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**EVALUATION OF A FOURIER TRANSFORM INFRARED
CONTINUOUS EMISSION MONITOR FIELD TEST
AT A TSCA INCINERATOR***

by

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EVALUATION OF A FOURIER TRANSFORM INFRARED CONTINUOUS EMISSION MONITOR FIELD TEST AT A TSCA INCINERATOR*

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ABSTRACT

A Fourier transform infrared (FTIR) spectrometer was field tested as a continuous emission monitor (CEM) at the Toxic Substances Control Act (TSCA) incinerator at K-25 in Oak Ridge, Tenn., from August 23 to September 3, 1993. This paper reports results obtained from this field test.

The FTIR spectrometer and the long-path cell used for the field test were specially designed and constructed, so that optical alignment of the system can be easily performed in the field. The system was tested in the laboratory and then in the field for instrument stability and signal-to-noise ratio. Time interval required for taking a new background spectrum was determined. It appears that the system performs well both in the laboratory and in the field.

The field test followed a standard operation procedure (SOP), developed for the test, based on a proposed EPA protocol for applying FTIR in emission testing. Sixteen compounds were selected as target analytes. Ethylene was used as a calibration transfer standard to ensure that spectral performance of the FTIR spectrometer in the field is consistent with that in the laboratory. Spike tests were regularly conducted with a known concentration of a mixture of six compounds and also with SF₆ to check the accuracy of the monitoring system.

Data sampling, processing, and reporting were automated to collect data every 10 min, and data were collected throughout the test as long as liquid nitrogen was available in the detector. The instrumentation and software performed flawlessly.

Although the field test was a success, further improvement is necessary. Suggestions for revising the SOP and the proposed EPA protocol are discussed.

INTRODUCTION

Incineration is constantly under public scrutiny because of concern about potential toxic emissions. The concern is heightened because incinerators are not continuously monitored for toxic organic and inorganic products of complete and incomplete combustion. Monitoring consists, initially, of a trial burn that determines safe operating conditions and provides one-time analyses of emissions. Afterwards, facilities will continuously monitor only carbon monoxide (CO) and carbon dioxide (CO₂). There is significant public concern that the trial burn is not adequate and a fear that unscrupulous operators will emit pollutants when burning feeds with low Btu levels. The picture is further complicated by the Clean Air Act of 1990, which mandates the monitoring of air toxics. A continuous emission monitor (CEM) would address these public concerns and demonstrate that an incinerator is not emitting toxic pollutants.

There is no single technology that can be used to monitor all air toxics. Organic pollutants can be monitored using Fourier transform infrared (FTIR) spectrometry and mass spectroscopy (MS). Techniques that would monitor metals are still in the early experimental stages.

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Both FTIR and MS techniques have been used to monitor incinerators. The methods are not necessarily competitive. FTIR excels at measuring volatile organic components, CO, hydrogen chloride (HCl), and components in complex matrices. Mass spectroscopy has lower detection levels and excels at identifying components with high masses and masses for which there are no other interfering masses. Mass spectroscopy cannot differentiate molecular ions from fragments that have identical masses. Such a lack of differentiation is a serious problem in the monitoring of methane and ethylene, which are common products of incomplete combustion (PICs). This paper focuses on FTIR monitors.

FTIR technology has been used to identify and quantify components in mixtures for over 20 years. The instrumentation is relatively straightforward. The spectrometer contains an emission source, focusing optics, an interferometer, and a detector. The sample is contained in a heated long-path cell. Path lengths of 5-10 m are commonly used. These cells have a volume of approximately 2 L. A slip-stream of stack gas enters the cell at a rate of 1 L/min. Data collection takes less than one minute.

The Environmental Protection Agency (EPA) has acted on the provisions of the Clean Air Act and issued a protocol for utilizing FTIR spectrometry as a CEM. We have adapted this protocol into a standard operating procedure (SOP). The protocol was written by Entropy Environmentalists, Inc., with input from laboratories such as ours that have been developing FTIR technology. We have previously used FTIR Spectrometry to monitor an incinerator in the laboratory with the objective of developing a system suitable for use in the field. This "prototype" FTIR system was field-tested at the Toxic Substances Control Act (TSCA) incinerator at K-25 in Oak Ridge, Tenn, from August 23 - September 2, 1993. During this field test, the SOP was tested, and results were obtained that can become the basis for the EPA to accept FTIR data.

