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EVALUATION OF AN INFRARED OPEN-PATH SPECTROMETER USING AN EXPOSURE CHAMBER AND A CALIBRATION CELL

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This study evaluated the use of a controlled exposure chamber and calibration cell to investigate the performance of open-path remote sensing instruments for workplace monitoring of pollutants. An open-path infrared spectrometer was deployed in a chamber, and a range of homogeneous air concentrations were generated using the tracer gas sulfur hexafluoride (SF₆). Open-path measurements were compared with point-sample measurements taken along the beam and analyzed using an infrared (IR) and electron capture detector (ECD). A five-compartment calibration cell placed in the optical path was used to simulate heterogeneous concentrations of pollutants. Fifteen concentration patterns were created by injecting different volumes of SF₆ into each compartment. Open-path spectrometer measurements were compared with compartment concentrations measured using an ECD. Results indicated that stable homogeneous and heterogeneous concentrations of SF₆ were generated in the controlled exposure chamber and five-compartment calibration cell, respectively. Overall, individual open-path measurements were within 20% of point samples measured with the IR and ECDs. The open-path spectrometer measurements had a consistent positive bias of about 12%. Exposure chamber experiments uncovered remote sensing instrument design aspects that could adversely impact its use for indoor monitoring. The weight and size of the instrument make it useable only in a fixed position, thus limiting spatial coverage of the beam in a room. The instrument components were not airtight, resulting in overestimation of some open-path concentrations. A controlled test chamber and a long calibration cell placed in the optical path are useful tools for evaluating a variety of open-path spectrometer instrument performance issues relevant to industrial hygiene monitoring.

Infrared open-path spectrometers currently are being evaluated for the measurement of air pollutants outdoors.^(1,2) These systems typically use a remotely placed optical source that transmits an infrared (IR) beam across

distances that can exceed 100 m, and a receiver (spectrometer) that detects and measures pollutants present in the optical path. Concentration is measured and integrated over the entire length of the optical path, and is reported as the product of concentration and path length (ppm-meters). The open-path remote sensing technique is noninvasive, operates in real-time, and gives spatial average concentrations over distances that normally would require the use of large numbers of point-sampling devices. The use of an open-path Fourier transform infrared spectrometer (FTIR), in particular, will enable the measurement of multiple pollutants in air simultaneously; the pollutants potentially can be measured at very low limits of detection.⁽¹⁾

Most of the development and validation of open-path technology has focused on outdoor applications; the use of it for indoor industrial hygiene monitoring has received only limited attention.^(3,4) In a workplace, optical beams can be placed strategically to detect leaks of toxic chemicals and then can be used to monitor the area remotely, without the need for direct human intervention, to determine when it is safe for reentry. If the spectrometer is attached to a stepper motor, a large area can be monitored by projecting crisscrossing beams over the entire workplace, thus creating a network of measurements. These multiple measurements can be processed, using computed tomographic techniques, to produce chemical concentration maps that provide good spatial and temporal resolution.^(5,6) It may be possible to use these concentration maps to visualize the flow of contaminants in room air and to evaluate worker exposure to chemicals.

Before open-path remote sensing technology can supplement more conventional monitoring methods, it must be validated thoroughly for the industrial hygiene application. One difficulty with validating open-path spectrometers is the fact that the IR beam is open to the atmosphere. In a conventional infrared spectrometer the beam is enclosed in a gas cell; the cell can be purged with clean air to obtain contaminant-free background readings and can then be filled with known concentrations of pollutants to determine measurement precision and accuracy. In contrast, the open path cannot be evacuated to obtain background readings, nor can it be filled with known stable concentrations of chemicals.

Investigators have attempted with varying success to validate open-path spectrometer measurements outdoors by releasing gas into the air and comparing open-path measurements with point-sample measurements taken along the beam.^(7,8) However,

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there are inherent limitations to performing these validation experiments outdoors, because meteorological conditions are unstable and uncontrollable. When a gas is released into the air, concentrations will fluctuate along the beam, and the gas may intersect portions of the beam where there are no point samplers. It is difficult to determine whether discrepancies between path-averaged and point-sample concentrations are due to the poor spatial resolution afforded by a limited number of point samplers along the beam or poor instrument accuracy.

