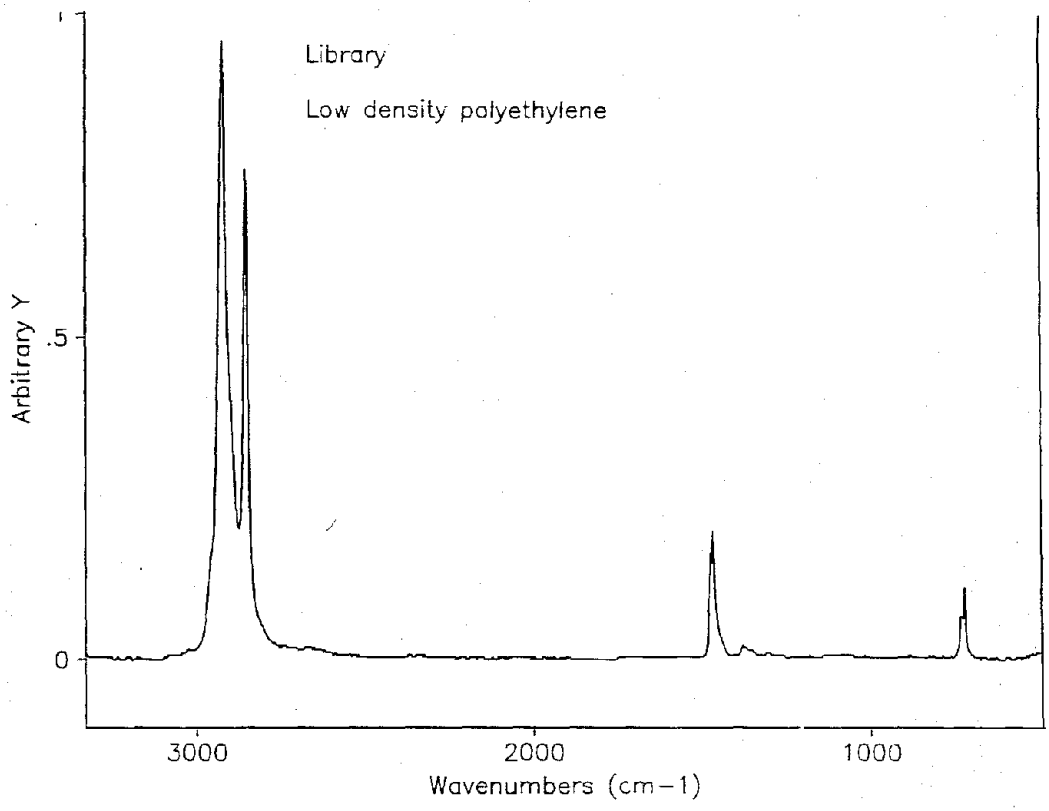

Figure A-4.