There are no EPA-accepted FTIR methods. The EPA has developed the protocol to define data-quality criteria. We have adapted the protocol and some requirements commonly found in EPA methods into our SOP. Standard EPA-method requirements are not directly translatable into an FTIR incinerator-monitor method. We will discuss our adaptations in the context of the field test results.

EXPERIMENTAL

Instrumentation

All measurements were made using a Mattson 7020 Infrared Spectrometer (ATI, Madison, Wisc.). The instrument contains a hot-wire source and a mercury cadmium telluride (MCT) detector. It can be operated manually using Mattson's WinFirst software and operated automatically using software specially written for this project. The instrument was connected to a personal computer (486DX50) through a serial port. Data were collected at 0.5 cm^{-1} resolution. Sixty-four scans were coadded and saved as a single interferogram file. This interferogram was Fourier-processed using Beer-Norton medium apodization.

A heated, variable-path-length, long-path cell was interfaced to the interferometer. The cell was specially designed (Infrared Analysis, Anaheim, Calif.) for this project to facilitate optical alignment in the field.

Sampling Handling

A slip stream of incinerator exhaust was removed from the stack and transported through a heated transfer line to the heated long-path cell. A series of heated valves allowed the slip stream to enter

the cell only, to enter the cell mixed with calibration gas, or to bypass the cell completely. The stack flow rate was kept at approximately 1 L/m. All calibration gases were purchased from Scott Specialty Gas. They consisted of sulfur hexafluoride (SF₆), ethylene, and a calibration mix consisting of benzene, methane, chlorobenzene, trichloroethylene, toluene, and 1,1,1-trichloroethane.

Analytical Procedure

Every morning and periodically throughout the day, the incinerator-exhaust flow to the cell was stopped. The cell was evacuated and back-filled with nitrogen to obtain an instrument background spectrum. After the background check, the cell was filled with ethylene, the calibration transfer standard (CTS), and the stability of the instrument determined. The cell was again flushed and filled with SF₆ standard, followed by the mixture standard. After these standard checks, the cell was opened to the stack flow.

Sample spikes were introduced periodically. At least twice per day, the three gas standards were mixed with the stack flow. The flow rate of each gas was monitored so that the spike concentration could be determined.

Analysis Software

A combination of commercially available and internally written program modules controlled by a BASIC master program comprised the data analysis software. The software operated the instrumentation, analyzed the data, and displayed and stored the results.

Specially written BASIC programs calibrated the instrument and ran it for a specified period of time. BASIC programs called for initiation and calibration (INIT) of the instrument and for data collection (SAMP). The data collected were automatically named and date-stamped.

Data analysis was performed using the partial least squares (PLS) package included with LabCalc software (Galactic Industries, Salem, N.H.). This software was modified to start automatically in the background, perform the data analysis, and store the results in a specially formatted data file. Our software then accessed this file and displayed the results on a screen by means of special graphics software.

The program continued to execute until an operator stopped it or the detector ran out of liquid nitrogen. Modules will be added to automatically collect background data at specified intervals, to do quality assurance/quality control checks, and to report if results are not within EPA performance specifications.

EPA PROTOCOL CONSIDERATIONS

The protocol establishes analytical quality control standards for data acceptance by the EPA. The SOP was written to convert the protocol into a series of procedures that could be tested during the K-25 field test. The protocol requires extensive pretesting preparations and data quality assurance checks.

Pretest preparations emphasize the importance of meticulous documentation and use of a CTS to determine if reference spectra recorded under one set of conditions can be used to analyze sample spectra acquired under a different set of conditions. Ethylene is the recommended CTS. Background information such as (1) the acquisition of a robust training set using pertinent static or dynamic methods and (2) the evaluation of analytical regions and software coordinated with validation of the training set are required. Instrument stability and performance were verified either

by using existing software or by injecting a calibration standard to determine if outliers exist when the data are compared to the training set.

Standard EPA methods require a blank sample, internal standard, calibration standard, matrix spike, and matrix spike duplicate. The protocol also requires instrument stability verification and a CTS to validate the calibration spectra obtained before field testing.

Calibration Transfer Standard

FTIR methodology is considerably different than standard methods, and specific techniques cannot be directly transferred. The CTS was designed specifically for FTIR, so it can easily be run. Before the field test, CTS tests were conducted before and after collecting reference spectra for the calibration training set. In the field, CTS tests were conducted every day that data were collected. The measured ethylene concentrations were always within 4% of the 121 ppm value of the CTS. The average measured concentration of ethylene was 120.0 ppm, and the average percent deviation was 2%. This equivalence demonstrates that the FTIR data collected during the field test are equivalent to the laboratory data. Thus, CTS data are within the acceptable limits required by the protocol, and the calibration training set and the quantitative method based on the laboratory test can be used to analyze the field-test data.

