Use of Long-path FTIR Spectrometry in Conjunction with Scintillometry to Measure Gas Fluxes.

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ABSTRACT

Fourier Transform Infrared (FTIR) spectrometry is rapidly becoming a technique of choice for analyzing volatile hazardous waste emissions (1). We have developed a field portable system that is capable of measuring gas concentrations at up to a 1 Km pathlength. The advantage of such a system is that it can analyze samples virtually in real time for myriad compounds simultaneously without introduction of any artifacts from sample collection. Because detection sensitivity increases with path length, analysis of compounds can often be made down to ppb levels. While we have concentrated on measuring gas emissions from biologic sources, we are also capable of monitoring hazardous gas emissions that have characteristic infrared absorbance peaks in regions of the IR spectrum that are not dominated by water or CO_2. Numerous volatile organic compounds such as chlorinated hydrocarbons, aromatic hydrocarbons, alcohols, ketones, esters, ethers and aldehydes fall into this category.

While gas concentrations are of interest, emission rates are needed to accurately evaluate waste sites. Obtaining such flux rates has become the focal point of our research. Recently, the Wave Propagation Lab (WPL) at NOAA (Boulder CO) has demonstrated that an optical-scintillation instrument can measure path-averaged momentum and heat fluxes. Development of scintillometers by WPL, which is currently in progress, will allow long-path measurement of water flux as well. Combining these long-path flux measurements with measurements of gradients of gas concentrations using the FTIR has the potential to provide an estimate of flux rates for numerous gases simultaneously. This technique will then have application in natural, agricultural and human impacted areas such as landfills and hazardous waste sites.

INTRODUCTION

Fourier Transform Infrared (FTIR) spectrometry is rapidly becoming a technique of choice for analyzing volatile hazardous waste emissions. A particular advantage of FTIR spectrometry is that it can be configured for long-path analysis. Large areas from which potential emissions are occurring can be analyzed at one time. We have developed a field portable system that is capable of measuring a number of infrared absorbing trace gases over long paths of up to a kilometer (Fig. 1). Many of these gases have either natural and/or anthropogenic sources and/or sinks. Increased path-length provides several advantages such as increased detection level (ppb levels) and long-path averaging of the high variability in gas emissions that may often be the case on a smaller scale (2).

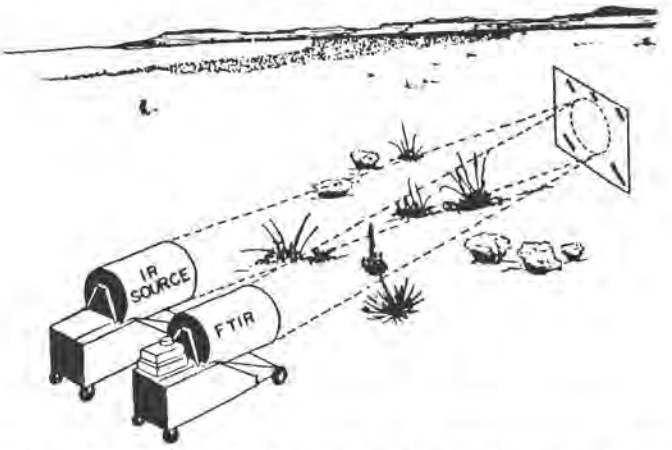


Figure 1. Schematic of an infrared radiation source and Fourier transform infrared (FTIR) spectrometer equipped with telescopes to allow long-path analyses. The flat mirror allows rapid manipulation of optical pathlength and analyses over undisturbed terrain.

The detection of the presence or absence of a particular IR absorbing contaminant is diagnostic of what volatile constituents are being released from a site. An even more valuable piece of information would be an estimate of the total flux of that contaminant through the site into the atmosphere over time. Such an estimate requires knowledge of the micrometeorological conditions of the site during the period when long-path FTIR spectrometry measurements are being made. Ideally, the micrometeorological measurements would also be path-averaged over a comparable path being sampled by the FTIR spectrometer. Scintillometry techniques, presently under development by atmospheric scientists at the NOAA wave propagation laboratory in Boulder, Colorado, provide a potential method to link path averaged FTIR spectroscopic profiling and micrometeorological characterization of the near surface atmosphere. Such a combined data base may make estimates of large scale contaminant fluxes possible.

