American Industrial Hygiene Association Journal

ISSN: 0002-8894 (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/aiha20

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To cite this article: STEVEN P. LEVINE , MARK A. PUSKAR , CHARLES L. GERACI , ARDITH A. GROTE & MICHELE BOLYARD (1985) Fourier Transform Infra-Red Spectroscopy Applied to Hazardous Waste: I - Preliminary Test of Material Analysis for Improvement of Personal Protection Strategies, American Industrial Hygiene Association Journal, 46:4, 181-186, DOI: 10.1080/15298668591394626

To link to this article: https://doi.org/10.1080/15298668591394626

Am. Ind. Hyg. Assoc. J. 46(4):181-186 (1985)

Fourier Transform Infra-Red Spectroscopy Applied to Hazardous Waste: I - Preliminary Test of Material Analysis for Improvement of Personal Protection Strategies

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Many chemicals, when mixed, can produce potentially hazardous effects which are harmful to human health and to the environment such as heat, pressure, fire, explosion, violent reaction, and toxic dusts, mists, fumes, and/or gases. Waste chemicals handled on a remedial action site are analyzed by simple chemical methods for compatibility. Because it is difficult to base worker and community protection programs on this limited knowledge of material content, extreme precautions must be taken to ensure safety to all working on or living near hazardous waste remedial action sites. The approach used in this study involved the application of Fourier transform infra-red spectroscopy (FTIR) to the analysis of samples taken from the Chem-Dyne remedial action hazardous waste site. The data generated are compared to the results obtained using compatibility and GC-MS analysis procedures. Illustrations are given of improved personal protection strategies, based on drum material composition obtainable by FTIR. The requirements for further testing are defined.

Introduction

The disposal of waste chemicals in drums has been a widespread practice throughout the United States. One-quarter of all abandoned waste sites have major drum-related problems, including handling, integrity, characterizing, and disposal.(1,2) A major effort in any cleanup is the bulk recontainerization of hazardous materials located in drums, labpacks, tanks, and holding ponds.(3)

Many hazardous chemicals, when mixed, can produce effects which include the sudden generation of pressure, fire or explosion, violent reaction, and toxic dusts, mists, fumes, and/or gases. Chemical wastes handled on-site must be tested to determine if they are incompatible. If so they must be separated.(4)

Waste chemicals handled on a remedial action site are analyzed by basic qualitative methods for compatibility. These compatibility testing methods include, for example, the addition of the sample to water and to hexane. The result of this test is used to categorize the sample into classes: organic non-halogenated, organic halogenated, and aqueous or polar organic. The only complete assay routinely performed is for PCBs in which a profile is obtained via gas chromatography (GC) with a Hall electrolytic conductivity detector operated in the halogen mode.

Complete characterization of each individual drum of waste by standard laboratory testing procedures [such as GC — mass spectrometry (MS)] followed by overpacking and subsequent transportation to an approved disposal facility is far too costly and time consuming. However, compatibility testing followed by bulking compatible drum contents into large containers prior to transportation to an approved disposal facility proves to be the most cost and time effective method of handling the barreled waste.

Until now, the goals of compatibility testing schemes were to generate enough knowledge of the characteristics of the waste to:

1) identify and separate potentially hazardous wastes on-site during the remedial action, thus limiting the possibility of accidental mixing of incompatible wastes.
2) identify compatible drums, so they may be bulked on-site, prior to disposal, thus saving considerable transportation and disposal costs.
3) complete the shipping manifest in a manner which will be acceptable to the site manager, state or federal agency supervising the cleanup, transporter, and receiver of the waste.

The majority of the analytical methods currently employed involves a group of simple chemical tests (*e.g.* water solubility, pH, flammability) following a flowchart scheme which ultimately classifies the material into general categories.(3,5-12)

One of the major compatibility classifications is the organic liquids. This includes the categories: polar organics, halogenated organics, and non-halogenated organics. Not only are the organic categories the most complex from an analytical point of view, but the organic category is also the most important from a toxicological standpoint.

For example, when a drum is categorized as nonhalogenated organic, no other data is generated to determine if the drum contains a slightly toxic chemical: toluene (TLV 100 ppm, 8.7 g/kg LD50), a moderately toxic compound: benzaldehyde (TLV None, 1 g/kg LD50), a highly toxic compound: parathion (TLV 0.01 mg/m^3, 2 mg/kg LD50), or a mixture of all three. A detailed knowledge of drum material composition would permit the tailoring of personal protection strategies to those specific materials.(13-16)

This additional knowledge of drum composition might also help protect the workers at the waste disposal facility to which the waste is sent. These workers, who are protected by OSHA standards, may or may not wear proper protective equipment. A method that could assist in the rapid screening of incoming wastes and check drum and tank contents versus whatever information is shown on the manifest, would help to provide information necessary for the definition of worker protection strategies at waste disposal sites.

