Monitoring the effects of indoor pollution using Fourier transform infrared spectroscopy

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Abstract: Fourier Transform Infrared Spectroscopy (FTIR) was used to develop an effective way to monitor indoor pollution and to observe the effects of such indoor pollution under various conditions of the ventilation system of McCollum Science Hall of the University of Northern Iowa, Cedar Falls, Iowa. This was done by monitoring the absorbance of acetone as it evaporated over time, and the data were used to create concentration vs. time plots. Several parameters from these plots were used to judge the ventilation system, with some general conclusions including that having the system on reduces peak time and end time. Also, the ventilation system in the organic stockroom appears to be less efficient than in other areas, and the ventilation system in the organic lab appears to be more efficient compared to other areas. The method developed and used during this research is an effective way in which to monitor indoor air pollution and could be employed in other buildings.
Introduction and Background: The purpose of this research was to determine an effective way to monitor the indoor air pollution that may occur in a chemistry facility on a daily basis, and to make conclusions about the ventilation system of McCollum Science Hall of the University of Northern Iowa, Cedar Falls, Iowa. This was accomplished by using a volatile organic compound and determining the amount of time that it took for the compound to dissipate in selected rooms of McCollum Science Hall. This type of research is especially important in a science building where hazardous chemicals are handled daily, since each encounter with these chemicals has the potential to lead to a spill. A student may face significant health risks without a proper ventilation system so that determining the efficiency of the ventilation system of McCollum Science Hall was meaningful to many individuals.

The research was done using infrared spectroscopy, which is the study of spectra produced when a compound absorbs infrared radiation. The absorption of infrared radiation occurs due to the vibration of molecule, which causes a change in the dipole moment. A dipole moment occurs whenever the electrons involved in a chemical bond are shared unevenly. This uneven distribution of electrons is seen in all molecules except for homonuclear molecules, or those that consist of identical atoms. The vibration of a molecule is continuous, so that regular fluctuations in the dipole moment are established. These fluctuations produce an energy field around the molecule with a certain frequency. When radiation hits the molecule with a frequency exactly matching that of the energy field, the molecule absorbs the radiation. The frequencies of energy caused by molecular vibration correspond to the frequencies of infrared radiation, leading to the type of spectroscopy used to analyze chemicals in this manner.

The vibrations that are possible in a molecule are grouped into two categories: stretching and bending. The stretching vibrations are either symmetric or asymmetric and the bending vibrations include in-plane rocking, in-plane scissoring, out-of-plane wagging or out-of-plane twisting. The types of vibrations possible for a particular molecule depend on the chemical bonds that are present in the molecule. For molecules with more than one chemical bond, interactions between vibrations of the individual bonds occur, leading to variations in the vibrations. Each combination of vibration type and chemical bond corresponds to a particular frequency. The differences in chemical bonds that exist
between molecules mean that all molecules absorb a unique set of frequencies. This unique spectrum, or "fingerprint," that is produced for each chemical makes infrared spectroscopy well suited for qualitative studies.

Quantitative measurements can also be made using infrared spectroscopy. Such measurements require the use of Beer's Law, which states

\[ A = \varepsilon b c \]

In the equation, \( A \) represents the absorbance of a compound at a certain frequency, \( \varepsilon \) represents a proportionality constant, \( b \) represents the path length through the sample and \( c \) represents the sample concentration. The instrument used must first be calibrated, which determines the value of the proportionality constant. Once calibration is accomplished, Beer’s Law may be used to determine an unknown concentration by measuring the absorbance of the sample with a spectrometer.

