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COMPARATIVE TESTING OF AN FTIR REMOTE OPTICAL SENSOR WITH AREA SAMPLERS IN A CONTROLLED VENTILATION CHAMBER*

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A portable Fourier transform infrared remote optical sensing spectrometer was deployed and tested in a constant ventilation test chamber by using a tracer gas source. Continuous beam path measurements were collected and compared to air samples obtained from a computer-controlled, multiple-point sampling array connected to a flame ionization detector. Measurements were gathered at two different room ventilation rates and at two different dispersion conditions. A homogeneous dispersion condition had a uniform tracer concentration over the beam path and an inhomogeneous dispersion condition had a nonuniform tracer concentration distribution over the length of the beam path. Overall, the beam measurements and the point sample readings showed good agreement regardless of the room ventilation rate. Comparative data obtained from the inhomogeneous dispersion conditions did have higher variability, probably as a result of the different spatial and temporal resolution of the two sampling techniques. The tests demonstrate that a remote sensing system can be applied to an indoor room scale setting, but the dispersion of contaminant in the beam path is an important factor to consider when interpreting the beam data.

A crucial step in the evolution of new technology for monitoring workplace contaminants is the transition from the laboratory to a field test situation. Often, this process involves testing in a chamber that provides some control over conditions yet allows for a full-scale simulation of field problems.

Fourier transform infrared–remote optical sensing of emissions (FTIR-ROSE) is a novel technology that has yet to find use in occupational settings.(1) This device combines an FTIR spectrometer with a remote detector and optics for aiming the IR beam over distances that may exceed 100 m.(2,3) In a conventional FTIR spectrometer, the IR beam is confined to a fixed path length inside a gas cell. An air sample is introduced into the gas cell and the sample spectrum is compared to a background spectrum obtained when the cell is purged with clean air. In the FTIR-ROSE configuration, the IR beam passes directly through the air in the workplace over a specified (perhaps variable) path. In a sense, the workplace replaces the gas cell in the ROSE configuration.

FTIR-ROSE technology has potentially unique advantages for monitoring workplaces. With this device, multiple components in a complex mixture of contaminants can be accurately identified and quantified with very low detection limits. Rapid sampling, typically a few seconds per spectral scan, provides the capability to detect transient emissions in real time. The unobtrusive IR beam can monitor many locations in the workplace without the need for pumps, sampling lines, and other devices that can interfere with workers. Eventually, it may become possible to automatically scan the workplace with an IR beam and use computer tomography(4) to map the concentration of several chemicals in real time and space while workers go about their normal activities.

Conversely, because FTIR-ROSE technology is in its infancy, its suitability for workplace situations has not been established. One difficulty is in collecting background readings, which provide a reference that compensates for instrument factors such as beam intensity, alignment, and instrument noise along with, to a certain extent, environmental factors such as carbon dioxide and water vapor levels. Often, obtaining a background spectrum from

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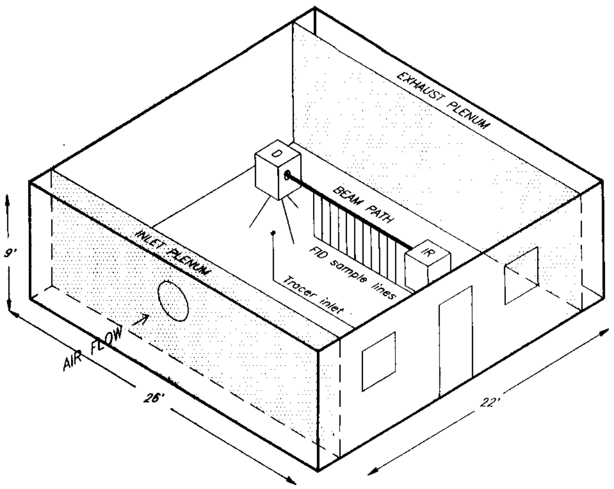

