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## COMPARISON OF PASSIVE-REMOTE AND CONVENTIONAL FOURIER TRANSFORM INFRARED SYSTEMS FOR CONTINUOUSLY MONITORING INCINERATOR EMISSIONS\*

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## COMPARISON OF PASSIVE-REMOTE AND CONVENTIONAL FOURIER TRANSFORM INFRARED SYSTEMS FOR CONTINUOUSLY MONITORING INCINERATOR EMISSION5\*

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### ABSTRACT

Significant improvements in detection technology are needed to comply with the requirements in the Clean Air Act of 1990, Title III, which requires the monitoring of air toxics. Fourier transform infrared (FTIR) spectroscopy can satisfy these requirements in two different modes. Conventional FTIR spectrometers can be installed on-stream so that a vapor stream enters an infrared cell for analysis. Other types of FTIR spectrometers can detect chemical plumes remotely, measure the natural emissions of the molecules in the plume. The samples do not come to the instrument, and the instrument has neither source nor reflector mirrors.

We will discuss the applications of FTIR spectroscopy for both conventional and passive-remote FTIR spectroscopy. Some applications of conventional FTIR include a continuous emission monitor for measuring incinerator emissions and determining indoor air quality. Passive-remote FTIR spectroscopy can be used to identify and track a chemical plume. It can also be used to detect fugitive emissions. Hence, it can be used as an independent means to assure compliance with environmental regulations in real-time. Because of the relatively simple instrumentation, passive-remote instruments can be helicopter- or vehiclemounted for mobile detection of plumes.

### INTRODUCTION

Fourier transform infrared (FTIR) spectroscopy is an excellent method to identify and quantify selected target analytes in a complex mixture without first separating the compounds. This capability is especially important because of the requirements of Federal regulations such as the Clean Air Act of 1990, which requires the monitoring of air toxics (Title III), the Resource Conservation and Recovery Act (RCRA), and the Toxic Substances Control Act (TSCA). This report describes applications of FTIR technology to continuously monitor incinerator emissions and to detect and track fugitive emissions.

Currently, there is no on-stream method to determine the destruction of hazardous substances such as benzene or to continuously monitor for hazardous products of incomplete combustion (PICs) in incinerator exhaust emissions. An on-stream continuous emission monitor (CEM) that can differentiate species in the ppm and ppb range and can calculate the destruction and removal efficiency

(DRE) would demonstrate the safety and reliability of incinerators. This information can be used to address reasonable public concern about incinerator safety and aid in the permitting process.

In addition to a CEM, another technique is needed to independently determine if a facility is in compliance. Fumes of suspected toxic substances cannot be traced to their source. Passive-remote FTIR technology offers the opportunity to identify and track plumes to their source and to independently monitor a facility to determine if it is in compliance. The equipment is completely portable and can operate in a moving vehicle.

We will discuss advances in the use of conventional FTIR as an on-stream CEM for an incinerator and passive-remote FTIR capabilities to detect and quantify a plume.

### BACKGROUND

FTIR spectroscopy utilizes the two atmospheric windows in the mid-infrared range to detect target analytes. These windows do not contain carbon dioxide (CO<sub>2</sub>) or water absorption bands. Most organic compounds absorb strongly in one or both of these regions. The 3200- to  $2500-cm^{-1}$  region contains primarily carbon-to-hydrogen bond information and is used to detect aromatic hydrocarbons such as benzene.

There is a distinctive absorption pattern in the second atmospheric window that is commonly called the "fingerprint" region and is observed in the 1300 co 800-cm<sup>-1</sup> region. Most organics have distinct absorbances in this region. The molar absorptivity of organic compounds is normally high, resulting in sensitivity at a very low level (ppb-ppm).

In conventional FTIR, the sample must be brought to the instrument. Data are obtained as transmission (or converted to absorbance) spectra. Quantitation is possible because the infrared source provides a constant, high-temperature background and the sample chamber is at a constant temperature. The large difference in temperature between the sample and the source maximizes absorbance of infrared energy.

Remote systems lack the sample cell, eliminating the need to bring the sample to the spectrometer. There are two types: active-remote and passiveremote systems. Active-remote instruments, unlike conventional FTIR spectrometers, are not self-contained. The infrared source is aimed at a pickoff mirror, which is positioned so that the sampling area is located between it and the detector of the FTIR. The infrared energy, once emitted from the source, passes through interferometer and then the area being sampled until it reaches the pick-off mirror, where it is reflected back through the sampling area and enters the detector. The source, interferometer, and detector are not necessarily housed in the same container.