Instrument Throughput and Stability

The instrument response is summarized by the signal-to-noise (S/N) ratio: the higher the S/N ratio, the more sensitive the instrument. Conversely, as instrument performance begins to deteriorate, the S/N ratio decreases. Ultimately it will reach a point at which the results obtained are not reliable.

To ensure stable and reproducible conditions for monitoring throughput, the cell was evacuated and filled with pure nitrogen. The peak-to-peak intensity of the interferogram, measured by the detector, represents a total energy throughput for all wave numbers. The system was stable during the field test. The average throughput was 2.04 V; variation was within 3% of the average value.

The throughput of the system did not change after 7 d of continuous monitoring, indicating that the cell and the mirrors were not contaminated by the flowing gas stream and that the cell design is adequate for continuous monitoring. The system will be field tested for an extended period of time to determine how long the cell will perform adequately before cleaning is required.

Instrument Noise

The sensitivity of the instrument is determined by its noise. As the noise decreases, the sensitivity is enhanced by the same factor. The noise is determined from the ratio of two background spectra. Ideally, the background spectra will be identical, and the noise will be 0 absorbance units (AU). Our objective was to minimize the noise by designing efficient optics and providing a high-temperature source. The instrument noise was within the limits defined by the EPA protocol.

Spectral noise was evaluated at three different wave-number regions: 3100-2700 cm^{-1} , 2200-2000 cm^{-1} , and 1100-900 cm^{-1} . Noise levels were stable, averaging 0.0012 AUs at 3000 cm^{-1} , 0.00034 at 2000 cm^{-1} , and 0.00027 at 1000 cm^{-1} . The noise at 3000 cm^{-1} was four times higher than at the other two regions because of the relatively low sensitivity of the detector in this region and the low source temperature.

A spectral feature can be identified when the S/N ratio is equal to or greater than four; the lower detection limit for each compound can then be calculated from its peak height in the spectrum.

Table I lists the current lower detection limit of each target compound. This value is based on single-compound spectra and represents what the FTIR spectrometer could attain during this test. Multiple-component mixtures are measured in emission monitoring. The detection limit for each compound is affected by the noise, the spectral interferences from other compounds, and the software used to quantify the data. Therefore, the actual detection limit for the field-test data would be not as good as the values listed in **Table I**.

Instrument Background and Sample Blank

The EPA methods normally require a sample blank. For example, in soil sample analysis, a NIST traceable soil with no contaminants is selected. The soil is extracted and analyzed alongside target soil samples. The objective is to determine if the extraction and analysis process is introducing contamination that will result in false positives. Unfortunately, there is no such thing as an incinerator "blank." We propose a nitrogen blank, as an alternative which will be obtained when a new background spectrum is obtained. The background spectrum is obtained by evacuating the cell and back-filling with pure nitrogen. Immediately after this background spectrum is obtained, the cell can be reevacuated and refilled with nitrogen. This second fill can be considered the blank and analyzed as a sample.

The frequency of collecting background spectra must be experimentally determined. Many EPA methods require a background or blank sample run every 15 samples. Because the FTIR spectrometer is operating continuously, there is no prescribed time for collecting a background spectrum. If a sample is collected every 10 min, fifteen samples would correspond to a background collection every 2.5 h.

The background spectra from the field test were evaluated by obtaining a ratio of background spectra obtained during the day using the first background spectrum collected as the denominator. The resulting spectrum is called the "100% line" (referring to 100% transmission, which corresponds to 0 AU). The two critical features of the 100% line are the increase in noise and the change in slope. An increase in noise adversely affects the detection level and the quantitative accuracy. After evaluating the change in slope, we determined that a background run every 6 h would be sufficient to maintain data quality. We selected 4 h as an acceptable interval, to allow for a margin of error. The ratio of the background spectrum obtained after 4 h to the previous background spectrum can also be used as a sample blank to verify that there was no cell contamination.

Internal Standard

Many EPA methods require an internal standard. An internal standard is a component that could not possibly be present in the sample that is added to every sample to validate the quantitative data. The instrument should detect the standard component at its spike concentration. An appropriate internal standard for emissions samples is sulfur hexafluoride (SF₆). It is never a product of combustion and is nontoxic. Continually adding SF₆ to the effluent is unnecessary and would only add to atmospheric pollution. We propose adding a single SF₆ spike after running the sample blank to demonstrate that the system is operating.