FIGURE 1. Large-scale ventilation test chamber for indoor tracer gas experiments. The diagram illustrates inhomogeneous concentration distribution conditions with the tracer source placed upstream of the FTIR-ROSE beam path and the FID multipoint sample array. The D refers to the infrared detector and the IR indicates the infrared source and interferometer unit.

uncontaminated air during a work shift is inconvenient or impossible. One approach to this problem is to collect and store a library of background readings covering a range of humidity and other environmental conditions. This technique has been used with some success in other FTIR-ROSE applications, such as monitoring chemical warfare agents on the battlefield.[5,6]

When applying the FTIR-ROSE monitor to the workplace, the effect of room factors such as ventilation and source location on the concentration reading must be considered. These room factors alter the dispersion of contaminant from the source into the beam path. Unlike most sensors, which indicate the air concentration at a single point, the FTIR-ROSE integrates the concentration over the entire path. This results in an output reading of ppm × length (ppm-m); dividing this reading by the path length gives the average concentration over the beam path. A consequence of averaging along the beam path is the interaction of contaminant dispersion with the concentration reading. A 10-m beam path would produce the same reading from 10 ppm uniformly dispersed over the path or from 100 ppm over 1 m and 0 ppm in the remaining 9 m.

Although the FTIR-ROSE system is different from the traditional point area monitors or personal monitors employed in industrial hygiene, the data produced by the ROSE system have common features with both samplers. Like many area monitors, the ROSE system can provide rapid real-time data on chemicals at specific locations; like personal monitors, the ROSE system can provide data that are averaged over time and space. Thus, the FTIR-ROSE monitor is a sampling device with characteristics somewhere between an area monitor and personal monitor that will likely require a different sampling strategy to use effectively in the workplace.

The purpose of this investigation was to compare the FTIR-ROSE configuration to a conventional point sampling array in a controlled setting. In addition to verifying the application of beam path sampling, the ability to control the source location and ventilation conditions should provide insight into the sampling issues that are likely to confront future studies.

EXPERIMENTAL MATERIALS AND METHODS

The FTIR-ROSE system was deployed and tested in a large indoor chamber. A series of experiments was conducted to compare beam-averaged measurements to readings gathered by multiple-point sampling. A passive tracer was released from a point source into the room under constant ventilation conditions. Air concentrations were determined at a series of points along a horizontal beam path downstream of the source under different dispersion conditions.

Chamber Description

The test chamber was designed to provide uniform dilution ventilation in a horizontal plug-flow arrangement. Figure 1 illustrates the test chamber. The working section is 7.9 × 6.7 × 2.7 m or 143 m^3 (26 × 22 × 8.85 ft or 5050 ft^3). Two 0.6-m (2-ft) deep plenums completely cover the opposing side walls to distribute the supply and exhaust air. The inside walls of the plenums facing the room were covered with masonite panels that had 0.3-cm holes on 2.5-cm centers. The inlet plenum had an additional interior baffle made of 1-cm plywood with 2.5-cm holes on 10-cm centers. The room walls were of common wallboard construction and were extensively sealed to reduce leakage.

Mechanical ventilation was provided by two 64-cm (25-in.) industrial blowers in a push-pull configuration. With this arrangement, the static pressure between the inside and outside of the chamber was adjusted to <0.5 cm of water. The low-pressure drop across the walls combined with the sealing helped minimize uncontrolled infiltration. The fans were driven by dc motors fitted with tachometer feedback speed controllers that provided 1% speed regulation; the controllers allowed adjustment of the volume flow rate through the chamber from about 5.7 to 142 m^3/min (200 to 5000 ft^3/min). These ventilation rates gave room time constants of about 25 min to 1 min, corresponding to air exchange rates of 2.4 to 60 air changes per hour.

