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SUMMARY REPORT

Evaluation of the Fourier Transform Infrared (FTIR) Spectrophotometer for Analysis of Trichloroethylene (TCE) in the Presence of Freon®-113 in Carbon Disulfide Eluates of Charcoal Air Sampling Tubes*

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Results obtained using Fourier transform infrared spectrophotometry (FTIR) for the analysis of samples of carbon disulfide (CS_2) eluates containing trichloroethylene (TCE) and Freon® from charcoal air sampling tubes were evaluated by comparison with results obtained when using gas chromatography (GC). The FTIR yielded accurate results without regard to the presence of Freon.

Recent publications[1,2] have described the use of Fourier transform infrared spectrophotometry (FTIR) for the direct analysis of gas and vapor contaminants of workplace air. Many methods have been established for the collection and determination of trichloroethylene (TCE).[3] The National Institute for Occupational Safety and Health (NIOSH) recommends the collection of TCE using a charcoal tube—with subsequent elution of the TCE using carbon disulfide (CS_2)—and analysis of the CS_2 using gas chromatography (GC). The use of GC provides a convenient, tested, quantitative method for the analysis of TCE solutions and many other solvents.[3] In 1982, an experiment was reported wherein an infrared (IR) spectrometer was used to analyze CS_2 eluates of passive dosimeters.[4]

Theoretically, the 1982 experiment was a success, but no data were reported that substantiated the method. Other methods have been reported where IR spectrometers were used to analyze workplace air samples,[5,6] such as thermally desorbed sampling tubes.[7] Theoretically, IR methods should allow the same type of qualitative and quantitative analysis of CS_2 eluates as do GC methods.

MATERIALS AND METHODS

Samples were collected using 100 mg/50 mg SKC charcoal tubes at the same times and places as were the 20-L Tedlar® bags reported in the previous paper.[2] SKC model 222-3,4 sampling pumps were attached via flexible tubing to the charcoal tubes and bags used according to NIOSH Method 1022.[3] Charcoal tube samples were taken by the NIOSH Health Hazard Evaluation Team and were shipped directly, under refrigeration, to the NIOSH laboratory in Cincinnati where they were analyzed. Analysis of the samples for Freon® was performed incidentally to the analysis for TCE by NIOSH Method 1022. After GC analysis at NIOSH-Cincinnati, CS_2 sample vials were capped with new septa and sent to the University of Michigan for GC and IR analysis.

Prior to injection of each sample, the gas inlet/dilution manifold[2] was evacuated to <1 Torr. For each analysis, 0.1–0.3 mL of CS_2 solution was injected into a 500-mL Pyrex® glass chamber equipped with a septum inlet and attached to the manifold leading to the FTIR gas cell. The total volume of the inlet and gas cell system was 8.61 L. High purity nitrogen gas was used to purge the glass inlet and carry the vapor into the gas cells.

Samples taken for GC analysis were removed from the inlet manifold with a 1.0-mL gas-tight syringe. All analysis procedures were conducted in a manner identical to that reported in the previous paper.[2]

RESULTS AND DISCUSSION

The IR spectrum of CS_2 vapor has several strong peaks. In the fingerprint region of the spectrum (between 1400–650 cm^{-1}), peaks are present for most organic compounds. CS_2 has one sharp peak in this region. However, the concentration of analytes is trivial compared to that of CS_2. Therefore, it is important to select appropriate regions or peaks for the analysis of the target analyte(s) in order to avoid overlap with the very strong solvent peak and attain acceptable accuracy for quantitative analysis (Figure 1).

TCE has one peak that overlaps with the CS_2 peak at 849.5 cm^{-1} and a second peak at 944.8 cm^{-1}, that is free from the interference from the CS_2 peak. Thus, this latter peak was chosen for use in quantitative analysis of CS_2.

Freon-113, which overlaps with the TCE peak at 849.5 cm^{-1}, was found in some samples. However, using the least squares fit