The purpose of this study was to evaluate the use of a controlled exposure chamber and a calibration cell for investigating open-path spectrometer measurement accuracy for the industrial hygiene application. Stable homogeneous and heterogeneous pollutant concentrations were generated in these controlled environments, and open-path measurements were compared with point samples taken along the beam.

MATERIALS AND METHODS

A single-gas open path infrared spectrometer was deployed and tested in an exposure chamber and tested with a calibration cell placed directly in the beam path. Experiments were performed using homogenous and heterogeneous concentrations of the tracer gas sulfur hexafluoride (SF_6). All experiments were repeated at least three times. SF_6 was chosen because it has good absorption characteristics in the infrared region, has low human toxicity, and is not usually present in ambient indoor air. The path-averaged concentrations measured with the open-path spectrometer were compared with path-averaged concentrations calculated from point samples taken along the beam.

Open-Path Spectrometer

Data were collected using INFRASAFE, a single-gas open path infrared spectrometer (MDA Scientific, Inc., Lincolnshire, Ill.). INFRASAFE was not designed for indoor applications; it was developed primarily for outdoor fenceline and emission monitoring for path lengths up to 300 m. Therefore, to facilitate stability when used outdoors, the instrument is heavy and large. Although this construction is not ideal for the indoor application, the ability of the instrument to measure ppm concentrations of SF_6 over short path lengths relevant to indoor use enabled INFRASAFE to be used for studying open-path validation methods. The INFRASAFE device used in this study was equipped with narrow band pass filters designed to detect SF_6 .

This open-path spectrometer consisted of a separate transmitter and receiver. The transmitter housed the IR source (1300°K filament) and emitted broad-band radiation with a six-inch diameter beam. The receiver contained the narrow band pass filters that allowed energy to pass at wavelengths of 10.6 μm and 10.17 μm , to the sample and reference detectors, respectively. The spectrometer was first aligned in contaminant-free air, and baseline signal levels (millivolts) and path length (meters) were entered into the control system. When SF_6 was introduced into the path of the beam, the manufacturer's software calculated path-integrated gas concentrations (ppm-meters)

based on differential absorption at the two wavelengths. Path-integrated concentrations were then divided by the path length to convert to path-averaged concentrations (ppm). The source code for the calculation software was not provided by the manufacturer; therefore, the algorithm used for calculating concentrations could not be determined.

The spectrometer was tested in single and reflected-beam configurations. To create a single-beam, the transmitter and receiver were placed in a straight line at opposite ends of the beam path. To create a reflected-beam, the transmitter and receiver were stacked in a rack; the transmitted beam was aligned with two first surface mirrors across the room that reflected the beam 180° back to the receiver.

Chamber and Calibration Cell Design

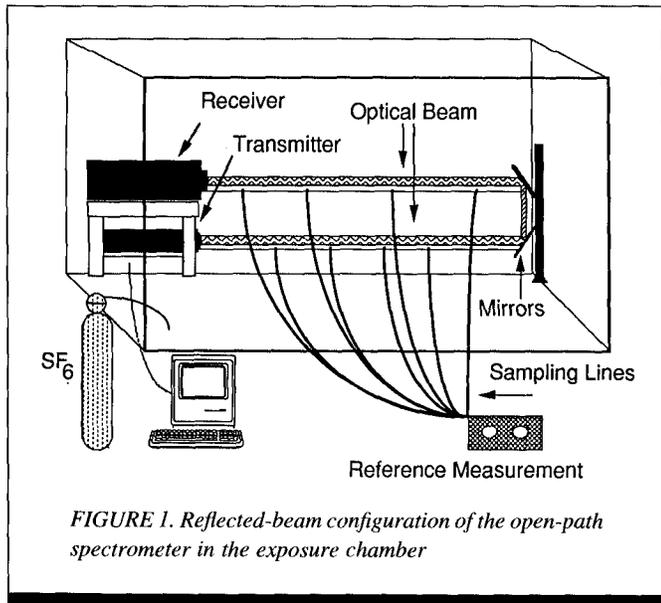
An exposure chamber and five-compartment calibration cell were used to validate measurements obtained with the open-path spectrometer. Both the chamber and calibration cell were sealed to minimize leakage of tracer gas and infiltration of room air. The sealed exposure chamber (10 ft \times 10 ft \times 8 ft) was designed to provide a controlled homogeneous concentration of a gas along the entire length of the optical beam. The chamber was equipped with a fan to ensure adequate mixing of test atmospheres, sample ports to inject tracer gas, and tygon sampling lines to retrieve samples. When the spectrometer was placed in the single-beam configuration, the sample lines were suspended at equally spaced increments along the bottom edge of the optical path. In the reflected-beam configuration, the sample lines were placed along both legs of the path (see Figure 1).