The tracer gas used in this study was acetone vapor. The vapor was generated by passing 1 L/min dry nitrogen through liquid acetone in a 250-mL glass bubbler kept in a water bath at 25 to 40°C ± 1°C. The bubbler output connected to a 4-L glass flask where the vapor equilibrated and then passed through polyethylene tubing to a 2-cm diameter porous ceramic spherical diffuser, creating a point source. The release of the nitrogen gas was controlled by a needle valve and measured by a rotameter calibrated with nitrogen. The source generation rate was varied by changing the bubbler temperature and nitrogen flow rate.

The chamber was equipped with a computer-controlled sampling system designed by the authors that sequentially monitored air concentrations at up to 16 points. Each sample point was connected by polyethylene tubing to a three-way solenoid valve and exhaust manifold. Air samples were drawn continuously from the sample lines into the exhaust manifold to minimize the response time delay caused by the dead volume in the tubing. Operating a valve diverted the sample flow from the exhaust manifold into a flame ionization detector (FID) from Century Instruments (Arkansas City, Kans.), which typically provided a 1.0-mV/ppm analog output signal. This output signal was amplified, passed to a 12-bit analog-to-digital (A/D) converter, and stored in the computer. The FID detector has a range of 0.2 to 2000 ppm with a response time (0% to 90% of full scale) of 1 sec. About 5 sec was required to obtain a stable reading from the room because of valve switching transients and the overall response time of the sampling network.

The experiments were controlled and the data collected by a laboratory computer with a clock, A/D converter, and a 32-bit input/output (I/O) module. Sampling valve selection was directed by computer software through a digital output module and relay interface connected to the valves. Control software was written in compiled BASIC with machine language I/O subroutines.

Twelve sample probes were arranged 0.3 m (1 ft) apart in a horizontal line array and were scanned continuously by the computer sampling network. Each port was sampled for 10 sec, followed by a 10-sec delay between samples to preclude any switching artifacts. At this sample rate, the entire array was scanned every 5 min, and typically 30 to 90 min of total time was accumulated in an experimental trial. An overhead, remote-controlled traversing system powered by dc motors positioned the sample array without requiring people to enter the room. In addition to monitoring the sampling ports in the chamber, the computer network also scanned three other points. These ports corresponded to the air inlet, air outlet, and calibration gas. These additional samples furnished a check on the room background, source emission rate, and the detector calibration.

The chamber was equipped with a three-component sonic anemometer to provide measurements of air velocity.(7) This was a prototype instrument constructed by the Commonwealth Scientific and Industrial Research Organization (CSIRO) of the Australian government. The instrument uses ultrasonic sound pulses to detect the air velocity, and it can unambiguously resolve the air velocity vector components; it has a linear response, an absolute calibration that depends only on sensor spacing, and a wide dynamic range (0.5–2000 cm/sec). The frequency response was limited to 20 Hz for this unit, although it is capable of higher data rates. Signal processing modules provide digital outputs and analog voltages proportional to the velocity components along each axis. The digital signals are real-time readings of three velocity components and temperature. These data were linked via an RS-232 serial port into the laboratory computer and used to characterize the mean velocity and turbulence intensity.

FTIR-ROSE Description

The data were collected by using a Nicolet ST-1 FTIR spectrometer equipped with a liquid nitrogen–cooled mercury cadmium telluride (MCT) detector (Nicolet Instrument Corp., Madison, Wis.). The optical bench was modified by Nicolet for remote optical sensing. The essential changes consisted of (1) splitting the He-Ne laser beam and positioning it to provide a coaxial alignment beam, (2) removing the closed gas cell and replacing it with elliptical mirrors that directed the modulated IR beam outside the cabinet, and (3) mounting the MCT detector to a telescope. The IR source (1300 K filament) and swinging mirror interferometer (2-cm^{-1} resolution) were mounted together in a cabinet on a tripod, from which emerged a partially collimated 8-cm^2 IR beam with a coaxial 1.5-mW laser beam to assist with aiming. The separate tripod-mounted detector was fitted to a 20-cm Cassagrain telescope, which collected the IR probe beam. Several 40-cm square, flat-front surfaced mirrors mounted on tripods were used to direct the IR beam around the room. Sample scanning, data collection, and spectral analysis were performed by using an IBM-compatible 386 personal computer.