The purpose of this article is to describe our long-path FTIR spectrometer instrument, to present some analyses of various IR absorbing trace gases measured using a long-path configuration, and to discuss means of combining micrometeorological data with long-path trace gas concentrations to obtain estimates of fluxes. In particular, combining of long-path scintillometry with long-path FTIR gas concentration data shows the promise of providing a better tool for quantifying gas fluxes from both natural and polluted sources.

MATERIALS AND METHODS

FTIR Spectrometer

Our FTIR instrument is a Nicolet 740 optical bench interfaced with a Nicolet 660 work station having a storage module capable of storing 344 Mbytes of data. The 740 unit is capable of 0.3 cm-1 wavenumber resolution (3,4). The optical bench is mounted on a wheeled cart in conjunction with a 60 cm diameter Cassegrain telescope. The infrared source (1000-W halogen quartz lamp or globar) is mounted at the focal point of a second 60 cm diameter Cassegrain telescope. The infrared beam is transmitted to a 60 cm square flat mirror and returned to the receiving telescope which focuses it on an adjustable aperture (Fig. 1). The beam is then recollimated and transmitted into the optical bench and subsequently through the interferometer to the detector. The 740 bench is equipped with an Hg-Cd-Te (MCT-A) detector. All sample collection and processing is handled using Nicolet software. Each collected sample can be either a single scan or a composite of a series of scans. Sampling rates are a function of scans per sample and the pathlength of the moveable mirror. Most samples discussed in this paper were collected at 0.5 cm-1 wavenumber resolution. Each scan takes about a second at this resolution. Generally 16 scans per sample were collected although in some cases a 1-minute sampling rate was desired which resulted in 60 scans per sample. Each sample yields an interferogram (Fig. 2) which must then be processed to give an absorbance spectrum (Fig. 3) from which gas concentrations can be calculated. This quantification is performed using a multivariate least squares fit (LSF) program developed by Haaland and Easterling (5). This technique allows quantifying of multiple components whose lines overlap in a given spectral region with better precision and accuracy than can be obtained from a single peak analysis.

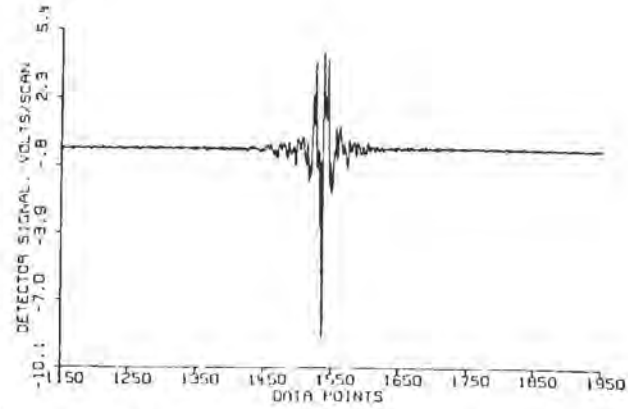


Figure 2. Example interferogram from the FTIR. The energy source was a quartz-halogen lamp, pathlength 407 m, atmospheric pressure 724 torr and instrument resolution 0.3 cm^{-1}.

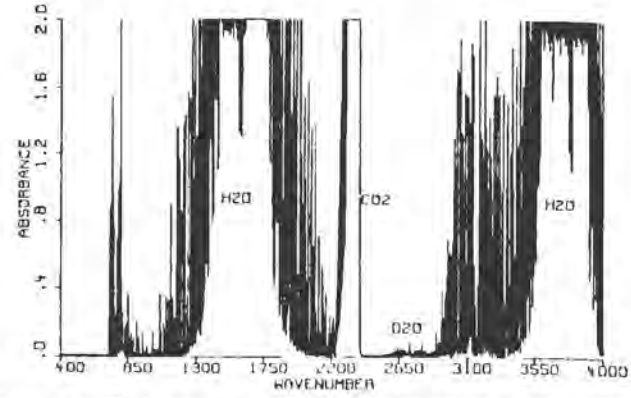


Figure 3. The raw data of the interferogram is processed to give an absorbance spectrum that allows calculation of gas concentrations. This interferogram was collected at Maricopa, Arizona over a cotton field, 10:26 hrs, 12 June 1988, pathlength 407 m.