A calibration cell with an overall path length of 70 cm was designed to provide up to five different controlled homogeneous concentrations along a section of the optical beam. This series of concentrations was used to simulate a beam traversing heterogeneous concentrations of a contaminant in air (e.g., a plume). The calibration cell was constructed of wood and had five individual compartments that were separated by polyethylene film; each compartment was equipped with a mixing fan, an injection and sampling septum, and a purging port. Experiments determined that SF_6 gas concentrations were stable in each of the compartments for over 30 minutes.

For the calibration cell experiments, the spectrometer was deployed in a large laboratory, and both single and reflected-beam configurations were used. When the calibration cell was placed in the optical path, the beam traversed both the air in the room and the air in the cell (see Figure 2).

Validation Equipment and Calibration

Concentrations measured by the open-path spectrometer were compared with point-sample concentrations measured using a MIRAN IA infrared gas analyzer (Foxboro, Inc. E. Bridgewater, Mass.) and an ITI Gas Chromatograph (Burlington, Mass.) with Valco Electron Capture Detector (Houston, Tex.) (GC-ECD). In terms of sensitivity to SF_6 , concentrations were quantifiable in the low ppm range using both the GC-ECD and the MIRAN. In terms of required sample volume for analysis, the GC-ECD had a small sample cell volume of 500



μL , and the MIRAN had a larger sample cell volume of 5.6 L. At a given concentration the GC-ECD required for analysis a smaller volume of air adjacent to the optical path than the MIRAN. Therefore, the MIRAN was used only for the single-beam chamber experiments; the GC-ECD was used for single and reflected-beam experiments in the chamber and experiments using the calibration cell.

The MIRAN was calibrated over a range of 5 to 70 ppm, and the GC-ECD was calibrated over a range of 5 to 250 ppm, by diluting commercially available (National Safety Gas, Inc.), factory-analyzed gas cylinders of 100 ppm SF₆ and 30% ($\pm 1\%$) SF₆ in air. Both instruments were calibrated using closed-loop fixed volume chambers; dilutions were prepared by injecting known volumes of SF₆ through a septum into the loop using a gastight syringe. Depending on the working range for the experiments, 5 to 15 concentrations were used to create the calibration curves. The working concentration ranges for SF₆ were limited by the maximum detectable level of 100 ppm-meters, reported by MDA Scientific, Inc., for the open-path spectrometer. Beyond this level excess absorption by the reference detector results in decreased measurement accuracy. Complete calibrations were performed before and after each experiment. Three measurements were averaged for each point on the calibration curve. During the course of the experiments the calibration was checked using three concentration points on the curves.

Chamber Experiments

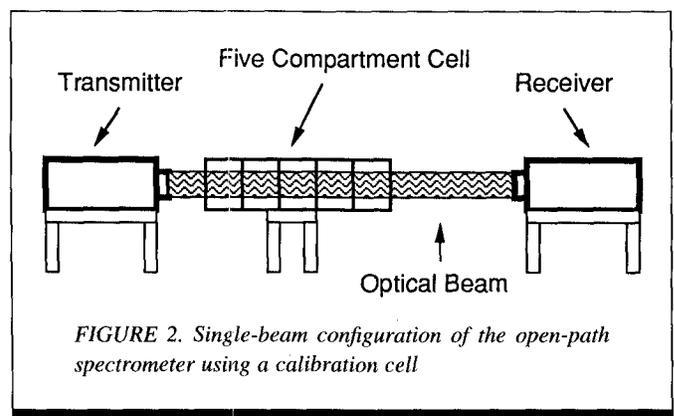
Homogeneous gas concentration experiments were performed in the exposure chamber using the open-path spectrophotometer aligned in both the single and reflected-beam configurations, resulting in path lengths of 1.9 m and 5.35 m, respectively. For the homogeneous experiments, to achieve incremental increases of 5 ppm in the chamber, SF₆ was released into the chamber from a compressed gas cylinder using a two-stage high-purity gas regulator and a rotameter. The flow was