The FTIR-ROSE can be configured with the mirrors to create a variety of beam geometries. The simplest case is a straight line (I geometry) with the interferometer and detector modules placed at opposite ends of the beam path. With a single mirror, a V geometry can be created by aiming the beam at a mirror at the point and reflecting it back to the nearby detector module. Similarly, other geometries can be created by arranging mirror segments to create beam paths shaped like an L, C, S, U, Z, or another letter.

System Calibration

The flame ionization detector was calibrated over a range of 1 to 1500 ppm by using commercially available gas cylinders (Matheson Gas Products, Secaucus, N.J.). Two factory-analyzed primary gas standards, 100 ppm and 1500 ppm (both ±1%) acetone in air, were diluted immediately prior to calibration in hydrocarbon-free (HCF) air to create working standards. Dilutions were prepared by measuring a volume of HCF air into 20-L Tedlar® bags with a calibrated dry test meter and then injecting a volume of the primary gas into the bags with a gastight syringe. This yielded intermediate concentrations of 1, 5, 10, 50, 200, 500, and 1000 ppm. A 10-ppm standard was routinely used for on-line calibration checks during sampling runs.

Quantitative analysis was performed with a least squares fit (LSF) program and a spectral library covering the range of 5000 to 700 cm^{-1} at 2.0-cm^{-1} resolution. LSF quantization used the 53-ppm acetone reference spectra in two analysis windows at 2800–3200 cm^{-1} and 1150–1260 cm^{-1}. The spectral library was obtained from previous tests with a 10-m fixed path gas cell. Factors underlying the choice of this library and the least squares algorithm are described in detail elsewhere.(8)

The calibration of the FTIR-ROSE relied on the stored reference spectra obtained with the 10-m gas cell filled with 53 ppm acetone. All quantitative analysis used this library standard and assumed a linear response obeying Beer's law. However, the response for a known acetone concentration that used a 10-cm gas cell mounted in the beam path was checked. Because of the short path length of this gas cell, the FTIR calibration was directly verified only at the highest tracer concentrations (>1000 ppm). Quantitative analysis of spectra obtained from the 10-cm gas cell filled with a 1500-ppm standard showed agreement within 2%.

RESULTS

Two different source placements were evaluated under two air exchange rate conditions. Multiple experiments were conducted

TABLE I. Summary of Experimental Conditions

Test Condition	*Homogeneous High Flow*	*Homogeneous Low Flow*	*Inhomogeneous High Flow*	*Inhomogeneous Low Flow*
Time constant (min)	2	4	2	4
Air change rate (per hr)	30	15	30	15
Air velocity (cm/sec)	6	3	6	3
Source location	inlet	inlet	center	center

at each condition and at various source emission rates to give a range of contaminant concentrations in the room. The high airflow condition corresponded to a room time constant of 2 min (30 air changes per hour) with an average air velocity of 6 cm/sec. The low airflow condition corresponded to a time constant of 4 min (15 air changes per hour) and an average velocity of 3 cm/sec. The room time constant was measured by observing the decay in the outlet concentration of the tracer gas after suddenly switching off the source.

Table I summarizes the four experimental conditions. Each experiment was conducted over 30–90 min, spanning a minimum of 10 room time constants to ensure steady-state conditions. All experiments were replicated at least three times. FTIR beam measurements obtained at a rate of 2 scans/sec were co-averaged over 128 scans to produce interferograms that were stored for later analysis.