Optical Scintillometer

Scintillometers are devices that sense the scintillation (intensity variations) in propagating electromagnetic (EM) waves. The refractive index structure parameter C_n^2 characterizes the magnitude of this scintillation. EM scintillation results from turbulent fluctuations in the atmospheric refractive index which in turn result from fluctuations in atmospheric temperature and humidity fields. The scintillometer currently being used is an optical scintillation inner-scale meter. In the experiment reported here, it is deployed on a horizontal path of 150 m at a height of 4 m. This instrument measures the variance of the log intensity of diverging Laser light detected through a 1mm-diameter hole as well as the variance of the logarithm of aperture-averaged intensity from a 4.4-cm diameter, phase incoherent uniformly-illuminated source. The ratio of these variances gives the inner scale of turbulence l_o. The refractive index structure parameter C_n^2 is also determined from this instrument's data. An approximate correction for saturation of scintillation of the Laser variances is computed to extend the range of validity of the instrument. The heat and momentum fluxes are deduced from C_n^2 and l_o using Monin-Obukhov similarity relationships. This is the "inertial dissipation" method of determining these fluxes. In this configuration the instrument can be considered a fluxes scintillometer.

For comparison purposes, a three-axis sonic anemometer having platinum resistance-wire thermometer near its center is also deployed at a height of 4 m on a tower near the optical scintillation instrument. The sonic anemometer measures all three fluctuating components of velocity at a 25 Hz data rate. The correlation of the vertical component of velocity with the streamwise horizontal component gives the momentum flux (divided by air density). The friction velocity U_* is the square root of the negative coefficient of this correlation. The correlation of the vertical component of velocity with the temperature from the resistance-wire thermometer gives the temperature flux (heat flux is temperature flux multiplied by the air's heat capacity.)

RESULTS AND DISCUSSION

FTIR

Much of our initial work has involved testing the utility of the long-path FTIR for monitoring gases from natural as well as anthropogenic origins. Gases that we most routinely analyze include H_2O, CO_2, CH_4, N_2O, and CO but many other gases can also be quantified simultaneously or at some later time. This points up one of the primary advantages of FTIR. Spectra collected can be reprocessed at a future time to look for gases that may not have been of primary interest at the time that the original analysis was carried out. Below are examples of some uses of our long-path FTIR.

Gas Emissions

The FTIR was set up to measure a 100 m path over a small shallow lake on the Isleta Indian Reservation near Albuquerque, N.M on June 26, 1989. A series of samples was taken to establish ambient gas concentrations. The bottom sediments were then disturbed by a person walking around to force degassing of the sediments. A second series of samples was collected during this period (Fig. 4). Emission of CH_4 is readily apparent. These emissions were in fact point source emissions and the measured concentrations denote a mean concentration for the entire 100 m path, most of which was not undergoing degassing. This artificially induced gas emission points up the ability of the FTIR to quantify such emissions but the experiment was designed to test our ability to measure changes in path-averaged atmospheric concentrations and was not meant to estimate gas fluxes.

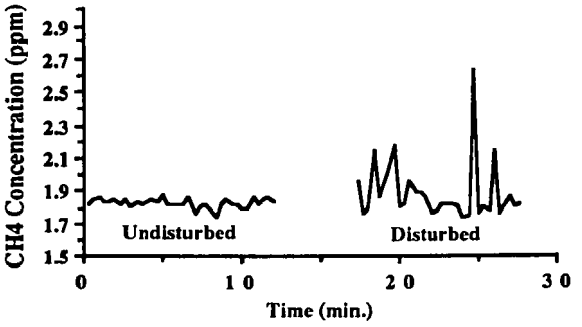


Figure 4. Field demonstration of the ability of long-path FTIR to detect CH_4 emissions from a shallow lake in New Mexico. Measurements were made over the lake on June 26, 1989 before and after bottom sediments were disturbed along a portion of the pathlength.