stopped at every incremental increase in concentration, and the air in the chamber was mixed for several minutes before sampling. Point samples were then quickly obtained through tygon sampling lines using 30 mL polypropylene syringes; four and eight evenly spaced samples were obtained along the single and reflected-beams, respectively. At a given concentration, three samples were taken through each sample line. Before samples were withdrawn from the chamber, the syringes were conditioned to reduce absorption of SF₆ on the walls by fully filling and emptying the syringes three times.⁽⁹⁾

The concentration displayed by the open-path spectrometer was logged concurrently with point sample collection. This step-wise addition of SF₆ and measurement by open-path and point-sample methods was repeated until maximum working concentrations of 55 ppm and 20 ppm were reached for the single and reflected-beam configurations, respectively. Both the MIRAN and the GC-ECD were used for the single-beam experiments; only the GC-ECD was used for the reflected-beam experiments. At each concentration the path-averaged concentration measured with the open-path spectrometer was compared with the path-averaged concentration calculated by averaging all the point-sample measurements obtained along the beam.

Calibration Cell Experiments

For the heterogeneous gas concentration experiments the open-path spectrometer was placed in a larger laboratory and was aligned in the single and reflected-beam configuration, using path lengths of 7.28 m and 15.25 m, respectively. To obtain baseline signal levels, the five-compartment calibration cell was placed in the optical path before addition of SF₆. Heterogeneous air concentrations were then created by sequentially injecting known volumes of SF₆ into the compartments of the calibration cell. Each injection increased the concentration in a cell compartment by 6.5 ppm-meters. A total of 15 injections were used to create 15 different path-averaged concentrations (see Figure 3). After each injection, two minutes were allowed for mixing, and air samples were drawn from the compartments and analyzed by the GC-ECD. The concentrations displayed by the open-path spectrometer were logged during the entire experiment and were compared with path-averaged concentrations calculated from measurements obtained using the GC-ECD. The



GC-ECD path-averaged concentrations were calculated by summing the individual path-integrated calculations for each compartment (measured ppm using the GC-ECD \times compartment path length) and dividing by the overall spectrometer path length.

When using this open-path spectrometer, only a path-averaged concentration is displayed; the end-user cannot access the algorithm used to calculate SF₆ concentrations from the reference and absorbed signal levels. The manufacturer developed the algorithm by generating concentrations of SF₆ in a gas cell, measuring them with the open-path spectrometer, and using least squares to generate a line of best fit for the data. Using this calibration line, an algorithm was developed to convert the ratio of absorbed to reference signals and path length to ppm-meters. During operation the ratio of the input baseline signal levels establishes the zero concentration point on the calibration line. Because the algorithm is empirically based and proprietary, and the equipment is not designed to be calibrated, it is important to verify that the algorithm correctly determines concentrations for target chemicals for a range of path lengths and concentrations.