Passive-remote systems are very different from both conventional and active-remote FTIR systems. A passive-remote FTIR spectrometer consists of entry optics, an interferometer, and a detector. The entire system is contained in a small package weighing as little as 16 lb. The spectrometer uses ambient energy as its source. The sampling area can be anywhere within the field-ofview of the spectrometer. The spectrometer receives emission spectra. The amplitude of the emission is a function of concentration and the temperature of the background and the plume. Passive-remote systems are ideal for detecting a solvent plume and tracing it to its origin, but the technology is much more difficult to develop and field. The primary obstacle is the constantly changing background. A moving spectrometer would encounter sky, grass, trees, asphalt, cement, and building materials as backgrounds. Each background has its own temperature and would emit a different background infrared envelope. The infrared blackbody window even varies when a sky background is used, due to the absorption of infrared energy by atmospheric water, which is emitted in pulses.

Most passive-remote instruments try to cope with the variable background by collecting a background spectrum with no analyte present and subtracting this background from the sample. Similar results can be obtained by determining a ratio of the spectrum with analyte to the background. These are often poor alternatives because it may not be possible to obtain a comparable background. Also, data analysis will have to follow data collection so that appropriate background and sample spectra can be determined.

More sophisticated approaches include using internal blackbodies, which provide a reference point and time domain analysis, which digitally filters the interferogram to remove the background before Fourier processing to the spectral domain.<sup>1</sup>

### FTIR AS A CONTINUOUS EMISSION MONITOR

A laboratory incinerator<sup>2</sup> was used on a liquid toluene stream to evaluate FTIR spectroscopy as a CEM. Toluene is relatively nontoxic. When incompletely combusted, it produces PICs such as benzene and methane. The FTIR software can detect methane, benzene, toluene, carbon monoxide, and chlorobenzene. Chlorobenzene was inserted into the library because its spectral absorbance overlaps the absorbances of benzene and toluene. It cannot be present in a toluene burn; it's detection would indicate a serious problem with false positives. The infrared spectra of methane, toluene, chlorobenzene, and benzene are shown in Figure 1.

The incinerator was allowed to operate at equilibrium, approximately  $1100^{\circ}$ C, and then cooled to  $920^{\circ}$ C while sampling continued. During cooling, soot began to form. Figure 2 shows the infrared spectrum of a sample collected when the incinerator was at  $1100^{\circ}$ C (equilibrium), after cooling was initiated (initial modification), and after the temperature had decreased to  $920^{\circ}$ C. The equilibrium spectrum shows only waterbands in the aromatic region. Once cooling began, the baseline gently rose in the aromatic region ( $3100-3000 \text{ cm}^{-1}$ ). After cooling had progressed, the aromatic region and the prominent methane absorption at  $3017 \text{ cm}^{-1}$  occurred and are clearly seen.

A summary of the results is given in Table 1. Cases I and II of Table 1 show the effect of decreasing the temperature of the incinerator while slightly increasing residence time and excess air. Cases IIIA, IIIB, and IIIC were from the same experimental run, with decreasing temperature. At the initial temperature of 1100°C in Case IIIA, combustion was complete. The temperature was allowed to decrease and another sample was collected (Case IIIB - initial modification). After the temperature stabilized at 920°C, a third set of data was collected (Case III). Case IV is similar to Case IIIA, but the residence time was reduced by 41%. Temperature reduction was attained by allowing the wall heat loss to increase.

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# Figure 2. Spectral Comparison of Toluene Incineration during Temperature Modification

# Table 1. Data for Destruction of Toluene

	Case I	Case II	ATTI seaD	Case XIIB	Case IIIC	Case IV	
Incinerator Temperature ("C)	1005	900	1100	Decreasing from 1100	920	1100	
Residence Time (sec)	2.17	1.27	1.06	1.22	1.22	0.63	
Air Ratio	1.37	1.42	2.55	1.57	1.57	1.62	
Toluene Feed Rate (g/min)	0,107	0.105	0.063	0.058	0-058	0.063	
Air Rate (cm <sup>3</sup> /m)	7332	7332	7524	7524	7524	12631	
Toluane Concentration in the Inlet (ppm $\times 10^{-3}$ )	3.58	3.09	2.05	1.89	1.89	1.22	
Hethane Concentration in the Inlet (ppm x 10 <sup>-4</sup> )	5.59	5.60	5.47	5.47	5.47	5.65	
Concentration in the Exhaust (ppm $\approx 10^{-1}$ )							
Methane	0 8	815 ± 303	Ö	351 ± 207	5580 ± 1200	ND	
Toluene	۰.	28 🕿 13 -	o	o	161 ± 27	O	
Benzene	ND* 1	34 ± 23	ND	412 ± 9.8	379 ± 247	13 ± 9	
Carbon Honoxide	0 4	54 ± 34	o	175 ± 18	1429 ± 129	158 ± 18	
Chlorobenzena	ND	ND	ND	ND	ND	ND	
Soot Observed	O Excess		o	×ces#	########	Some	
Toluene DRE (X)	>99.94	92.4	>99.95	>99.9	91.5	>99.8	