Gradient Profiling

A primary focus of our study is to develop the capability to measure gas fluxes over a long path which should average out the large spatial variability encountered using small scale techniques such as chambers. Micrometeorological techniques such as Eddy correlation and Bowen Ratio techniques have been shown to be capable of measuring heat and water fluxes under certain ideal conditions. We have explored the possibility of using the diffusivity constants obtained from Bowen stations and eddy correlation stations to plug into a flux gradient equation:

$$F = k \cdot dc/dz \qquad (1)$$

where F is the mixing ratio flux, dc is the difference in a gas concentration (in ppm) measured at two heights and dz is the difference in those two heights. We experimented with this technique over a tall grass prairie site near the Konza Prairie Long Term Ecological Research (LTER) site near Manhattan, KA in July of 1989. The FTIR source and spectrometer were mounted on the lift gate of a truck so that the instrument could be raised from near-ground level at 1 m to a 2 m level. Samples were collected at one height for 10 minutes then moved to the other height for a 10 minute collection period. This procedure was repeated over a 3 day period (July 25, 26, 27, 1989 - Julian days 206, 207, 208).

Our initial results were encouraging as a significant gradient could be detected for most gases. In some cases, such as for water vapor, this gradient agreed reasonably well with that measured by a Bowen Ratio station in the same vicinity (Fig. 5). During some of our measurements the gradient was opposite to that expected. Presumably, this was the result of the rapid changes in surface layer conditions that were missed due to the time required to move the instrument from one height to another and realign it.

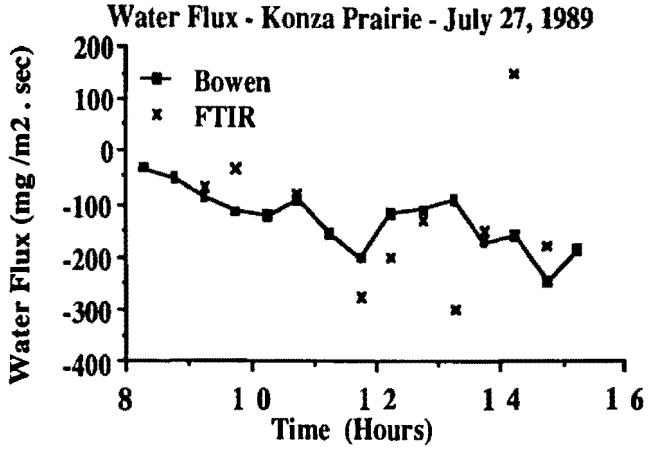
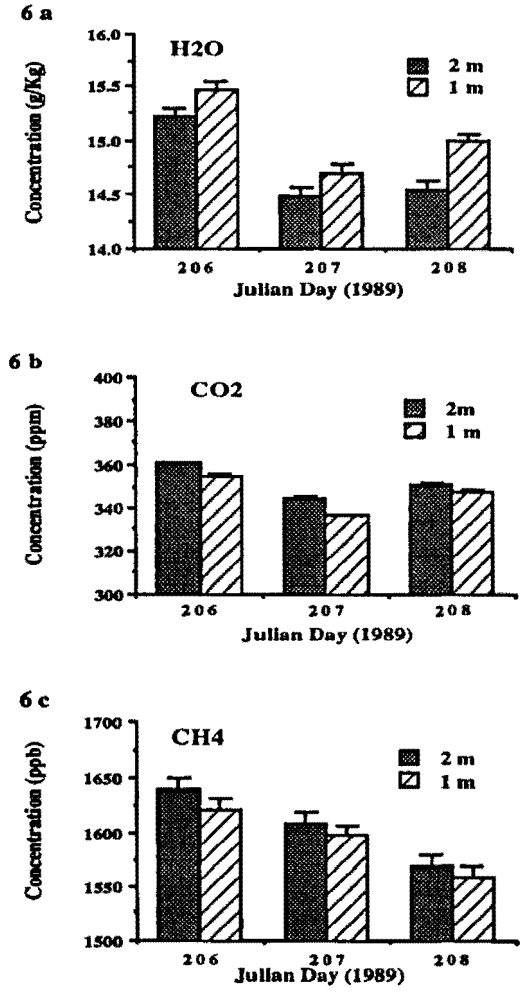


Figure 5. Water fluxes as quantified by long-path FTIR and a Bowen Ratio station at the Konza prairie research area on July 27, 1989. The values are based on the concentration gradient at 2 m and 1 m heights above the ground. Negative values indicate that the direction of the flux is away from the soil and vegetation.