During the field test, the sample line was periodically spiked with SF₆. A 10.1-ppm, SF₆-calibrated gas source was allowed to mix with the stack gas at periodic intervals to test the instrument. *Fig.1* shows sample spectra taken before and after the injection of SF₆. By subtracting the H₂O and CO₂ peaks, the distinct SF₆ peak can be clearly identified. Over a two-day period, the SF₆ spike was detected at an average deviation of 5.6%. The results are shown in **Table II**.

(Place Fig. 1 here)

Matrix Spike

Target analytes that would be present in a contaminated sample can be used as a matrix spike. The matrix spike is added to a sample to demonstrate that the system can detect target pollutants should they be present. Most EPA procedures require a real sample to be split in half. One of these would be spiked and the other analyzed without a spike. This procedure is not possible for incineration monitoring because there are only single samples. However, because analysis time is short, the average of the results obtained before and after a matrix spike can be obtained and subtracted from the quantities detected in the matrix spike.

The matrix spike selected consisted of six components potentially present in the stack gas. The six components were benzene, trichloroethylene (TCE), chlorobenzene, 1,1,1-trichloroethane, toluene, and methane. Except for methane, the detected concentrations of spiked compounds were close to the predicted values. It was noticed that concentrations of water and CO₂ in the stack emission on September 1 were not stable, and values could not be calculated. Hence, these data from September 1 are excluded. The deviations of the detected values from the predicted values are below 25% for the remainder of the data.

The calculated values for benzene agree within 20%, with an average deviation of approximately 10%. The TCE values were within 7% of the predicted value, with an average deviation of 5%. The methane deviation was higher. Further work will be done to improve the analytical results. *Table III* lists the quantitative results for the mixture.

Initial methane data analysis indicated a relatively large error. The data were reevaluated and different analysis methods were investigated. Methane analysis is complicated by the presence of methane in the stack gas. When the matrix spike is added to the stack gas, the concentration of methane naturally present must be estimated and added to the quantity in the matrix spike. The high level of water (~40-50%) in the stack gas also complicates quantitation. When the pure calibration mixture was run without stack gas, quantitation was within 2%. We believe that advanced data analysis using a digital filter of time-domain data will reduce, if not eliminate, the effect of high water concentration.

RESULTS OBTAINED AT THE TSCA INCINERATOR

The incinerator effluent was exceptionally clean throughout the two-week test period. Detected components were present only in low concentrations. In this section, we will review our results for each analyte under investigation.

There was one serious anomaly in the data. Every time we opened the transfer line into the cell, the first sample analyzed contained high concentrations of CO, methane, ethylene, chlorinated hydrocarbons, and benzene. The chlorinated hydrocarbons and benzene were found only in this first sample. The CO, methane, and ethylene concentrations were much higher than those obtained in subsequent samples. We believe this first sample is an artifact of our sampling system, which does not have a bypass valve for the stack gas when the gas is not passing through the cell. When the sampling system was shut down to run background checks or standards, the flow through the transfer line was stopped. It appears that the gas in the line had the effect of concentrating PICs, probably by dissolving them on the teflon surface. These PICs outgassed when the flow was resumed. The effect is most pronounced with CO. After the first sample, all PICs except CO were no longer detected, or they returned to a stable value. The sampling system used in the future will have a bypass installed to eliminate this problem.

Carbon Dioxide and Carbon Monoxide

Both CO and CO₂ are continuously monitored at the TSCA incinerator. However, the results obtained by using these CEMs are not directly comparable to the FTIR results. The CEMs, manufactured by Siemens, are calibrated daily. They can only function after the water is removed by condensation. The dry air then enters the CEMs. The concentration obtained is normalized on the basis of a 7% oxygen content. The FTIR spectrometer sampling system neither removes the water from the stack gas nor normalizes on oxygen concentration. Water removal would result in precipitation of some CO and CO₂, because they are both soluble in water; CO₂ is much more soluble than CO. Given these differences, the results obtained by using these two different methods agreed reasonably well.