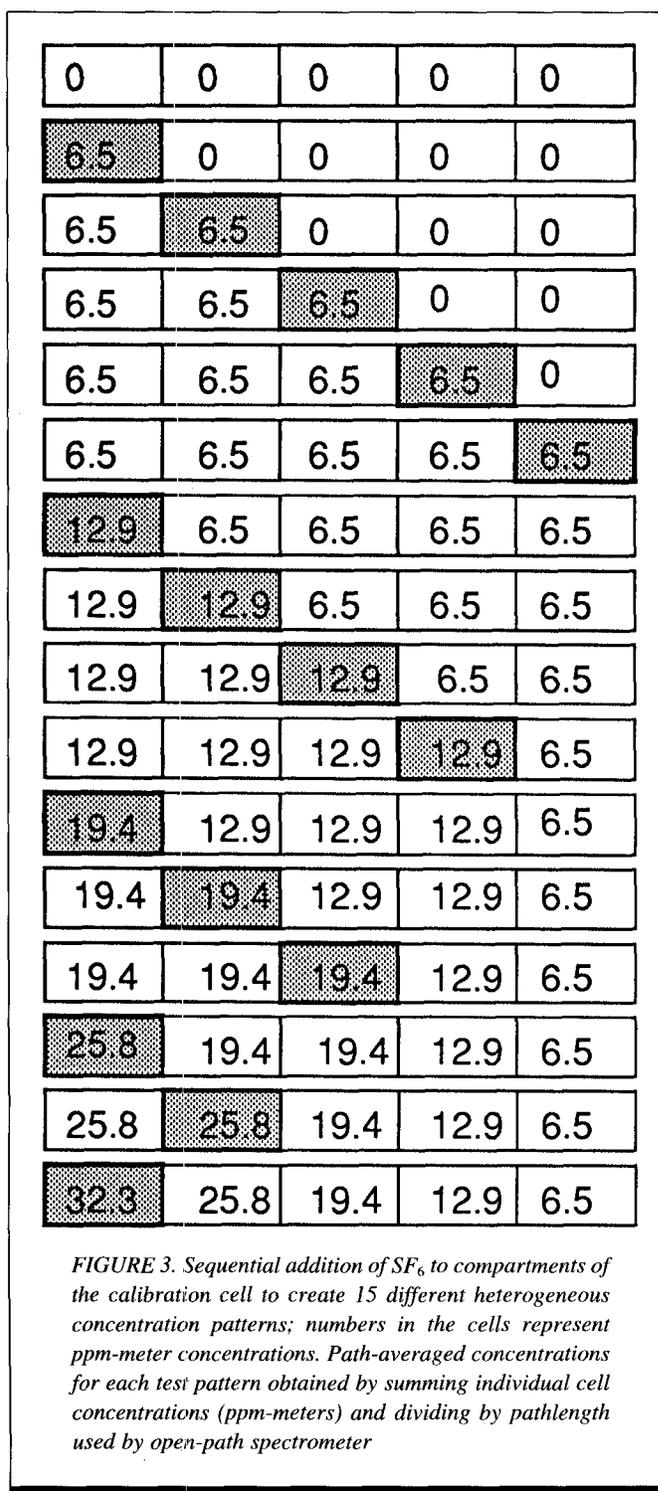
To test this aspect of the algorithm, the windows separating the five compartments in the calibration cell were removed, creating a single compartment, and homogeneous concentrations of SF₆ were generated in the long cell. Measurements were taken using a limited number of representative indoor path lengths. Calibrations were performed by adding known volumes of SF₆ to the cell, mixing the air using all five fans, and obtaining open-path spectrometer and GC-ECD measurements as before. The spectrometer was aligned in a single-beam configuration using path lengths of 1.5, 3.0, 4.5, and 6 m, and in the reflected-beam configuration using path lengths of 11.9, 12.9, and 16.9 m. The open-path concentrations were compared with path-averaged concentrations calculated from measurements obtained in the calibration cell using the GC-ECD.

RESULTS

Chamber Experiments

For the homogeneous gas concentration experiments, good correlation ($R^2 = 0.99$) was found between the path-averaged concentrations calculated using the MIRAN or the GC-ECD point samples, and the path-averaged concentrations measured with the open-path spectrometer. A regression line was fitted through the combined MIRAN and GC-ECD data using a zero intercept, and yielded a slope of 1.11 (see Figure 4). The slope of the regression line was found to be significantly greater than unity when examined with a t-test ($p < 0.01$). Using the statistical package SYSTAT, an analysis of variance found no significant difference between the results obtained using the MIRAN or the GC-ECD, or results obtained using a single or reflected-beam configuration.

To accurately compare point with open-path measurements in homogeneous concentration experiments, it is assumed that the chamber concentrations are indeed homogeneous. To check this assumption, for each of the experiments conducted using the MIRAN and GC-ECD, the standard deviation and coefficient of variation was calculated for the mean concentration of all point samples taken along the beam at each concentration level. As



shown in Table I, homogeneous concentrations were achieved in the chamber for the single-beam experiments that were performed using the GC-ECD; similar results were obtained for all of the homogeneous chamber experiments.