Figure 6 shows the concentration gradients that were monitored for the 3 days of sampling. There are several noteworthy items: 1. Water gradients were always negative as should be the case under stable surface layer conditions (concentration at 2 m < concentration at 1 m).

2. Daytime CO_2 gradients always showed a positive gradient presumably reflecting the active CO_2 absorption by plants. 3. Of even greater significance was the positive gradient for CH_4 coupled with the significant daily decreases in CH_4 concentrations. A rain event on July 23 (Julian day 204) was followed by a drying period for the remainder of the sampling days. The sequence is likely due to a high rate of oxidation of CH_4 by methane oxidizers in the local soils coupled with a decrease in atmospheric concentrations as the general area dried. The regional source of the CH4 is not known. 4. CO concentrations also demonstrated a constant drop through the sampling period. A shift in the wind away from the direction of an interstate that ran just north of the site to a more southeasterly direction is a likely explanation for this change as anthropogenic sources far exceed biological sources.



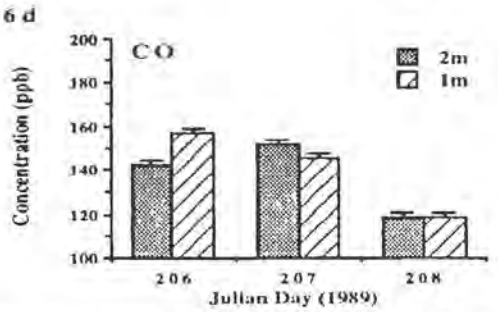


Figure 6. Concentration gradients for H_2O (6a), and trace gases - CO_2 (6b), CH_4 (6c) and CO (6d) measured by long-path FTIR on the Konza Prairie research area during the period July 25-27,1989.

Air Quality Monitoring

In an effort to explore the possibility of using the FTIR to monitor air quality in the metropolitan area of Albuquerque, N.M., we set up the instrument to analyze over a path of about 500 m. A portion of this path was just over the level of automobile traffic on one of the main traffic arteries in Albuquerque. The sampling period extended from rush hour through the period of peak fireplace smoke emission. A primary purpose of the experiment was to determine if methyl chloride (an indicator of wood smoke source) could be detected. Concentrations of this gas were found to be too low to be detectable even at the 500 m pathlength due to both low concentrations and relatively poor absorption by this molecule. However, examination of the spectra showed that a number of gases which are not normally detectable under clean air conditions were present in elevated levels. Ethylene and methanol were easily detected while ammonia and formaldehyde were also above detectable concentrations.

These tests were carried out on two nights during the winter of 1989-90. The first was on Dec. 8, 1989. This had been declared a no-burn night by the City's Environmental Health Department meaning that no wood burning in fireplaces or stoves was permitted. Gas sampling began at about 5:40 PM and continued until 11:00 PM. Figure 7 shows the level of gas concentrations for N_2O, CO, CO_2, CH_4, H_2O and C_2H_4. Values are means of 10 - one minute samples. All of the gases, with the exception of N_2O, increased until about 7:30 PM at which time they began a steady decline. The second sampling date was Jan. 12, 1990. This night was not declared a no-burn night by the city although it was expected to be marginal with respect to weather conditions which could assist with the dispersal of gases emitted by wood burning or automobiles. Indeed, surface wind conditions for both nights were quite similar. On Jan. 12, most of the data for the period 7:40 to 9:15 were lost due to problems with the FTIR instrument (Fig. 8). As with the Dec. 8 date, all gases tracked each other with the exception of N_2O which showed an inverse pattern. However, unlike the December collection, the gases showed a steady increase from 5:00 PM until about 10:00 PM when they began to decline. While the time of peak levels was different, concentration of peak levels for all gases were generally comparable. Table 1 gives the maximum, minimum, and mean concentrations for all of the gases measured for the two sampling dates. The sampling intervals for the two dates were not the same although total sample numbers were similar.