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In each run, the FTIR worked perfectly as a CEM. These incineratoroperating parameters were varied while continuously monitoring for methane, toluene, benzene, and carbon monoxide emissions. The effects on emissions of the varying conditions of Cases I to IV were easily followed by the CEM.

As reported in Table 1, the concentrations of analytes were below detection level when the incinerator was at 1100°C (Cases I and IIIA). During cooling (Cases IIIB and IIIC), both methane and benzene were formed. As the incinerator continued to cool, toluene also appeared. The concentration of CO also correspondingly increased.

The error values in Table 1, two standard deviations, were relatively large. However, the concentrations of the methane, benzene, and CO showed a systematic increase. Further work needs to be performed to lower the error in the quantitative values.

Although a pure toluene feed was used, the first products formed during incomplete combustion were benzene and methane. Both of these compounds are extremely stable and their formation is expected. If no burn had occurred, the toluene concentration would have been 100,000 ppm. Hence, the DRE for toluene during the upset was 99.94%, which is marginally below the level set for nonhazardous wastes. The value for DRE has less meaning when it can be demonstrated that benzene and methane are formed and emitted in significantly higher concentration.

### PASSIVE-REMOTE DETECTION

A passive-remote FTIR sensor must be able to qualitatively identify Title III, air toxics in the environment. The instrumentation must first detect the air toxic with a low probability of false positives or false negatives. Hence, it must be able to detect the air toxic in an environment that would contain other components with overlapping absorbances. It must also function in a continually changing background so that it can track a plume to its source, which requires a field test to demonstrate. The field test described was performed at a Drug Enforcement Administration site. Pure chemicals were released in the environment to simulate the concentration found at illegal drug laboratories. A ground-based XM21 spectrometer was used to monitor the plumes.

The ability to identify a target analyte in a mixture containing components that have overlapping absorbances can be tested in the laboratory. A remotepassive FTIR was constructed from a conventional Nicolet 6000 FTIR. Mixtures containing totally overlapping absorbances were quantitatively released in front of the instrument. The ability to detect single components in the presence of completely overlapping spectral bands was determined using partial least squares (PLS) and classical least squares (CLS) software.

### PLUME DETECTION

Plume detection was performed using an XM21 and a modified Midac interferometer provided by the U.S. Army Chemical Research, Development, and Engineering Center (CRDEC). The XM21 is ground-based, while the modified Midac was flown in a helicopter at altitudes of 200-500 ft above ground level and speeds of 80-120 knots. The XM21 was designed to operate under battlefield conditions. Its internal blackbody helps to stabilize the response, and the MCT-A detector is optimized for the atmospheric window of  $1200-900 \text{ cm}^{-1}$ . The optical system is simple to eliminate alignment problems.

The XM21 was able to detect the plumes emitted by the simulated methamphetamine laboratory, which consisted of a Freon-11 release from a window in a trailer. The Freon-11 was also detected with a large signal-to-noise (S/N) ratio. The spectrum of a Freon-11 standard and the actual spectrum obtained during the release are shown in Figure 3.

The cocaine laboratory simulation consisted of an ethyl ether release. The ethyl ether releases were performed by pouring each liquid into 8-ft-long trays in open sheds and evaporating the liquid with the use of heat lamps. The XM21 was able to detect both this solvent in the open area and in the canopy. The ethyl ether reference and actual release spectrum are shown in Figure 4. Real-time data analysis was performed with a modified Midac passive-remote FTIR spectrometer. The spectrometer had a video camera coaxially mounted. A video display board was mounted in the personal computer to overlay data collection on video tape.

This spectrometer is capable of real-time data analysis using the advanced signal processing algorithm described above. Approximately 50,000-100,000 training spectra are required to develop a digital filter for a single component. To date, a digital filter has been developed for only one component, SF<sub>6</sub>. The Midac performed real-time analysis for SF<sub>6</sub>. For the other releases (i.e., Freon-11, acetone, acetic acid, and ether), data were collected. The data for these solvents can be evaluated when digital filters for these components are developed.