The results for the two techniques were recorded and compared over the test period. The two CO₂ monitors usually correlated well. The TSCA CO₂ monitors registered concentrations of 6.5-8%, while the FTIR monitor showed concentrations in the 3.5-4.5% range. Carbon dioxide was the only analyte that did not show a higher concentration when the sampling line was first opened. In general, the concentrations obtained using FTIR did not vary as much as those obtained by the TSCA monitors. *Fig. 2* shows typical CO₂ and analytical standard spectra. The top and bottom spectra show the amplitude of sections of CO₂ bands for 6.6 and 2.1% analytical standards in the 940-930 cm⁻¹ region. The middle spectrum shows the stack gas spectrum. The stack-gas spectrum shows some additional water bands. Analysis of the stack-gas sample indicated a 4% CO₂ concentration, which correlated well with these standards.

(Place Fig. 2 here)

Initially, there was a large deviation between the CO concentrations obtained by the incinerator monitors and the FTIR spectrometer. Also, the two Siemens incinerator monitors had values that differed significantly from each other. Siemens Monitor 1 showed CO concentrations of 3-6 ppm, while Siemens Monitor 2 had concentrations of less than 2 ppm. The difference between the two Siemens monitors was often a factor of two to three. The first FTIR data collected had a much higher concentration of CO than either TSCA monitors. On the first day of data collection, most CO concentrations were measured between 60 and 80 ppm. However, during the field test, the CO concentrations continually decreased. The decrease became most apparent after August 31, when the sample was allowed to flow into the cell through the transfer line even when data were not being collected. By the afternoon of September 1, the CO concentration detected (sample CIA136) using the FTIR spectrometer was 4.4 ppm, compared to the Siemens' values of 4.65 and 2.35 ppm.

It appears that the large initial values of CO were artifacts of the transfer line. This problem can easily be avoided in the future by installing a bypass valve.

The concentrations obtained by using the FTIR spectrometer are based upon the peak area of selected CO absorbances. In *Fig. 3*, part of the CO-absorption-band region for a stack-gas sample is overlaid on the same region for an 86 ppm standard. The spectral absorbance of the stack gas and the standard are almost identical. The CO bands are so strong that water did not have to be subtracted.

Because of the different methods used to monitor CO and CO₂, it would be valuable to place a Siemens CEM immediately after the cell or an FTIR spectrometer at the output of the on-line Siemens monitor to determine if the differences are real or due to the effect of precipitation of water.

(Place Fig. 3 here)

Ethylene

FTIR spectroscopy is the only technology that can continuously monitor ethylene. It is the recommended calibration transfer standard in the EPA protocol, but continuous monitoring of ethylene is not required, because there is no monitoring technology. Ethylene is a product of incomplete combustion and will appear in high concentrations if an incinerator is upset. Ethylene standards ranging from 3 to 102 ppm were run for quantitative analysis. No ethylene was detected at or above 3 ppm except in the first sample whenever the transfer-line valve was opened. As discussed earlier, the first sample always contained a higher level of emissions.

Ethylene was detected below quantitation levels at approximately 0.5-1 ppm. However, the S/N ratio at these concentrations is only 1, and a positive identification is not possible at this time. We anticipate that advanced data analysis methods should enhance the S/N ratio, so that detection is possible at high parts per billion levels.

Sulfur Hexafluoride

Sulfur hexafluoride was entered into the data set because it was used as a matrix spike and because it absorbs in the same region as several other analytes and, thus, can be used as a test for false positives. It would not be a normal effluent from the incinerator. As expected, it was not detected in the stack gas, but it was always detected when used as a matrix spike.

Hydrogen Chloride and Chlorinated Hydrocarbons

Hydrogen chloride is a product of complete combustion and is produced in the incinerator. The facility removes it by passing the effluent through several scrubbers. The lye scrubber is exceptionally efficient. No HCl was detected in any stack sample, including the first sample after the transfer line was opened.

Chlorinated hydrocarbons are products of incomplete combustion. They were detected in only the first sample after the transfer line was opened and then only at levels below our quantitation limit.

Methane and Benzene

Methane and benzene are critical compounds that need to be monitored. Tests using our laboratory incinerator have shown that methane is one of the first PICs formed when the incinerator is upset. When methane fuel is used to increase the Btu value of the material being burned, it is a direct measure of combustion efficiency. Methane is not currently monitored. A flame ionization detector (FID) monitors total hydrocarbons where quantitation is based on the response factor of methane. "Total hydrocarbons" is defined as everything that burns. Hence, the FID would report carbon tetrachloride as a concentration of methane.

We detected less than 20 ppm of methane in the stack gas. Our quantitation may be biased on the high side on the basis of the matrix-spike results, which were also high. In *Fig. 4*, spectra for a methane standard, stack gas, and a water standard are plotted. The methane peaks are noted by the vertical lines. The presence of methane at a concentration of approximately 16 ppm is clearly seen.