At present, safety strategies at hazardous waste remedial action sites are centered around two criteria:(5,8,11) 1) the total hydrocarbon (THC) vapor concentration in the air as determined by a portable non-specific vapor monitor [usually a photoionization detector-equipped monitor (PID)]. 2) the possibility that conditions immediately dangerous to life and health (IDLH) may occur.

Both of these criteria are very useful but are subjective since the PID is non-specific and variable in response, and the conditions on the site may be subject to uncontrolled variables. The most often used "Levels of Protection" on a site are called Levels B and C. The protective ensembles defined by these levels include self-contained breathing apparatus (SCBA) in Level B or a full face respirator in Level C.(5,6,8) The protective garment utilized is not specified in the guidelines, but is usually chosen to prevent skin contact with whatever chemical being handled poses the greatest risk. In practice, Level B may be chosen simply because so little information is available about the composition of the drums that are being handled. Because of this, it is often assumed that conditions IDLH have a significant probability of occurring. The wearing of Level B ensembles, or even of Level C ensembles with overly protective garments, may increase the possibility of heat stress and will also reduce productivity.(15,16)

The test reported in this paper addresses these shortcomings in the present approach to personal protection at hazardous waste sites through an attempt to improve the knowledge of the composition of the drum materials. The analytical tool chosen for this study was Fourier transform infra-red spectroscopy (FTIR). Samples were obtained from drums at the Chem-Dyne hazardous waste remedial action site and subject to compatibility, FTIR, and GC-MS analysis. Illustrations are presented of potential personal protection strategies that may be employed given the additional information obtained through FTIR analysis.

Materials and Methods

Samples

A preliminary test was performed using samples of uncharacterized hazardous drum waste. Samples were aliquots of archived samples from the sample storage shed at the Chem-Dyne hazardous waste remedial action site, Hamilton, Ohio. All drum samples were supplied along with the results of compatibility tests. A list of the samples and their compatibility test results is given in Table I. One tank sample was also taken for FTIR-attenuated total reflectance (ATR)(17,18) analysis. This sample had been previously analyzed by GC-MS. The spectra obtained using the FTIR-ATR technique have been subject to an off-line computerized and manual spectral search since that capability was not present in the FTIR used in this study. The results of the compatibility and FTIR-ATR assays were used as guidance in the design of the protocol for obtaining corroborative assays via GC-MS.

The viscosities of drum and tank samples ranged from free-flowing liquids to gelatinous solids. A quality control of 1-chloronaphthalene was also analyzed and library searched. Spectra were analyzed off-line through the use of the Sadtler manual and computerized forward search routine.

Instrumentation and Methods

All samples were analyzed using an Analect fx-6200 FTIR spectrometer. An ATR accessory equipped with a germanium crystal was also used. The data system of this instrument was used only for instrument operation, since the capability to perform spectral searching was not present. The infrared spectra were searched using the Sadtler Spec-Finder routine, followed by manual interpretation of the results. This is a forward search routine designed for use with spectra of pure compounds or commercial products. Mass spectra were obtained using a Hewlett-Packard 5840/5982/5934 or a HP/VG 5840/MM 7070 system.

Results and Discussion

The ATR cell is an open flat crystal on which a drop of liquid or a glob of sludge could be placed, a spectrum acquired in a few seconds, the cell cleaned by wiping and rinsing and the next sample run. This aspect of the ATR test was a success. IR spectra were acquired in every case using the

TABLE I
Comparison of Summarized Results of Compatibility, FTIR-ATR, and GC-MS and GC-ECD Analyses Performed on Drum and Tank Samples

Sample Number	Compatibility Class	FTIR-ATR[A]	GC-MS, GC-ECD
T5	Organic/Hal	Hydrocarb oil,[B,C] subst aromatic[B]	Alkanes, chlorobenzenes, xylenes
D2167	Org/Hal/PCB	Glycol ethers[B,C]	Glycol ethers, PCB
D3509	Organic	Phthalates,[B] silicone alkyd[C]	Mixture including phthalates
D2911	Organic	Phenol	Not analyzed
D2335	Organic	Aqueous	Not analyzed
D2267	Organic	Phthalic acid derivative[B]	Styrene, xylene, toluene (minor phthalate)
S671	1-chloronaphthalene (QC known)	Correctly identified[C]	

[A] See Table II for complete FTIR-ATR and GC-MS information.