The modified Midac spectrometer software determines a response calculated from the interferogram-based real-time signal processing algorithm. The algorithm response was plotted as a function of time. The algorithm response for the helicopter-mounted spectrometer before it reached the target area (background data) is shown in Figure 5; the response appears as a straight line below zero arbitrary units (AU). The zero line on the plct is the trigger threshold at which detection begins. As the concentration of the detected analyte increases, the amplitude of the response also increases.

When the helicopter-mounted FTIR spectrometer passed over the target area, it detected analyte due to the increase of the amplitude of the response. After the helicopter passed over the plume, it circled around and again passed over the plume at a different altitude. For these releases, the helicopter altitude varied from 200-500 ft. Each time the helicopter passed over the plume, the FTIR spectrometer detected analyte. The results of these sequential passes over the target area can be seen in Figure 6. The response in AU is plotted against interferogram number, which correlates with time (4 scans per sec). The large increase in response each time analyte was detected is easily observed from the figure.

### QUANTITATION

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The ability of an FTIR spectrometer to discriminate between a target analyte and an environmental impurity that may have an overlapping spectral

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absorbance is critical for FTIR to be used for compliance assurance. We have previously reported<sup>3</sup> two systems consisting of a two-component mixture consisting of isopropanol (IPROH) and dimethyl methyl phosphonate (DMMP) and a three-component mixture consisting of methanol (MEOH), DMMP, and IPROH. These components all have overlapping peak absorbances in the atmospheric window. Passive-remote FTIR was able to detect each of the analytes in these mixtures. For this paper, a MEOH-DEM-DMMP mixture was analyzed. The data are for pure methanol, pure DEM, pure DMMP, and mixtures of the three-components in the ratios 1:1:1, 1:2:3, and 3:2:1. The spectral data for pure MEOH, pure DEM, pure DMMP, and their mixtures in the volume ratios of 1:1:1, 1:2:3, and 3:2:1 are shown in Figures 7 through 12, respectively.

PLS and CLS data analysis were performed on this data set. The results of these analyses are shown in Table 2. PLS clearly outperformed CLS. If the lowest concentration of MEOH is removed from the data set, MEOH quantitation was within 10% using PLS. CLS results were much more variable with most values deviating from the release concentration by 40-50%. DEM and DMMP PLS results were closer to the 20% values obtained with the MEOH-DMMP-IPROH results obtained above. CLS results were significantly worse for DEM. However, DMMP results using CLS were only slightly less accurate than those obtained using PLS. CLS did have a false negative at the lowest DMMP concentration.

#### CONCLUSION

Our tests with a laboratory incinerator demonstrate that FTIR spectroscopy can be used to monitor incinerator emissions. We confirmed qualitative identification of methane, toluene, and benzene. Chlorobenzene, used to test for false positives, was not detected.

This initial work casts doubt on the relevance of determining only the DRE for waste destruction and risk assessment. In the incineration of toluene, as the incinerator changed conditions, intermediate products that are more toxic than toluene were formed. Future CEMs will need to monitor these toxic intermediate products as well. When the incinerator is not performing efficiently, even though the DRE exceeds regulatory limits, the emissions of intermediary products must still be counted in the risk assessment. The concentration of these products can be monitored with FTIR.

Passive-remote FTIR spectrometers have been successfully field tested and have demonstrated the capability of identifying a chemical plume. The instrumentation has also been laboratory tested to show that a single component can be identified in a mixture containing completely overlapping absorbances. FTIR technology, therefore, has demonstrated the potential for use in as an independent method for compliance assurance under Title III, Air Toxics of the Clean Air Act of 1990. The light weight and portability of the instrumentation is a significant advantage.

Work must continue in developing a large spectral library data base consisting of analytes and backgrounds. Appropriate QA/QC procedures must also be developed to ultimately obtain certification by the Environmental Protection Agency (EPA). REFERENCES

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Figure 11. Passive-remote IR spectra of a mixture of MEOH-DEM-DMMP with a volume ratio of 1:2:3 in the spectral region 1200-940 cm<sup>-1</sup> Figure 12. Passive-remote IR spectra of a mixture of MEOH-DEM-DMMP with a volume ratio of 2:1:3 in the spectral region 1200-940 cm<sup>-1</sup>