Calibration Cell Experiments

For the heterogeneous gas concentration experiments good correlation ($R^2 = 0.99$) was found between the path-averaged

TABLE I. Point Sample Measurements Taken Along the Optical Beam using Homogenous Concentrations in the Exposure Chamber

Mean Concentration ^A (ppm)	Standard Deviation	Coefficient of Variation
5.6	0.06	0.010
10.0	0.14	0.014
11.4	0.14	0.012
14.8	0.06	0.004
19.3	0.14	0.007
20.2	0.12	0.006
26.9	0.08	0.003
28.1	0.17	0.006
29.4	0.06	0.002
35.9	0.23	0.006
40.0	0.20	0.005
41.2	0.44	0.011
48.6	0.59	0.010
51.0	0.71	0.014

^A Mean concentration of four points taken along the beam, at each concentration level, measured using the GC-ECD

concentrations calculated from the individual compartment samples measured with the GC-ECD and the path-averaged concentrations measured with the open-path spectrometer. A regression line was fitted through the combined single and reflected-beam configuration data using a zero intercept and yielded a slope of 1.11 (see Figure 5). The slope of the regression line was significantly greater than unity when examined with a t-test ($p < 0.01$). An analysis of variance found no significant difference between the results obtained using the single or reflected-beam configuration.

For the experiments evaluating the algorithm, calibrations performed for path lengths ranging from 1.5 to 16.8 m using homogeneous concentrations in the cell resulted in good correlation ($R^2 = 0.99$) between the concentrations measured with the open-path spectrometer and the concentrations measured with the GC-ECD. Regression lines fitted through the data using a zero intercept yielded slopes of 1.11, 1.12, 1.13, and 1.12 for the single-beam configuration, for path lengths of 1.5, 3.0, 4.5, and 6 m, respectively. Regression lines for the reflected-beam configuration yielded slopes of 1.11, 1.10, and 1.12 for path lengths of 11.9, 12.9, and 16.8 m, respectively. There was no significant difference found among the slopes of the regression lines obtained at any of these path lengths for either configuration, and the slopes of the regression lines were all found to be significantly greater than unity ($p < 0.01$).

DISCUSSION

Data obtained using the exposure chamber and calibration cell indicate that these are useful methods for evaluating the strengths and limitations of using open-path spectrometers for indoor industrial hygiene air monitoring. Exposure chambers can be used to test instrument response in a simulated work room where the

entire optical beam can be immersed in a uniform concentration of a pollutant. This enables the open-path measurements to be compared with point-sample measurements in a stable and controllable environment. The chamber can be used to evaluate problems involved with the measurement of mixtures of chemicals of industrial hygiene concern and with background measurement collection when temperature, humidity, and CO₂ conditions vary.

Although there are definite advantages to using exposure chambers, there are practical limitations in the maximum path lengths they can accommodate. In contrast, a calibration cell placed in the optical path would enable the use of long path lengths; however, calibration cells can only cover a limited portion of the optical beam. Therefore, to obtain the same ppm-meter concentrations for a given optical path length, much higher concentrations must be generated in a cell than in a chamber. Because air pollutant concentrations can only be controlled within the cell, if the target pollutant is present in the ambient air conflicting results may be obtained if the cell is used for measurement validation.

No significant difference was found between the path-averaged concentrations measured with the open-path spectrometer and calculated from point samples taken with the MIRAN or GC-ECD for the homogeneous gas experiments in the exposure chamber or calibration cell, for all path lengths tested. The individual open-path spectrometer measurements agreed to within 20% of the point samples measured by the MIRAN or GC-ECD.