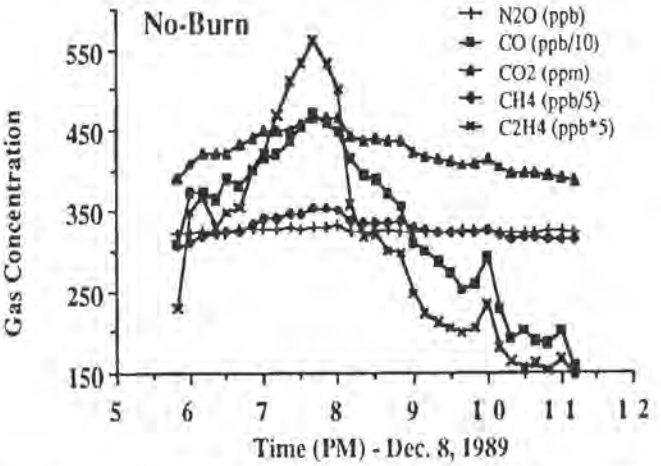


Figure 7. Trace gas concentrations measured by long-path FTIR during a no-burn (no wood burning allowed) night in Albuquerque, New Mexico. Values are concentrations from the means of 10 - 1 minute. samples on the night of December 8 1989. Pathlength was 500 m.

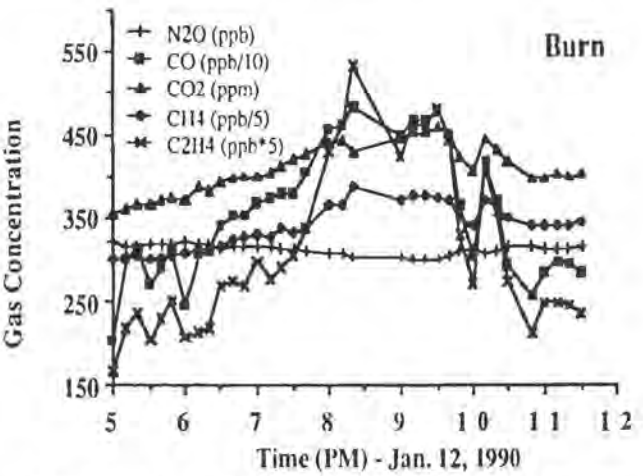


Figure 8. Trace gas concentrations measured by long-path FTIR during a night when wood burning was allowed (Jan. 12, 1990). Pathlength was 500 m.

Table 1. Mean, maximum and minimum concentrations of trace gases measued with long-path FTIR on Dec. 8, 1989 (No-Burn) and Jan. 12, 1990 (Burn) in Albuquerque, NM.

		Mean		Maximum		Minimum	
Gas	Units	No-Burn	Burn	No-Burn	Burn	No-Burn	Burn
N_2O	ppb	328	314	337	324	317	297
CO	ppb	3316	3438	4980	5274	1518	1763
CO_2	ppm	426	405	472	463	386	353
CH_4	ppb	1659	1678	1790	1946	1522	1494
H_2O	ppm	2037	2204	2181	2300	1926	2127
CH_3OH	ppb	7.7	15.2	31	30	0	3
C_2H_4	ppb	61.6	59.2	120	110	23	30
NH_3	ppb	16.7	19.7	36	26	9	14

While the small sample size precludes any definitive conclusions as to the relative contribution of wood-burning and automobiles to the elevated levels of the sampled gases, some things are suggested by the data.

1. The timing of the peak CO level on the burn night suggests that wood-burning contributes a considerable quantity of CO to the city air when permitted.

2. Based on mean and maximum CO levels for the two nights, firewood burning contributes proportionately more CH_4, CH_3OH and NH_3 than vehicle emissions while automobile exhaust shows a greater contribution of CO_2, N_2O and C_2H_4.

3. On the burn night, the CO concentrations were extremely erratic during the time of maximum traffic. This indicates a nearby source due to heavy vehicle traffic. Later in the evening, the concentrations were less erratic suggesting a less proximate source which would likely be the case for wood smoke.