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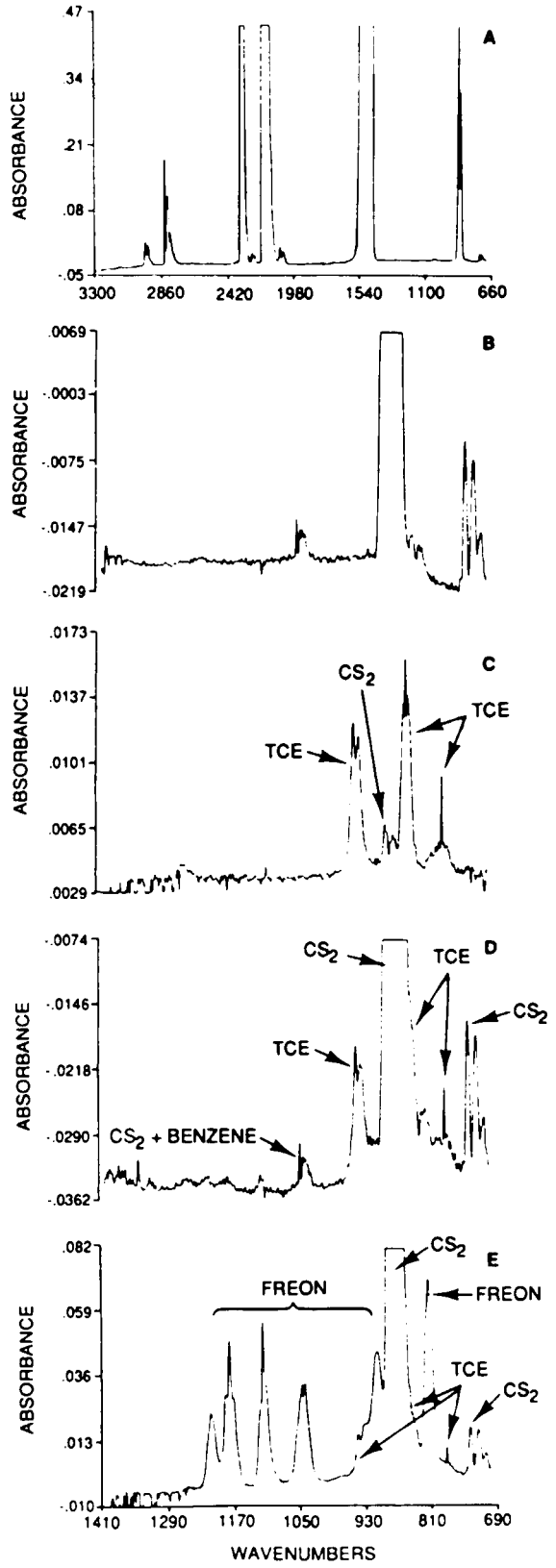

Figure 1—Infrared spectra of CS_2, and samples containing TCE and Freon®-113. In each case, note the scale of the Y-axis. Tracing (A) Full spectrum of CS_2 (300 μL injection); (B) CS_2 fingerprint region (300 μL injection); (C) Sample 11 (5.0 μL injection, 173 ppm TCE); (D) Sample 23 (300 μL injection, 2.5 ppm TCE); (E) Sample 6 (300 μL injection, 1.95 ppm TCE and 6.5 ppm Freon-113).

(LSF) program,[2] this kind of interference does not affect the accuracy of results. Results are summarized in Table I. The results obtained when using the FTIR method are not significantly different from those obtained with the GC methods.[8] According to the GC analysis, Samples 6 and 7 are the only two samples that contain Freon-113 at concentrations higher than 1 ppm. No significant difference between these two methods was shown for these two samples.

When considering the use of FTIR rather than GC for the analysis of CS_2 eluates of charcoal tubes, two additional factors should be discussed. First, an IR spectrum of all the airborne contaminants eluted from the charcoal is obtained as a natural result of this procedure. From the spectrum, qualitative analysis of the eluate may be accomplished. Thus, if contaminants are present in the air that were not previously suspected, they may, in many circumstances, be identified and quantitated. The qualitative analysis capability was not explored in this study. Secondly, this procedure is very rapid. The entire time per sample when using a spectral resolution of 2 cm^{-1}, regardless of the number of target analytes present, is <4 min, including injection, pump down, and data analysis.

TABLE I
Results of Analysis of Trichloroethylene and Freon®-113 in the CS_2 Eluate from Charcoal Air Sampling Tubes (ppm v/v)

Sample Number	Trichloroethylene		Freon-113
	GC	FTIR	GC
1	0.59	0.69	(0.09)[A]
2	0.62	0.89	(0.11)
3	0.89	0.81	(0.10)
4	0.84	0.86	ND[B]
5	0.41	0.62	ND
6	2.03	1.95	6.5
7	1.30	1.33	1.3
8	3.31	2.83	ND
9	3.55	3.24	ND
10	3.04	2.24	ND
11	165.3	147.0	ND
12	168.5	151.0	0.40
13	0.80	0.67	0.45
14	1.02	0.62	ND
15	0.62	0.71	ND
16	1.18	1.59	0.46
17	2.63	2.25	0.85
18	2.80	3.35	0.68
19	1.49	1.46	(0.24)
20	0.55	0.80	(0.11)
21	2.16	0.87	ND
22	2.79	2.61	ND
23	4.51	4.66	(0.11)
24	1.10	1.13	ND

[A]Values in parentheses are between the laboratory limit of detection (LOD) and limit of quantitation (LOQ) and should be considered semi-quantitative data. The LOD and LOQ for the analysis of Freon-113 were 0.02 and 0.06 mg/sample, respectively.

[B]ND = not detected, less than the LOD for Freon-113.

With some additional method development effort and the addition of an autoinjector, the time required for this procedure could be significantly reduced. For CS_2 with many target analytes, this may be significantly faster than can be accomplished by GC. For simple chromatographic analyses, GC will be as fast, if not faster than, FTIR.

CONCLUSIONS

The FTIR has been shown to be capable of determining TCE in the CS_2 eluates of charcoal tubes, with results that are comparable to those results obtained using gas chromatography.

Additional work will be needed to investigate the questions of the speed and cost of this method, and the utility of this technique for the identification of unknown constituents in the air samples.

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