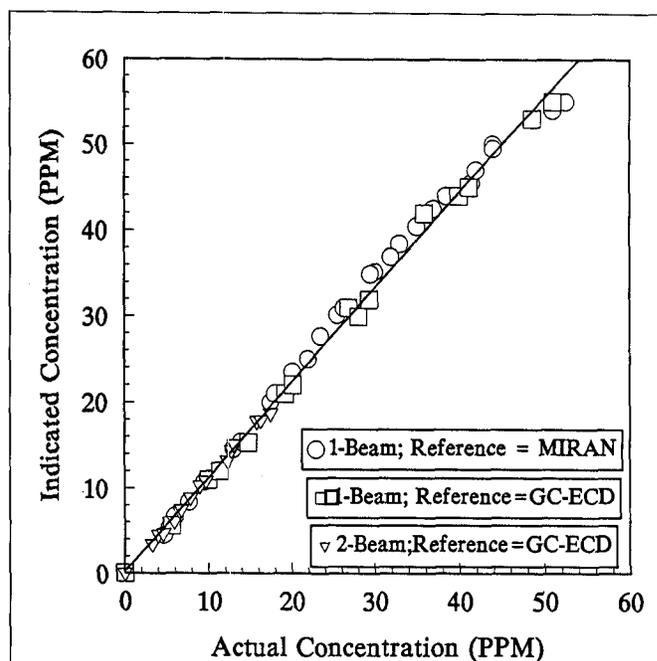


FIGURE 4. Actual versus indicated concentrations of SF₆ in the exposure chamber, using a single-beam and reflected-beam configuration in homogeneous air concentrations. Actual and indicated concentrations measured using either the MIRAN or GC-ECD and the open-path spectrometer, respectively.

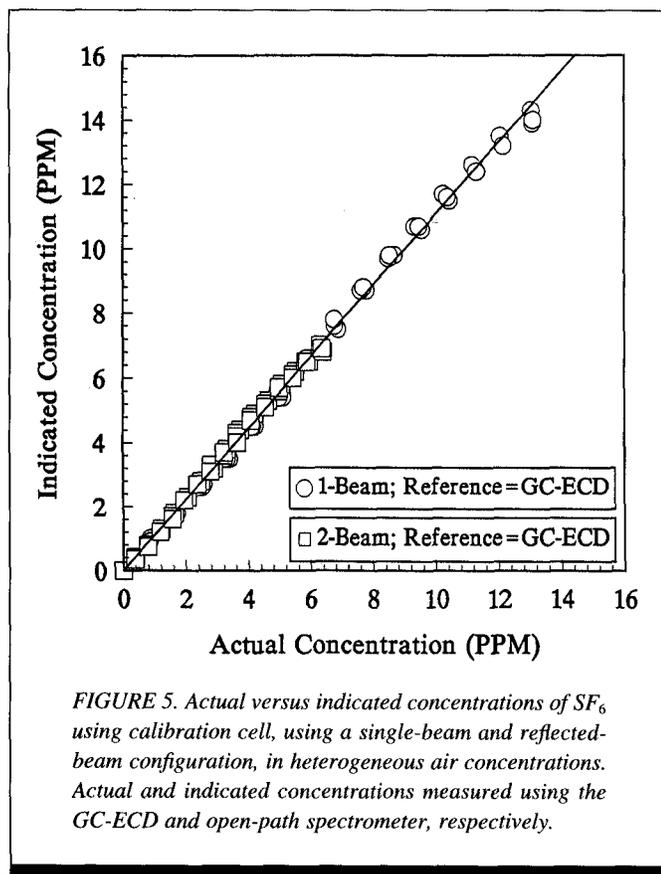


FIGURE 5. Actual versus indicated concentrations of SF₆ using calibration cell, using a single-beam and reflected-beam configuration, in heterogeneous air concentrations. Actual and indicated concentrations measured using the GC-ECD and open-path spectrometer, respectively.

In addition, there was no significant difference between the results obtained using the single or reflected-beam configurations.

From this study it appears that placing a calibration cell in the optical path might be a feasible method to perform routine instrument calibration checks or to compare open-path with other measurement methods. However, it must be more thoroughly validated that concentrations of pollutants measured inside a calibration cell are comparable to measurements obtained when the pollutant intersects all or a portion of the optical path. In addition, there may be limitations to using a calibration cell when high open-path concentrations are of concern. It may be necessary to use cell concentrations that are orders of magnitude higher than open-path concentrations due to the differences in cell and room pathlengths; this could result in nonlinearity problems. As with any method that uses a calibration cell, the materials used to construct the walls, optical windows, and sampling ports must be carefully selected to minimize pollutant adsorption, reactivity, and spectral interference. For this study the use of SF₆, which is relatively inert, allowed the construction of a fairly simple calibration cell; this would not be the case for most volatile organic compounds of concern in indoor industrial applications.