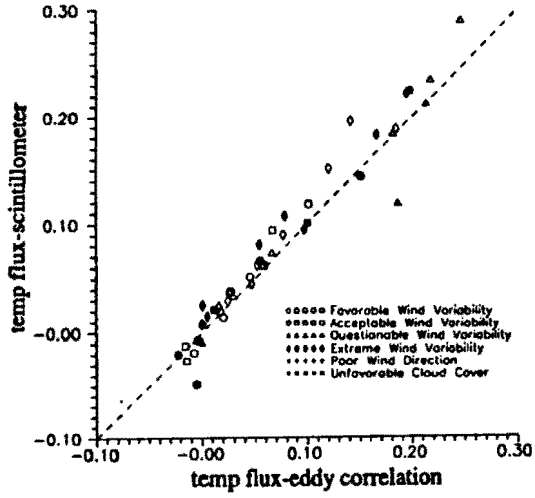


Figure 9. Temperature flux measured by the flux scintillometer compared with temperature flux from eddy correlation. Units are ^oC m s^{-1}.

Scintillometry

Comparison of the temperature flux and friction velocity determined from the fluxes scintillometer with that obtained from the point sensors are shown in figures 9 and 10. These data show that the fluxes scintillometer gives good values even in nonideal atmospheric conditions. Even during periods of intermittent cloud cover and unfavorable wind conditions, when the validity of the Monin-Obukhov similarity is limited or unknown, measurements obtained from the fluxes scintillometer compared well with the point sensor measurements.

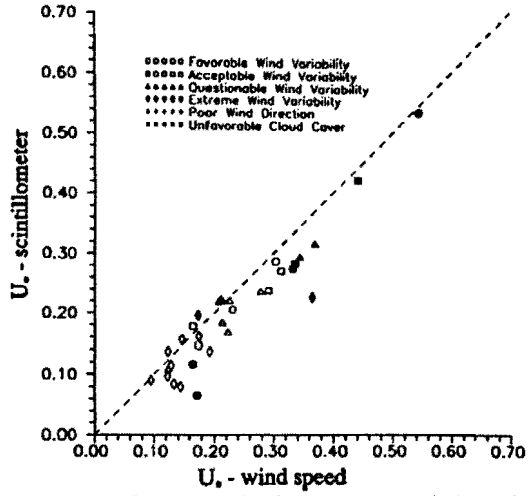


Figure 10. Friction velocity measured by the flux scintillometer compared with friction velocity deduced from wind speed, surface roughness, and eddy-correlation temperature flux. Units are m s^{-1}

Integration of FTIR and Scintillometry

Our next step in the integration of the FTIR system with the long-path measurement of water and heat fluxes involves two steps. The first is the development of 2 scintillometers by the WPL. One uses a 10.6 μm wavelength Laser source; the other uses a 3 mm wavelength Gunn diode source. The combination of thes two scintillometers with the fluxes scintillometer allows long-path measurement of the fluxes of latent heat sensible heat and momentum. Modification of the FTI system to allow it to measure gas concentrations at tw heights almost simultaneously is the second proposed modification. This will be accomplished through the use of a periscope system that will eliminate the lengthy delays in realignment that are necessary when the entire instrument must be moved vertically as was the case in th Konza Prairie experiment.

Two related methods will be used to derive gas fluxes from FTIR and scintillometry measurements. These are outlined

by Andreas (6). The first uses the Obukhov length L from measurement of vertical momentum, u_* and the temperature and water vapor fluxes. These, combined with the gas concentrations, G_1 and G_2 measured at the two heights z_1 and z_2 by the FTIR, will give an estimate of the corresponding flux scale g_* from the relation

$$\Delta G = G_2 - G_1 = (g_*/K)[\ln (z_2/z_1) - u_g(z_2/L) - u_g(z_1/L)] \quad (2)$$

as suggested by Hicks and Liss (7). The gas flux is then given by $F_g = -u_* g_*$. The second method will use a modified Bowen ratio method which is based on the belief that u_g should be the same for all conservative scalars. In particular, the difference in water-vapor mixing ratio, ΔQ, also satisfies equation 2