The instrument used during research was a MIDAC M Series Fourier Transform Infrared Spectrometer. FTIR spectroscopy is a specific type of infrared spectroscopy, which makes use of a mathematical equation, called the Fourier Transform, which states that

\[ l(v) = \int_{-\infty}^{+\infty} i(x)[\cos 2\pi vx - i \sin 2\pi vx] \, dx \]

In the equation, \( l(v) \) represents the intensity of the radiation as a function of mirror displacement (time), \( i(x) \) represents the intensity of the radiation as a function of frequency, \( v \) represents frequency and \( x \) represents mirror displacement. The instrument is composed of three main components: the source, the telescope and the interferometer, specifically a Michelson interferometer. The source provides infrared red radiation when a metal bar in the center of a mirrored surface is electrically heated (see Figure 1). The telescope is a large scope with mirrored surfaces on the inside, which serves to focus the beam of infrared radiation. The interferometer, which sits on top of the telescope, is the component that differentiates Fourier Transform IR spectroscopy from conventional IR spectroscopy (see Figures 2 and 3).
The interferometer contains two important parts. The first is a beamsplitter (see Figure 4). The beamsplitter is a device that divides the beam of infrared radiation into two components, which allows an interference pattern, or interferogram, to be created. This interferogram makes it possible for frequency data to be converted to time data. The interferogram is made when the beamsplitter transmits approximately half of the incoming infrared beam, and reflects the other half. This means that one half is reflected by a stationary mirror, while the other half is reflected by a moving mirror. These two beams are then recombined at the beamsplitter and sent to the detector. The movement of the second mirror varies the difference in the path length of the two beams, which leads to the interferogram. The second crucial part of the interferometer is the driving mechanism that controls the movement of the mirror. This mechanism must be precise in order for the position of the mirror to be known exactly at all times, requiring constant speed and planarity.

Advantages of FTIR spectroscopy over conventional spectroscopy are numerous. The advantage most relevant to this research is that an open-path technique is possible with FTIR spectroscopy. This technique allows for the chemical of interest to be in the atmosphere between the source and the remaining components of the spectrometer, rather than being confined to a sample container. This is beneficial because it allows for real-life data, and for the relatively easy relocation of the spectrometer into various rooms that were studied. Fourier transform instruments also have fewer optical components than conventional instruments. This means there is less reduction of the signal than what occurs in conventional instruments, giving more accurate absorbance values. Each spectrum obtained is actually the average of a number of spectra gathered, which also contributes to a more accurate reading.

The final significant advantage of FTIR spectroscopy is what makes it different from conventional spectroscopy, namely the use of the Fourier Transform and the interferogram. The data from the interferogram is put through the Fourier Transform, which changes the information from time data back into frequency data. However, now a wide range of frequencies reaches the detector simultaneously. This greatly reduces the amount of time required to create a complete spectrum, from 2-3 minutes for a conventional IR spectrometer to 10-15 seconds for a FTIR spectrometer.
Experimental Details: The first task was to choose a chemical to use during research. Many chemicals were tested, including hexane, acetone, benzene, ethanol and butyl alcohol. Acetone was chosen due to the reasons that follow. First, acetone has a low toxicity, which is important since those involved in the research were exposed to the chemical daily for many weeks. Secondly, acetone evaporates quickly, which allowed for more trials to be done over the course of the research. Next, acetone is fairly inexpensive and readily available. Finally, acetone has three absorbance peaks in the infrared region that are very consistent and easy to identify.

Gathering data consisted of setting up the instrument in the room being studied and spilling 300mL of acetone on the floor between the source and the remaining components. This was done under various conditions of the ventilation system and in several rooms of McCollum Science Hall. Two or three trials were done for each condition set and care was taken to spill the acetone in the same location for each trial. The first spectrum was obtained at the time of the spill (time 0), and thereafter every minute until the acetone peak at $1217\text{cm}^{-1}$ was no longer detectable (see Figure 5).

Before the spectrometer was used to look at acetone, a background spectrum had to be obtained (see Figure 6). This was necessary due to the large amounts of water and carbon dioxide in the air, which absorb at many frequencies in the infrared region. Once the background spectrum was scanned, the instrument subtracted this data out of the sample spectra automatically. Baseline corrections were also necessary in order to flatten out the baseline and determine its absorbance value. The baseline value was then subtracted from the absorbance value of acetone at $1217\text{cm}^{-1}$. This correction was done using the program GRAMS 386, the same program used to obtain spectra. The resulting absorbance values were then plotted against time for each trial (see Figure 7).