[B] Manual spectral identification following computer search.

[C] Computer search only.

T = tank, D = drum, S = quality control.

above method. This was true without regard to viscosity or apparent optical density or turbidity. Aqueous samples including acids were not in the scope of this initial study. All samples had been pre-screened by conventional compatibility testing. Samples had been previously analyzed for PCB content by GC with Hall electrolytic conductivity cell detection.

For drum compatibility testing and selecting worker and community protection, unequivocal identification of the contents of each drum is desirable. However, the compatibility testing protocols presently used do not provide that information.(5-12) These methods provide information largely limited, for organic compounds, to classifications such as "chlorinated" and "aqueous or polar organic". Therefore, the present methods would be significantly improved if classifications such as "aliphatic ketone, chlorinated aromatic, nitroaromatic phosphate, polyacrylate, aromatic and aliphatic solvents" could be obtained.

In order for that to be accomplished using FTIR methods, an infrared spectrum must be obtained for each sample, and this spectrum must be interpretable by the instrument data system. The data system supplied with the instrument used for this test was not designed for that task. Therefore, the plotted spectra were sent to Sadtler laboratories for manual interpretation and computer search. Details of the computer search are given elsewhere.(19,21)

In Table I, the GC-MS, compatibility and FTIR data are summarized and compared. Table II gives the detailed results of the GC-MS analyses.

Two limitations of this study should be noted at this point: 1. The spectral search routine employed is most effective with pure compounds and pure commercial products.(19-21) Thus, it is likely that complex mixtures will result in poorly identified components. Studies in progress will define the utility of on-line structural group identification, library search with Boolean logic, and reverse search algorithms.(19,21-23) 2. The ATR crystal employed had the optimal chemical and physical properties, but did not permit the direct observation of certain characteristic spectral features of organic chlorine compounds.(17,18,20) This choice of crystal material was made because the chemical reactivities of the samples and their effect on various crystal materials was unknown. Germanium is among the hardest and least reactive of the available ATR crystal materials, Studies in progress will compare the utility of ATR with various crystals and the use of thin film liquid cells.

The criteria that can be applied to any new analytical method used at a hazardous waste site to screen drums for composition must be assessed against existing compatibility testing procedures. The goals of the FTIR procedure are to allow the tailoring of disposal and personal protection strategies to specific materials based on a degree of knowledge of drum material composition beyond that which is attainable through compatibility testing.

Table I shows that the FTIR-ATR technique provided more information about sample composition than did compatibility testing. However, the data must be examined from the standpoint of the significance of the compounds and compound classes that were successfully detected, and those that were missed.

TABLE II
Results of the GC-MS Assay Performed on Samples from the Chem-Dyne Site

Sample Number	Description	Components by GC-MS Assay
T5	Black Liquid	C12-C20 alkanes (P) Dichlorobenzenes (M) Methyl ester (M) Xylenes and toluene (M) Dichloroethane (*) Chlorobenzene (*) Trichloroethane (*) Unident. chlorinateds (*)
D2167	Black Liquid	Ethylene glycol monoethyl ether (P) 2(2-methoxyethoxy) ethanol (M) Diethylene glycol monoethyl ether (*) Ethylene glycol monobutyl ether (*) Butanol (*)
D3509	Brown Sludge	Methylene chloride (M) Ethylene glycol monoethyl ether (M) Dioctyl phthalate (M) Unident. chlorinated (M) C9-C11 alkanes (M) Ethoxy ethyl acetate (*) Toluene (*)
D2267	Brown Gel	Styrene (M) Xylenes and toluene (M) Phthalic anhydride (*) Propyl acetate (*) Butyl isobutyrate (*)

Estimates of principle, major and minor components are order of magnitude. Physical principle (P), major (M) and minor (*).

In order to directly compare the principal species in those two classes (identified and missed), reference should be made to the following examples. In some cases, these compounds and compound classes are listed along with their PELs for comparison. It is acknowledged that PELs are not intended for use as relative indices of hazard or toxicity. However, the following examples are illustrative of the general types of compounds that may be encountered on a hazardous waste site.