The homogeneous dispersion conditions were created by placing the source in the room air inlet, which generated a stable and uniform tracer concentration throughout the room. Figure 2 shows an example of the tracer concentration measured by the FID sample points spanning across the room center line under homogeneous, low flow conditions, along with the outlet (OUT) and inlet (IN) sample points. The plot shows the average and standard deviation of a series of 10-sec samples gathered at each point over a 75-min interval. Measurements obtained by moving the sample array throughout the room showed a similar uniform concentration distribution for both low-flow and high-flow conditions. Homogeneous conditions allowed for the comparison of the FID and FTIR monitors by using various path lengths and beam geometries (e.g., V-, C-, and M-shaped) for the FTIR-ROSE. These different paths were created by reflecting the beam around the room with mirrors as described previously.

Figure 3 shows a scatter plot comparing the tracer concentration simultaneously measured over 15-min time intervals by the FTIR-ROSE and the FID under homogeneous dispersion conditions. Each dot on the graph represents an average of all points in the FID line array compared to the average of the FTIR-ROSE over the same time period. No apparent systematic difference was observed for the various beam paths and geometries under these conditions. The data on the plot include high- and low-flow conditions and various beam path geometries. A regression line was fitted through the data by using a zero intercept, yielding a slope of 1.15 and correlation coefficient of $R^2 = 0.97$. The slope was found to be significantly greater than unity when examined with a t-test ($p < 0.01$).

Inhomogeneous dispersion conditions were created by placing a point source approximately at the geometric center of the room. This created a highly time-variable nonuniform distribution of tracer in the room with the long-term average spatial distribution approximating a classic, Gaussian-shaped plume spreading downstream from the source toward the outlet.[9] Figure 4 shows an example of the tracer concentration measured by a line array of FID sample points spanning the width of the plume under inhomogeneous conditions. This figure illustrates the crosswind concentration profile of the plume with the sample array positioned 2.4 m (8 ft) downstream and at the height of the source and oriented perpendicular to the direction of the airflow. The data show the expected Gaussian-shaped concentration profile along the crosswind direction.

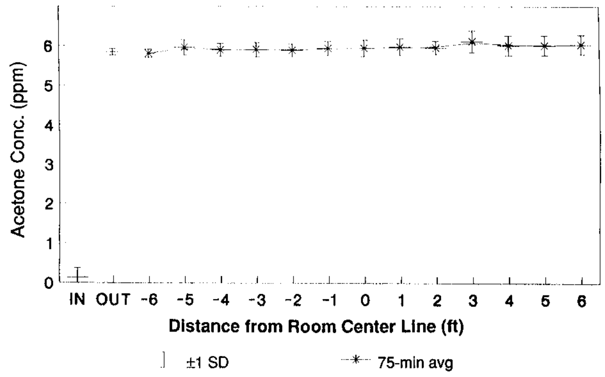

FIGURE 2. Tracer concentration measured horizontally across the room center line with the FID sample array under homogeneous, low-flow dispersion conditions. The plot shows the average and standard deviation of a series of 10-sec samples gathered at each point over a 75-min interval.

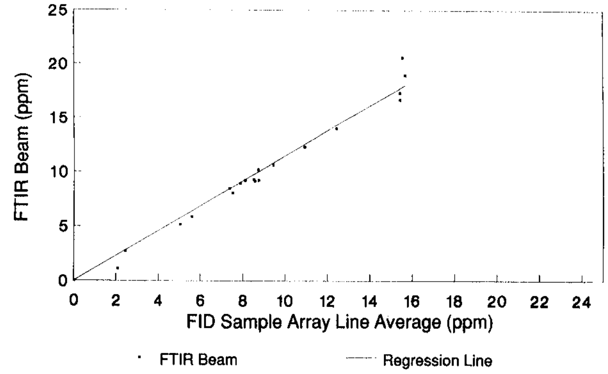

FIGURE 3. Comparison of FTIR-ROSE beam measurements and FID sample array measurements for high- and low-flow, homogeneous dispersion conditions