$$\Delta Q = Q_1 - Q_2 = (q_*/K)\ [\ln (z_2/z_1) - u_q(z_2/L) - u_q(z_1/L)] \quad (3)$$

where q_* is the water vapor flux scale. Since $-u_* g_* = F_g$ is the gas flux we are seeking and $-u_* q_* = F_q$ is the water flux, and since we assume ug=uq, Equations 2 and 3 yield:

$$F_g = F_q\ (\Delta G/\Delta Q) \quad (4)$$

To use equation 4, we obtain ΔG and ΔQ from the FTIR and F_q from the scintillometers.An important assumption of the application of gradient profiling is that the various gases for which fluxes are being derived behave similarly to heat and water vapor in the atmosphere. The scintillometry system measures path averaged fluxes of heat, momentum and humidity with scintillometers measuring at 1 mm, 10μm, and 1μm. Gaseous flux estimates are then based on these measurements of atmospheric conditions. All gases might not have identical flux profile relationships that lead to equation 2; that is it may be that $u_g \neq u_q$ (8). It will be critical in future work to determine the flux-profile relationships for trace gases.

CONCLUSIONS

Our research to date with long-path FTIR spectroscopy and path-averaged scintillometry has resulted in the following conclusions.

1) Long-path FTIR has the analytical sensitivity to measure numerous atmospheric trace gases both anthropogenically and naturally derived at ambient concentrations.

2) Increased pathlength commonly adds to the sensitivity with which we can measure atmospheric trace gases.

3) Optical scintillometry is a potential tool for measuring path-averaged fluxes of heat, momentum and humidity in the atmosphere over the same path in which gas concentrations are being determined by long-path FTIR.

4) Gradient profiling using long-path, path-averaged FTIR spectroscopy has been used to show distinct vertical structure of H_2O, CO_2 and CH_4 in the atmosphere over a prairie.

5) Coupling gradient profiling of atmospheric trace gases with the long-path FTIR and path averaged scintillometry is a promising means to begin to estimate gas fluxes at larger spatial scales from various landscapes. Applications are seen for both field screening of hazardous waste and toxic chemical emissions to the atmosphere and for many global greenhouse gases.

ACKNOWLEDGEMENTS

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DISCUSSION

JOHN EVANS: I was just curious how you calibrate the instrument and secondarily, what sort of precision and accuracy of the path length average concentration you get for something, like methane, for example.

DOUG MOORE: We calibrated FTIR using white cell, .25 meter white cell in the laboratory. And for things like methane we get detection limits of about 30 ppb, plus or minus.

JOHN EVANS: What sort of precision and accuracy can you get on normal measurements say, in the atmosphere?

DOUG MOORE: Well, yes, that's going to be path dependent. I'm not sure I understand. What numbers do you want it in?

JOHN EVANS: Well, we see a graph up there with some numbers up and down. Are they 1%, 10%, 50% accuracy precision?

DOUG MOORE: We're better than 5% accuracy. Probably better than that on the long path.

TOM PRITCHETT: When you were actually measuring this flux, do you have to shine the beam directly over the source, or do you shine the beam downwind of the source in looking at any downwind transport?

CLIFF DAHM: Our applications are quite a bit different than many other people who are looking at point source. We're not looking at point source emission. We're looking at something that's broadly distributed across the environment. So, what we're looking at is something where we really need to know something about fetch length from which the sources are generated. But we're not looking at a point source. If we are looking at a point source, we would go into point source analysis, we would have to be downwind and perpendicular to that point source, or over that area of point source. What we're looking at, though, is landscape emissions of things that tend to be distributed rather, at least reasonably, uniformly over the environment.

TOM PRITCHETT: So, basically you're looking at the flux as coming directly underneath your beam, essentially?

CLIFF DAHM: That's a very difficult question as to exactly where those gases are coming. They're coming from downwind. It's very dependent, of course, on wind field conditions at the time you're making the measurements. But you can be generating input terms to your vertical structure of the atmosphere that can be anywhere up to 100 times the height you are above the ground, 100 times upwind of that direction. Again, it depends very much on meteorological conditions at the time of the emission.

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