The next part of the study involved calibrating the instrument. This required a sealed room with a determinable volume. These requirements were met by the paint booth facility at the Iowa Waste Reduction Center in the Cedar Falls industrial park. The paint booth is a small room that could be entirely sealed off and had a ventilation system that could be turned on and off, allowing the room to be airtight for trials and evacuated afterwards to facilitate quick calibration. The volume was determined to be $84.01\text{m}^3$. The
FTIR spectrometer was set up inside the paint booth, and known volumes of acetone were evaporated using a hotplate. The hotplate was used so that the entire volume of acetone could be placed in the air rapidly, and a fan was used to distribute the acetone evenly throughout the paint booth. A spectrum was then taken for each volume of acetone evaporated.

Using the temperature and barometric pressure, the air density was determined for each day calibration trials were done. The concentration of acetone in the paint booth for each volume evaporated, was determined using the equation

\[
C = \frac{d_{\text{acetone}}V_{\text{acetone}}}{d_{\text{acetone}}V_{\text{acetone}} + d_{\text{air}}V_{\text{air}}}
\]

In the equation, \(C\) represents the concentration of acetone in parts per million (ppm), \(d_{\text{acetone}}\) and \(d_{\text{air}}\) represent the density of acetone and air, respectively, and \(V_{\text{acetone}}\) and \(V_{\text{air}}\) (volume of the paint booth) represent the volume of acetone and air, respectively. By plotting absorbance of acetone at \(1217\text{cm}^{-1}\) against concentration of acetone times the path length, a calibration curve was obtained (see Figure 8). The slope of this curve is equal to the proportionality constant in Beer's Law. Three sets of data were obtained and the slope of each calibration curve was determined using the program Excel. The value of the proportionality constant, or the absorptivity, was used to convert the absorbance vs. time graphs to concentration vs. time graphs (see Figure 9).

**Results and Conclusions:** The average value of the absorptivity of acetone at \(1217\text{cm}^{-1}\) was 0.008ppm·m\(^{-1}\), which was compared to the literature value of 0.005ppm·m\(^{-1}\). One explanation for the considerable difference in the two values is that the experimental absorptivity was determined using a resolution of \(4\text{cm}^{-1}\), while the literature absorptivity was determined using a resolution of \(2\text{cm}^{-1}\). Other factors that may have caused the difference in values include an uneven distribution of the acetone due to unavoidable air currents in the paint booth, or unknown leaks in the paint booth. Care was taken to prevent these problems, but it is possible they still occurred. The experimental value was seen as acceptable since the difference expected due to the difference in resolution, and an absorptivity of 0.008ppm·m\(^{-1}\) was used in further calculations.
Beer's Law was used to convert every absorbance value into a concentration, and concentration vs. time plots were created for each data set. These plots were set to a curve of the form \( y = a + b \cdot e^{-(\text{time}/c)} \) using the program TableCurve. This allowed for numerical parameters to be obtained and used in comparisons. For each data set, the curve concentration = \( a + b \cdot e^{-(\text{time}/c)} \) was fit, and the parameters \( b \) and \( c \) and the quotient of the two were recorded. These parameters gave an indication of the steepness of the curve, or the rate at which the acetone was removed from the room. In general, the quotient \( b/c \) increases as the curve becomes steeper. However, there are exceptions to this rule, which is the reason for considering the \( b \) and \( c \) parameters individually as well as the quotient. These values, along with end time, peak time and peak concentration, are given in Table 1.

A general trend seen in the data is that the acetone evaporated more quickly when the ventilation system was on compared to when the ventilation system was off. This was indicated by a decrease in end time, which was seen in all rooms studies, and a decrease in peak time and peak concentration, which was seen in most rooms studied. This trend validates the conscientious use of the ventilation system whenever harmful chemicals are being handled.

Some differences were noted between the peak time and end time for different rooms. These differences were most likely due to the varying volumes of the rooms, although large differences indicate a variance in the efficiency of the ventilation system among rooms. The slight differences were not a concern due to the numerous factors affecting the performance of the ventilation system. These factors include changes in air circulation due to doors being opened and closed, indoor and outdoor temperatures, pressure changes and the time of day. Effort was made to control these variables, but that was not always possible, meaning those small differences in values, even between trials under the same conditions, were expected and seen.