Figure 5 shows a scatter plot comparing the tracer concentration simultaneously measured over 15-min intervals by the FTIR and the FID under inhomogeneous dispersion conditions. This configuration compares concentration readings from a straight-line array of FID sample points with a co-linear IR beam path that transected the plume at various distances downstream from the source. As before, each dot represents an average of the FID line array and FTIR-ROSE over the same time period. No apparent systematic difference between beam and FID readings was observed under these conditions for source-to-beam distances of 1.2 m (4 ft), 1.8 m (6 ft), and 2.4 m (8 ft), or between high-flow and low-flow conditions. The plot includes data points from both high-flow and low-flow experiments and for all three source-to-beam distances. A regression line fitted through the data points yielded a slope of 1.09 and a correlation coefficient of $R^2 = 0.85$, assuming a zero intercept. This slope was not statistically different from 1.

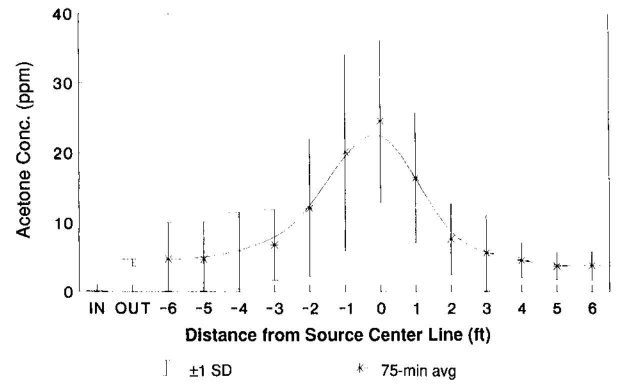

FIGURE 4. Crosswind tracer concentration measured horizontally across the room center line with the FID sample array under inhomogeneous dispersion conditions. The plot shows the average and standard deviation of a series of 10-sec samples gathered at each point over a 75-min interval. The sample array was positioned 2.2 m (7 ft) downstream from the point source.

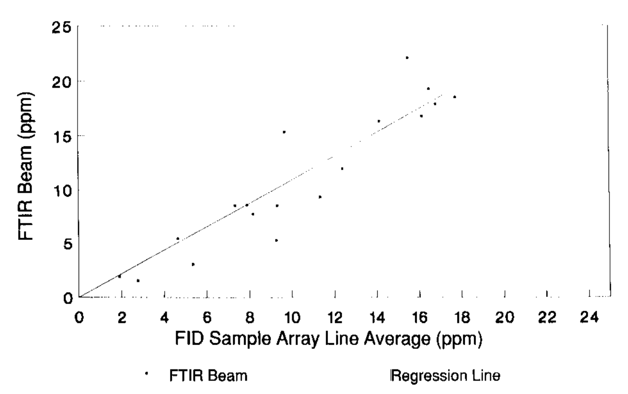

FIGURE 5. Comparison of FTIR-ROSE beam measurements and FID sample array measurements for high- and low-flow, inhomogeneous dispersion conditions

DISCUSSION

The chamber data indicate that the FTIR-ROSE monitor can be a viable method for indoor area monitoring, particularly under conditions that provide a high degree of air mixing and constant airflows. The degree of homogeneity (i.e., room mixing) had some influence on the results, but under both dispersion conditions the beam data and FID sample array showed good agreement. The FID and beam data gathered under homogeneous conditions were very highly correlated, indicating that sampling in a room with well-mixed conditions will present few difficulties compared to conventional point sampling. Under these conditions, obtaining an average of several point samples to estimate the room air concentration is appropriate. The FTIR-ROSE could acquire within minutes the room concentration data for multiple compounds without the need for sample lines, pumps, sampling tubes, or costly laboratory analysis. As a practical note, well-mixed conditions can be readily achieved in many rooms through the use of inexpensive axial fans.