Benzene is also an important measure of combustion efficiency. Tests using our laboratory incinerator have shown that benzene is formed in an incinerator when it is upset. A test using initially a pure methane feed and later a toluene feed produced benzene when the incinerator temperature was reduced. Because benzene is so thermodynamically stable, it is one of the first compounds formed when incinerator operating conditions are not correct.

The matrix-spike-test results demonstrated our ability to detect benzene within an error range of 20%, even when high concentrations of water were present. During this test, we did not detect benzene in any stack sample except the first one collected each time the transfer line was opened. The detection of benzene is difficult because it has a broad-band spectral absorbance that overlaps with that for water. The presence of higher water levels would increase the error range, and lower water levels would improve detection levels. The fact that we could detect benzene in the matrix spike demonstrates that the incinerator did not emit this pollutant at levels approximating the detection level.

Conclusion

The development of a CEM for incinerators has significantly progressed in the last year. The EPA protocol has been field-tested at a TSCA incinerator. The SOP that was written from the protocol demonstrated that the protocol can be followed. Such standard EPA requirements as sample blanks, internal standards, and matrix spikes can still be used, but they require modifications in their definitions to be appropriate for incineration monitoring. The calibration transfer standard was successfully used as a new requirement for FTIR-based incineration monitoring and as a replacement for a calibration standard.

The results obtained from monitoring the incinerator showed very low emissions. There was general agreement between the independently obtained CO and CO₂ results. We detected a low level of methane; other PICs were not detected.

From this data, we can state that FTIR spectrometry is a valid method for use as a CEM for incinerators. It can be used to detect virtually all of the components now detected in a volatile organic analysis sampling train (VOST) followed by laboratory gas chromatography/mass spectroscopy (GC/MS) analysis. Future work will focus on lowering the detection levels and improving quantitation.

Figure 1. Detection of SF₆ Spike in Stack Gas at K-25.

Figure 2. Comparison of CO₂ Detected in Stack Gas with Two Analytical Standards

Figure 3. Comparison of CO Bands in an Early Stack Gas Sample with an Analytical Standard

Figure 4. Comparison of Methane Present in the Stack Gas with a Methane Standard.

TABLE I. Detection Levels for Selected analytes

Analyte	Concentration, ppm	Absorbance, AU	Noise, AU	Detection Level, ppm
Benzene	20.3	.024	.0012	4.0
Methane	10.4	.05	.0012	1.0
Hydrogen chloride	18.8	.047	.0012	1.9
Carbon monoxide	15.6	.045	.00034	0.5
Ethylene	14.4	.099	.00027	0.16
Sulfur hexafluoride	1.47	.16	.00027	0.01
Dichloromethane	96	.11	.00027	1.0

TABLE II. Quantitative Evaluation of SF₆-Spiked Stack Gas

Date	Time	Concentration, ppm		Percent Deviation
		Detected	Predicted	
8/30	18:39	2.80	2.73	2.6
8/30	18:48	2.84	2.73	4.0
8/30	18:59	2.86	2.73	4.8
8/30	21:37	2.78	2.53	9.9
8/30	21:47	2.78	2.46	13.0
8/31	17:24	2.34	2.23	4.9
8/31	17:44	2.13	2.19	2.7
8/31	17:50	2.21	2.12	4.2
8/31	17:57	2.22	2.11	5.2

TABLE III. Results of the Matrix Spike Test

Analyte	Date	Time	File	Concentration (ppm)		Percent Error
				Calculated	Detected	
Benzene	8/29	12:23	CH9121	14.1	16.8	19.1
	8/29	12:33	CH9122	14.7	17.5	19.0
	8/29	12:43	CH9123	15.4	17.2	11.7
	8/31	19:23	CH1171	13.0	12.6	3.1
	8/31	19:39	CH1172	13.0	12.6	3.1
TCE	8/29	12:23	CH9121	6.4	6.0	6.3
	8/29	12:33	CH9122	6.6	6.4	3.0
	8/29	12:43	CH9123	6.9	6.7	2.9
	8/31	19:23	CH1171	5.8	5.4	6.9
	8/31	19:39	CH1172	5.8	5.4	6.9
Methane	8/29	12:23	CH9121	14.5	20.2	39
	8/29	12:33	CH9122	14.5	21.7	50
	8/29	12:43	CH9123	14.4	22.2	54
	8/31	19:23	CH1171	16.8	17.5	4
	8/31	19:39	CH1172	16.8	17.3	3

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