# Table 2. Comparison of PLS and CLS Data Analyses for MEOH-DEM-DMMP Mixtures

SEQUENCE MEOH-DEN-DHIMP							DUMP PLOW RATE									
			METHAN	VC-PLUN VC-n		vere		PISANA	SIS	CLS MUL	SIS		PLS MULY	58	as well's	35
	FILE NAME	ACTUAL	CALC	* DEY	CALC	S DEY	ACTUAL	CALC *	DEY	CALC *	DEY	ACTUAL	CALC 1	DE.	CALC *	027
N PC USON	40005	ñ (0	011	411	0.23	21.5	0	· a co		0.22		0	-0.02		0.03	
PURE MECH	12000	8.10	0.30	<u>.</u>	0 37	27.5	Ó	-0.01		0.08		0	-002		-0.01	
	AZONT -	0.23	0.13	142	0.37	-2.9	٥	-0.06		-0.26		0	-032		-0.06	
	AGO U	0.30	0.44	.00	0.55	.179	0	-0.05		-0.39		0	002		-0.07	
	A3012 *	0.07	0.00	-11.5	0.17	-10.3	Ď	-0.03		-0.21		٥	430		-0.05	
	A301.4	0.31	1 10	2	1 2/		n	0.02		0.08		0	-0.01		0.02	
	A3016	1.4	1,40	u)	2.57	105	ñ	014		0.64		0	022		Q14	
	A3018	1.9	217	10.9	CL1	14.9	•									
pure dem	A3021	. o	-0.07		-0,08		0.19	0.22	16.8	0.19	1.6	0	-001		100-	
	A3C23 *	0	-0.03		-210		029	022	11.0	0.28	-4.1	0	41.01		-0.01	
	A3C26	٥	-0.04		-0.09		0.38	0.45	189	0.42	100	0	000			
	A3027 *	0	201		-0.09		û 67	0.67	-0.6	0.56	-169	0	-0.02		-0.04	
	A3029	0	012		-0.05		0.97	0.67	-ગા.ા	0.45	-63.3	u U	-0.01		~~~/	
									157		17.2					
			0.25		0::0		0	0.03		û 22		0.19	0.14	-284	Q.19	-26
PURE DWMP	A3032				0.00		0	-0.03		Q15		0.29	025	-131	0.30	2.8
	A30.94 •	ر ب	0.01		0.04		0	-0.00		0.23		0.38	0.40	50	0.48	21.1
	Allia		-001				0	-0.02		009		0.57	0.78	157	0.82	22.2
	A3C30		-000		-0.01		ē	0.04		0.04		Q.97	1.10	136	1.12	155
	13040		, -u 		-0.06		Ō	0.04		-010		14	1.22	-5.8	1.31	-6.4
	A3042 *				-0.00	,	0	-0.00		-0.41		1.9	1.39	-26.6	1.31	-31.1
	1344						-							155		14.5
usou (	A3047	<b>a</b> 1	3 0.49	276.9	۵7:	461.5	013	0.25	96.9	Q.57	341.5	013	014	85	0.21	67.7
DENI	A3C49	0.2	2 0.24	109	0.44	1216	0.22	0.28	20	0.55	1632	0.22	200	11.4	0.29	212
DULP!	A3061	0.3	2 0.3	41	0.50	57.2	0.32	0.37	15.6	0.55	102.2	. 0.32	0.36	134	0.42	31.2
	A3053	• 0.4	7 04	4 .7.0	0.64	5 419	0.47	Q.41	-138	0.70	49.8	· 0.47	0.45	-4.9	0.50	6.6
	A3065	0.6	3 08	4 1.9 60.2	07	8 22.2 141.3	0.63	0.34	-45.4 39.5	1 0.50 5	-20 8 133 5	063	0.47	126	1149	302
11504-1	1305R	DO	-00	4 NA	-00	3 NA	0.13	0.05	-81	0 09	-30.8	0.19	-405	-128.4	-0.02	-110.5
DEU-2	13060	• 01	1 01	1 0.9	-00	0 -101.8	0.22	023	6.	5 013	-39.1	0.34	0.22	-69	0.29	-14.7
DL# 2	12062	0.	10 01	0 -37.6	0.0	3 -83.1	0.32	0.30	-6.9	9 0.23	-291	0.48	0.49	23	0.4/	-1.3
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	A3066	0	22 03	2 0.3	0.2	5 .211	0.63	0.49	-22	6 0.40	-36.7	098	5 0.57	20	) 0.94	-0.9
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NECH:3	A3069	۵	19 03	21 12/	3 01	4 .27.5	0.05	0.06	i 1.	6 -0Z	-442.9	0.1	a 12	-7.3	0.05	-58.5
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