Sample T5

Tables I and II show that the FTIR-ATR technique correctly identified the major components of the sample as a hydrocarbon oil, and identified the presence of an unknown substituted aromatic compound. GC-MS showed that the major components of the mixture were C12-C20 alkanes, xylenes and toluene, an unidentified methyl ester, and dichlorobenzene isomers. The FTIR-ATR missed the identification of dichlorobenzenes which is expected in this experiment for the reason given (#2) above.

The identification of a drum as containing waste oil and aromatic solvents would allow the use of minimal personal protective gear for the workers involved in the subsequent handling of that drum. Certainly, the situation that exists today would be improved; *i.e.*, at present, the drum is classified as "organic" and the workers must wear full Level B ensembles including self-contained breathing apparatus (SCBA). The open issue of the identification of chlorinated impurities in the hydrocarbon oil is significant, and will be addressed in a subsequent study through the use of an alternate ATR crystal.

Sample D2167

This sample, which is a black liquid, was classified as an organic, halogenated, PCB-containing liquid by compatibility tests. Gas chromatographic analysis with Hall detection run in conjunction with compatibility testing showed 160 ppm of Arochlor 1254. The FTIR classified the sample as ethylene glycol monoethyl (cellosolve), monobutyl and/or monoisobutyl ethers. No evidence of PCBs were found, although the use of the germanium ATR crystal would have reduced the probability of detection of PCBs.(18,24) GC-MS analysis showed the presence of cellosolve (ethoxy ethanol), 2(2-methoxyethoxy) ethanol and lesser amounts of carbitol (diethyleneglycol monoethyl ether), butyl cellosolve and butanol. No PCBs were found by GC-MS. GC-electron capture detection (ECD) analysis confirmed the presence and concentration of PCBs in this sample.

Personal protective measures recommended in the recent NIOSH Current Intelligence Bulletin(25) for glycol ethers include clothing that is impermeable to that class of compounds. The ACGIH TLVs and Notice of Intended Changes(26) bear a "skin" notation and a recommendation of a five ppm TWA. These changes are based on recent findings of reproductive and teratological effects in test animals. There is no recommended ceiling exposure value. Thus, a worker could be exposed to 150 ppm for 15 minutes and still be below the recommended TWA of five ppm.

Following present drum compatibility testing schemes, the material in this drum would be classified as "organic" or "PCB-contaminated organic", depending on the compatibility test scheme utilized. Since no further information is available, hazardous waste workers would dress in Level B (full protective clothing and SCBA) ensembles. However, the FTIR-ATR assay indicated that this drum contained a potentially hazardous compound, but that a Level C ensemble (employing a full-face respirator rather than SCBA) is more appropriate. This is due to the fact that neither glycol ethers nor PCBs are volatile. A total hydrocarbon (THC) air monitor calibrated for glycol ethers could be employed during the handling of drums containing this class of compounds.

The analysis of PCBs in this and other drum samples by FTIR-ATR will be explored in the next phase of this study. Preliminary work on this subject had been undertaken under an EPRI contract(24) by a group that reported that a dispersive instrument was insufficiently sensitive and lacked the requisite data handling capabilities for the infrared-ATR analysis of PCBs. At the time of that study, this group concluded that FTIR was not a practical field instrument. Advances in instrumentation have removed that obstacle to this approach, but the problem of sensitivity to PCBs in complex mixtures still remains. Knowledge of the PCB content of drums would permit the wearing of PCB-impermeable ensembles (Saranex-Tyvek suit and Viton gloves) when PCBs are present.(27,28)

Sample D3509

This sample was a brown/black sludgy liquid that was classified as an organic flammable liquid by compatibility testing. The FTIR assay indicated that it is a mixture of an ortho substituted aromatic compound, possibly a phthalate, with a silicone, possibly a silicone alkyd resin. GC-MS analysis shows methylene chloride, ethylene glycol monoethyl ether, dioctyl phthalate, an unidentified chlorinated compound, and C9-C11 alkanes. Minor components include ethoxyethyl acetate and toluene.

The FTIR technique identified the major phthalate component, but missed certain other components. The chlorinated component would have been missed because of the above mentioned optical properties of germanium. The compatibility testing procedure misclassified this sample as a nonhalogenated waste. GC-MS analysis missed the identification of the silicone alkyd resin because that class of compounds is not chromatographable.