Large differences between the values reported in Table 1 are significant, such as the difference between the organic stockroom data and the data from other rooms. The peak time and end time in this room were much larger than those seen in other rooms under the same ventilation system condition (on). This indicates that the ventilation system is
not as efficient in the organic stockroom as it is elsewhere. This is a concern, especially considering that many hazardous chemicals are stored in this room.

The peak time and end time seen in Room 2430 were also larger than those seen in other rooms. However, these values are not a concern for two reasons. First, this room is a large lecture room and the ventilation system is expected to be less efficient compared to what is seen in laboratories. Secondly, chemicals are not often used in this room, and when they are it is for demonstration purposes only, meaning that the chance of a spill is greatly reduced.

The values of the b and c coefficients and their quotients are similar for most condition sets. These values are extremely large for the general chemistry laboratory with the ventilation system off. This is due to a poor trial that was taken to be an exception the values were not considered a concern. The values for the organic laboratory, with the ventilation system both on and off, were significantly larger than the rest of the rooms, yet still reasonable. This means the acetone was removed more quickly, indicating an increased efficiency as compared to the other rooms studied. This is encouraging because of the numerous toxic chemicals that are frequently used in this room. This is also helpful if improvements are made in other areas of McCollum Science Hall, because the mechanics of the ventilation system in this room can be examined and copied.

The data collected and analyzed used acetone only. The results, however, can be used to make accurate estimates of what would occur if other chemicals were spilled. The volatility of a chemical determines how quickly it evaporates, and is indicated by the boiling point of the chemical. Benzene is a carcinogenic chemical with volatility similar to acetone, meaning that it is reasonable to assume benzene would behave similar to acetone when spilled. The highest concentration of acetone observed during research was 3.6ppm, so the highest concentration of benzene that would found in the air after a sizeable spill would also be approximately 3.6ppm.

To determine what concentration of a chemical is hazardous to a person's health, the Threshold Limit Value, or TLV, may be used. This is a standard set by the American Conference of Governmental Industrial Hygienists, and it represents the concentration of
a chemical that nearly all people can be exposed to daily without suffering any adverse health effects. For benzene, the TLV is set at 25ppm. This means that the highest concentration of benzene reached after a spill (approximately 3.6ppm) is well below the TLV. In real-life situations, spills of exactly 300mL would not occur. However, most spills would be of a smaller volume, so that the concentrations reached would still be of low enough that the ventilation system could easily prevent any health risks.

Comparisons are possible between acetone and chemicals of different volatilities as well. For chemicals with a higher volatility, it is expected that the peak concentration would be higher than those reached using acetone. However, such a chemical would be removed from the room more quickly, since a higher volatility means the chemical would evaporate into the air more quickly. For chemicals with a lower volatility, it is expected that the peak concentration would be lower than those reached using acetone. Such a chemical would remain in the room for a longer period of time, though, because a lower volatility means that it would evaporate more slowly into the air. The ventilation system would provide protection from spills of both types of chemicals discussed above. This is because chemicals with a high volatility would be removed from the room too quickly to pose a significant health threat, while chemicals with a low volatility would evaporate too slowly to pose a significant health threat.

Overall, the research validated the use of the ventilation system of McCollum Science Hall whenever work is being done. It also suggested the possible need for improvement of the ventilation system of the organic stockroom, and the high efficiency of the organic laboratory’s ventilation system. The method developed to monitor indoor air pollution and the information gathered will be helpful in further studies of a similar nature.

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References:
Figure 1: Infrared source.
Figure 2: Telescope and interferometer.
Figure 3. Inside of interferometer.
Figure 4: Diagram of beamsplitter.
Absorbance / Wavenumber (cm⁻¹)

Figure 5: Acetone spectrum.

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Figure 6: Background spectrum.
Absorbance of acetone vs. time

Figure 7: Absorbance vs. time plot.
Beer's Law plot of acetone

Rank 13 Eqn 1 \( y = a + bx \)

\( r^2 = 0.992377737 \) DF Adj \( r^2 = 0.990199947 \) FitStdErr=0.00565063596 Fstat=1041.55704

\( a = 0.067438516 \)

\( b = 0.0084092645 \)

Figure 8: Acetone calibration curve.
Figure 9: Concentration vs. time plot.