Most indoor environments are not well-mixed with homogeneous concentrations of pollutants in air. Therefore it is important to evaluate open-path instruments using heterogeneous air pollutant conditions. This study successfully used a five-compartment calibration cell to create 15 different stable concentration patterns. Results obtained using heterogeneous concentrations were not significantly different from results obtained using

homogeneous concentrations; the individual open-path spectrometer measurements usually agreed within 20% of the point sample measurements analyzed by GC-ECD. Similar results were obtained in a chamber study comparing open-path FTIR measurements with point measurements using acetone as the tracer gas.⁽⁴⁾

For this study the five compartments were adjacent to one another; an improvement in the experimental design would involve separating the individual compartments and scattering them along the beam. This was not performed because it would have required the use of additional optical windows, which would have absorbed even more incident IR energy, resulting in unacceptably low signal levels.

Although measurements obtained with the open-path spectrometer were in close agreement with path-averaged concentrations calculated from point samples measured with the GC-ECD and the MIRAN, they were consistently overestimated by 10–12%. This bias may be due to an error in the concentration calculation algorithm developed by the manufacturer or to a systematic difference in the instrument response from the instrument used to create the manufacturer's original calibration curves. Algorithm errors would most likely be traced back to the calibration curves and could be related to both the range and actual concentrations of gases that were measured.

Although the INFRASAFE open-path spectrometer was designed for long path lengths, it measured SF₆ with good accuracy at short path lengths that could be encountered indoors. However, indoor use may be limited by equipment weight and construction, the number of chemicals that can be measured simultaneously, and the limits of detection for the chemicals. The weight (>100 pounds) of the instrument makes it feasible to use only in a fixed position; to increase spatial resolution of concentrations, the size and weight would have to be reduced to facilitate rotation of the beam around the room. The spectrometer was constructed for use outdoors where the components could be located a distance from the potential plume site. Indoors, the instrument components would be located in the work area, and could be immersed in high air concentrations of pollutants. Therefore, for indoor use it is important that the equipment be airtight. In this study initial chamber experiments resulted in overestimation of some open-path concentrations by well over 200%. It was discovered that SF₆ had infiltrated the transmitter and receiver housings, resulting in excess gas absorption. The problem was solved by sealing the components and creating an internal positive pressure by installing a nitrogen purge at a flow-rate of 1.5 L/min.

The open-path spectrometer used in this study can only measure one chemical at a time; while this could be useful for very specific industrial hygiene applications, it would not be as versatile as an open-path FTIR spectrometer, which can simultaneously measure multiple chemicals and may provide lower detection limits than the filter-based infrared spectrometers.⁽¹⁰⁾

Studies need to be performed to thoroughly evaluate performance characteristics of open-path instruments for the measurement of chemicals of concern in industrial hygiene. These would include chemical selectivity, chemical sensitivity, measurement precision, measurement accuracy, limits of

detection, and problems associated with obtaining background measurements.

CONCLUSIONS

This study investigated the use of an exposure chamber and five-compartment calibration cell placed directly in the optical path for comparing INFRASAFE open-path spectrometer measurements with point-sample measurements. The open-path measurements were in close agreement, within 10–12%, with path-averaged concentrations calculated from point samples analyzed using a GC-ECD or a MIRAN. There was no significant difference found in measurement accuracy for a variety of path lengths among the experiments performed using the chamber or calibration cell, homogeneous or heterogeneous tracer gas concentrations, and single or reflected-beam configurations. The generation of stable and controlled concentrations in both the chamber and calibration cell resulted in low variability among the validation measurements. This ability to control pollutant concentrations along the beam allowed a more direct appraisal of measurement error than possible in outdoor experiments. The five-compartment calibration cell provided a successful method for simulating and measuring heterogeneous concentrations of SF₆ along a section of the beam.

This technology offers features that could complement traditional area and personal air sampling methods. However, validation of the method is still in its infancy. In addition to direct validation of the equipment, sampling strategies need to be developed to take advantage of the potential this technique provides for spatially and temporally measuring multiple chemicals in air. This work is underway.

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