The problem with tailoring a personal protection strategy based on FTIR-ATR and/or GC-MS analysis for a sample like this is that complete characterization of the sample may not be possible. Available data suggest that this sample is a mixture of resins with various solvents present at major and minor levels. Neither FTIR-ATR nor GC-MS gives the complete picture. In addition, the question of the unknown (trace level) chlorinated component is unresolved even after the use of GC-high resolution MS with two types of chemical ionization reagent gases. Therefore, in this case, it may be necessary to specify the use of Level B protective gear for the handling of this drum, and for the handling of drums with similar compositional uncertainties.

Sample D2911

This was a red sludge that was classified as an organic material by compatibility testing. FTIR analysis identified the material as technical grade phenol. The high absorbance of the phenolic OH group may mask the presence of minor components of the mixture. The spectrum was not examined for the presence of minor components of the mixture. The sample was not reanalyzed by GC-MS.

The identification of this mixture as phenol rather than just as an "organic sludge" is important in terms of precautions required during drum handling. Phenol carries an ACGIH TLV of five ppm, a short term exposure limit (STEL) of 10 ppm, and a "skin" notation. The IDLH level is 100 ppm. As with the case of the drum of glycol ether, the personal protection ensemble should include a splash suit that is impermeable to phenol and a full-face respirator. In addition, a THC monitor calibrated to phenol should be utilized.

Sample D2335L

This sample had been classified as an organic material by compatibility testing. FTIR showed that the sample was either water or contained sufficient water to prevent the detection of other IR absorbing species. The question of sample mishandling in the on-site mass production compatibility testing laboratory must be considered. The use of FTIR-ATR to check compatibility tests is useful to prevent errors, but an aggressive quality control program for both analysis methods is more important in protecting the health of workers.

Sample D2667

This was a transparent brown gel, classified as an organic flammable liquid, and identified by FTIR-ATR as a phthalic acid derivative. The GC-MS assay indicates that the major components of the mixture are styrene, xylene and toluene. Minor components include phthalic anhydride, n-propyl acetate, and butyl isobutyrate. The limited solubility of phthalic anhydride in the solvents used to dissolve the sample for GC-MS analysis can explain some of the discrepancy, but a quantitative assessment of the solubility of the components of this mixture was not performed.

Phthalic anhydride has an ACGIH TLV of one ppm and a STEL of four ppm, an IDLH level of 10 000 ppm, and incompatibility with strong oxidizers.(26,28) Mixtures containing this compound require personal protection consisting of garments that prevent skin contact and self-contained breathing apparatus.(28) This is necessary because of the acute effects of inhalation, ingestion or prolonged skin contact. Further work is necessary to determine if FTIR-ATR could have identified the phthalic anhydride itself, rather than just indicating that a phthalic acid derivative was present.

Sample S671

This was a quality control sample. The question of quality control for analytical procedures used in a mass production mode on a hazardous waste site is of prime importance. The quality control unknown used in this study, 1-chloronaphthalene, was correctly identified despite the loss of certain characteristic spectral features due to the use of the germanium ATR crystal. This was possible since the 1-chloronaphthalene was present as a pure compound and not as a minor component of a mixture.

Conclusions

1. The FTIR procedure has succeeded in identifying many, but not all, significant components of actual samples obtained from a hazardous waste remedial action site. Results were excellent for certain samples, but indicated that additional effort will be needed for other samples.
2. The additional data (compared to that available from compatibility testing alone) obtainable by FTIR may be employed to develop personal protection strategies based on sound industrial hygiene principles.
3. Certain limitations in the applicability of the method have been defined. These are:
    - The method is not applicable to aqueous solutions.
    - Further work must be performed to investigate the use of alternate ATR crystals or liquid cells to improve the detectability of chlorinated species in mixtures.
    - The use of improved spectral search procedures must be examined, since the FTIR instrument used in this study was not capable of performing spectral searches, especially on mixtures.

In addition, an improved hazardous waste drum analysis method must meet certain practical criteria that are inherent in the operation of a hazardous waste remedial action site. These criteria must be defined for such factors as cost, speed of analysis and applicability of instrumentation to field use conditions. Lastly, additional information obtained through the use of the proposed FTIR-ATR procedure must be incorporated into a coherent personal protection strategy that depends on a knowledge of drum contents rather than on the assumption that conditions IDLH are possible for each drum.

Acknowledgements

The authors would like to thank Bernard Orlan, U.S. Army Corps of Engineers, and Donald Bruce, U.S. EPA, for their cooperation and assistance during the on-site work on this project. Also, we would like to thank Dr. David Weitzman, U.S. EPA, who provided support and direction for this project. Funds for this study were provided by NIOSH grant 1-R01-OH02066-01 and NIOSH contract 84-2641.

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