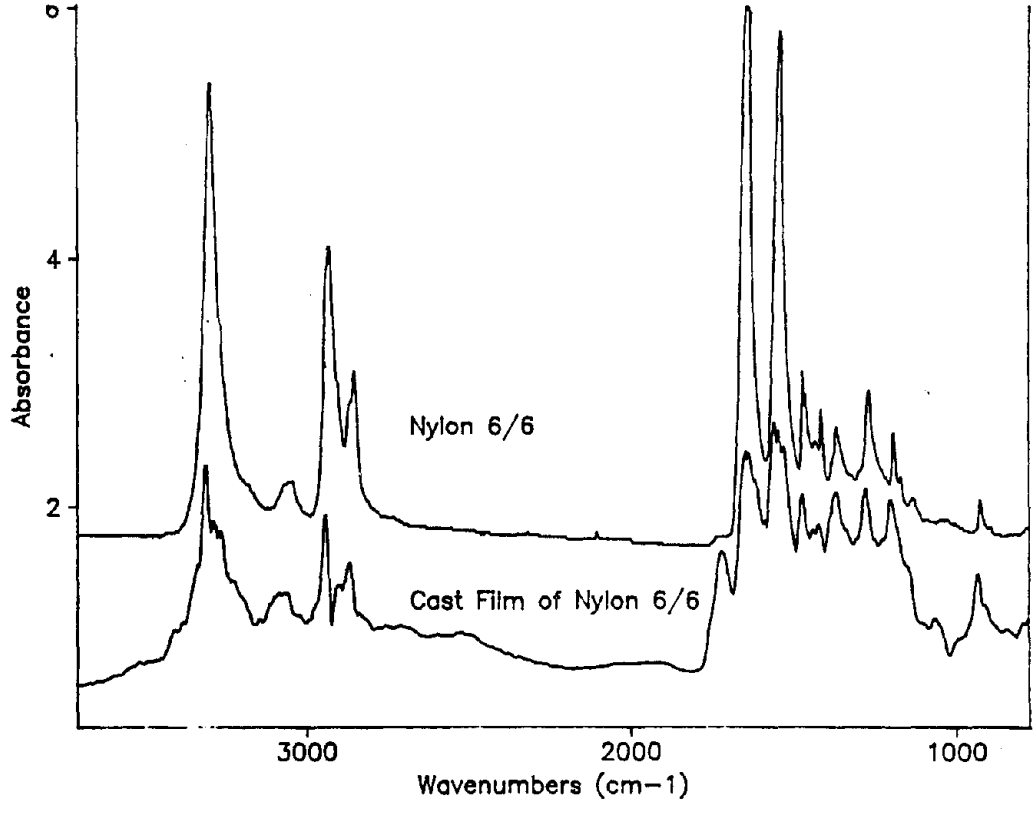

Figure A-5.

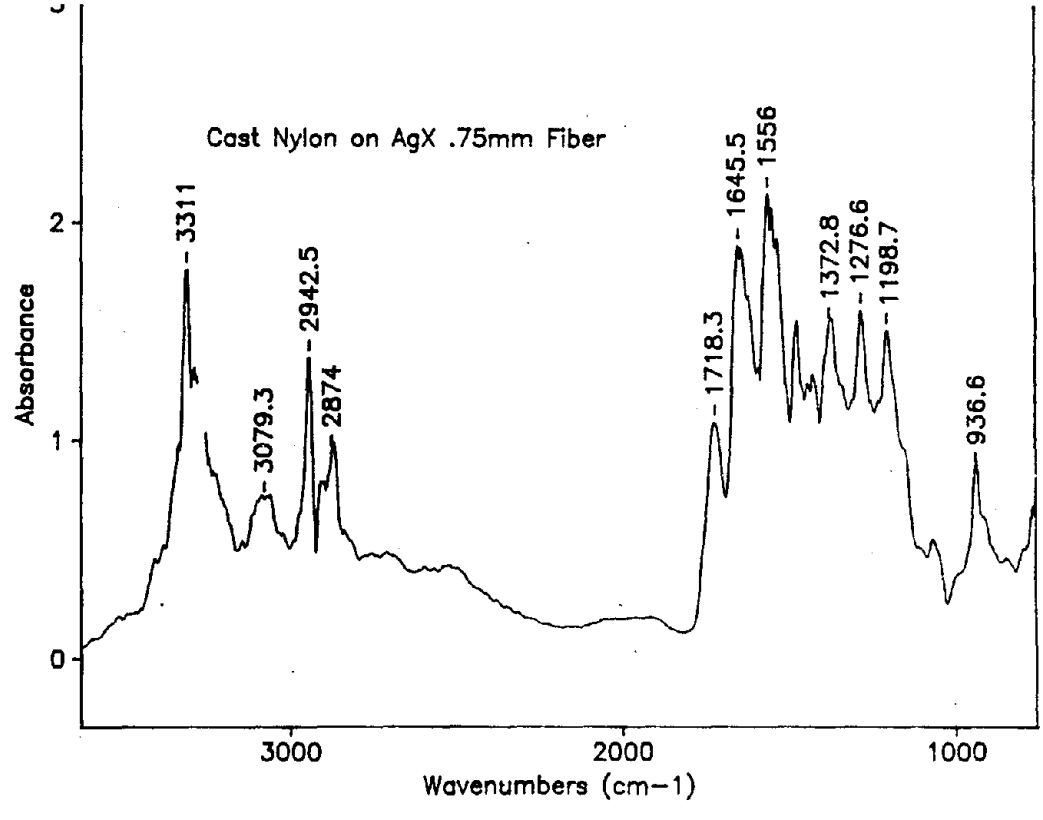

Figure A-6.