The data comparing point samples to beam samples under inhomogeneous conditions showed considerably higher variability compared to the data from homogeneous conditions. This outcome may stem from several factors, but the primary cause may be the limited spatial and temporal resolution of the FID array compared to the beam. The FID array sampled individual points along the beam path at 0.3-m (1-ft) intervals and for about 3% of the total scan time. In comparison, the FTIR measures the whole path virtually continuously in time over the sampling period. The FID array could, quite possibly, miss high concentration regions as the meandering tracer plume drifted in and out of the beam path. Figures 2 and 4 illustrate the difference in the long-term average concentration distribution caused by the two dispersion conditions; the scatter plots display differences in the short-term spatial distribution of contaminant observed by the two different sampling methods. Because the structure of the plume evolves on a time-averaged basis, comparisons of the FTIR and FID samplers should focus on ensemble average measures like those provided by the regression line statistics.

In all cases, the FTIR readings agreed within 50% of the FID array, and most of the readings fall within about 15%. The slope of the regression line for both dispersion conditions was greater than 1, suggesting a possible systematic difference between the FID and FTIR calibration of around 10%. Although the inhomogeneous data were not significantly different from an ideal line, this may only reflect the higher variability in this comparison.

Considerable effort was made to carefully calibrate the FID against known standards over the working range, but it was not possible to perform a similar direct calibration on the FTIR-ROSE. The FTIR quantitative analysis assumed adherence to Beer's law, although previous work with a 10-m gas cell has shown that acetone at very low concentrations (<1 ppm) may exhibit deviations from this assumption.(10) The test performed with a 10-cm gas cell at 1500 ppm showed excellent agreement, but the authors would have preferred to test several more points over a lower concentration range. More testing in a controlled room situation with other analytes and with careful calibration will be needed to resolve this issue.

Background spectra used for the analysis were collected before and after each experimental session in the chamber. This represents the preferred situation where one can directly obtain a background reading in situ. Choosing less direct background spectra may change the quantitative results, depending on the circumstances. As a preliminary test, surrogate background spectra gathered in a different environmental chamber were substituted in the analysis for acetone and compared to the results obtained by using the direct background from the experiments. The surrogate backgrounds spanned a well-controlled range of temperature and humidity conditions from 13 to 33 °C and 20% to 80% relative humidity. The analysis with the direct background gave an average value of 11 ppm; the worst case value found with a surrogate background was 13 ppm, which amounts to a change of around 20%. This suggests that the quantitative results may suffer modest degradation for a known analyte when using a surrogate background. However, more dramatic changes did appear in the residual spectrum, suggesting a more severe impact on the ability to identify unknown analytes.

Many issues remain to study with regard to specific applications of optical beam monitoring systems. For example, one application for a FTIR-ROSE system might be as a kilometers-long path "fence line" or perimeter monitor to scan for fugitive emissions of an acutely toxic compound. The detection limit and sensitivity of such a monitor would depend in part upon how rapidly and completely the contaminant was dispersed into the beam path. In addition, other concerns arise for outdoor measurements such as possible interference or background shifts when operating in rain, snow, or fog.

These experiments purposely focused on testing indoor conditions over a time scale of many room time constants where the average structure of the dispersion pattern became important. Typical rooms have time scales of 10 to 60 min (i.e., 1–6 air changes per hour) so appropriate averaging times could be several hours. Monitoring contaminants on such long time scales is appropriate for chronic toxins but inappropriate for acutely toxic agents.

The data presented in this paper may be regarded as an initial step toward deploying FTIR-ROSE technology in a workplace. Only a limited range of conditions have been examined, but the data show that the dispersion process can have a strong influence on the results produced by a particular monitoring device. The spatial and temporal distribution of the contaminant interact with the characteristics of the sampling device, whether it is a point, beam, or personal sampler. Deploying an FTIR-ROSE system in the workplace to monitor exposures will require a sampling strategy that exploits the unique characteristics of the beam measurements.

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