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# Project Summary

# GC-MS Suitability Testing of RCRA Appendix VIII and Michigan List Analytes

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**As a first step in a hierarchical scheme to demonstrate the suitability of present U.S. Environmental Protection Agency (EPA) analysis methods and/or develop new methodology, the gas chromatographic (GC) separation and mass spectrometric (MS) detection characteristics of 328 toxic and/or hazardous organic materials were investigated. The analytes in question are the nonpriority pollutant organic substances in the RCRA Appendix VIII listing plus those on the "Michigan List."**

**Volatile and semivolatile analytes were tested using the GC-MS conditions specified in EPA Methods 8240 and 8270, respectively, as modified by the Contractor Laboratory Program (CLP) protocol. Standard mixtures of analytes in organic solvent were analyzed by septum injection onto the analytical column without any prior sample workup procedures.**

**For analytes that proved suitable for GC-MS analysis by Methods 8240 and 8270, the relevant characteristics for GC retention and for MS detection are reported. For analytes not detected by GC-MS or omitted *a priori* from testing, recommendations for future work are made.**

***This Project Summary was developed by EPA's Environmental Monitoring and Support Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).***

## Introduction

The Resource Conservation and Recovery Act (RCRA) specifies over 300 toxic organic compounds in its Appendix VIII listing which may be used to identify hazardous wastes. In response to a petition by the state of Michigan, the U.S. Environmental Protection Agency (EPA) has proposed the amendment of RCRA Appendix VIII by the addition of over 100 other organic compounds. EPA is currently attempting to validate analytical methods for as many of these 400 plus compounds as possible. A hierarchical approach to these validation efforts is being pursued.

An example of a hierarchical approach to the development and validation of analytical methods for the determination of organic compounds in wastes is presented in Figure 1. The final report completely describes the procedures and presents fully the results obtained from implementation of this first phase of the hierarchical approach. The compounds were classified as candidates for the application of either EPA Method 8240 or Method 8270 to test for volatile or semivolatile organic compounds, respectively. Some compounds were not tested because they fell into one of the following categories:

- Priority pollutants — The chromatographic behavior of these compounds has already been thoroughly characterized.
- Unstable — Compounds known to degrade rapidly in aqueous sample matrices.

- Not amenable to gas chromatography (GC) — Compounds known to be too polar and/or too thermally labile to elute using Method 8240 or Method 8270 conditions.
- Not available — Compounds for which standards were not available from the EPA repository or from commercial sources.

For compounds determined to be amenable to the two methods, the following data were obtained:

- GC Performance — retention characteristics.
- Mass Spectral (MS) Performance — response factors, key ions for detection and quantification using extracted ion current profiles (EICP).

Figure 1 shows the context in which the results of this project lead to subsequent method development activities. Implementation of the hierarchical method development approach is expected to contribute to the development of a suite of analytical methods with a limited number of analytic procedures for determining a large proportion of the more than 400 organic compounds in the amended RCRA Appendix VIII. Covering extraction, cleanup, and determinative steps, this limited number of analytical procedures would form the core of a generic approach to the selection of appropriate analytical methods for hazardous wastes. The proposed system is generic in the sense that the specifications of type of analyte, type of matrix, and type of sensitivity and required specificity would generate, from the limited suite of component analytical procedures, the most appropriate set of analysis conditions.

This type of generic approach would permit reduction in the number and variety of methods required to characterize wastes and should provide cost benefits both to the government and to the regulatory community. Also, the generic approach would facilitate periodic updates of the method, as new information becomes available about specific analytes in specific matrices. Further, areas requiring method modification or method development could be clearly identified and easily prioritized for research resource allocation.

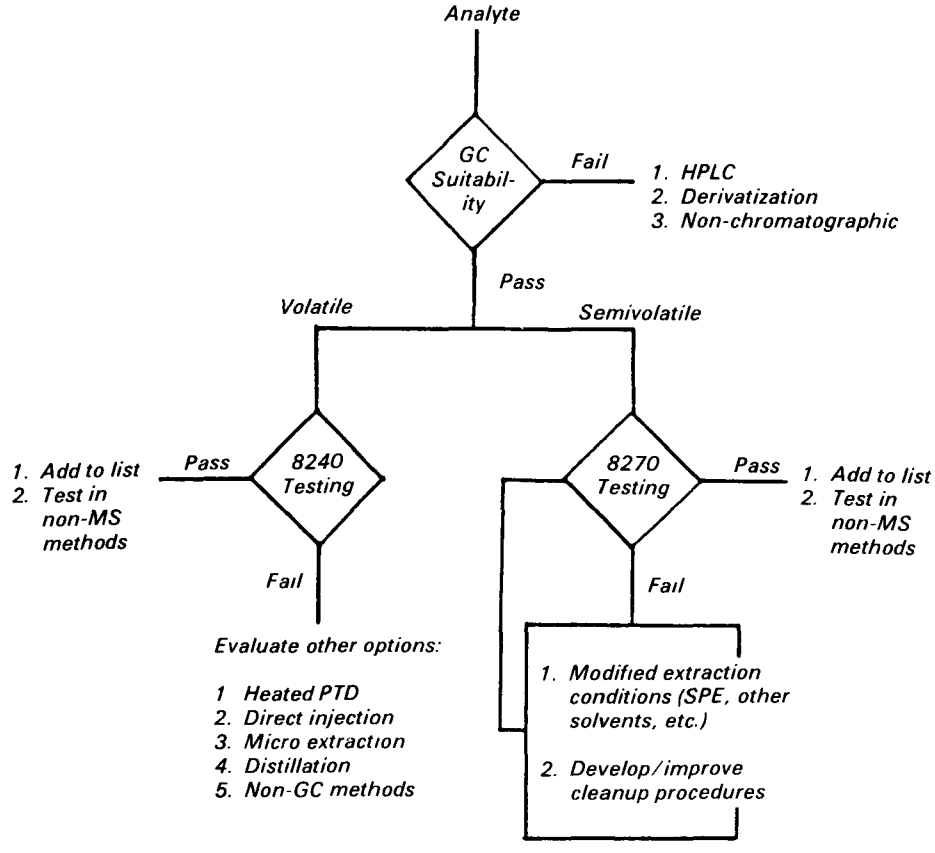


***Figure 1.*** *Hierarchical approach for analytical method development for organic RCRA analytes.*

## Experimental Approach

The initial set of analytes consisted of organic compounds included in RCRA Appendix VIII (*Federal Register,* October 1, 1984) plus those included in the Michigan petition (*Federal Register,* December, 1984) minus the EPA priority pollutants. After eliminating redundancies in the two lists, the remaining compounds were classified by their predicted suitability for SW 846 Method 8240 (volatiles), Method 8270 (semi-volatiles), or for their predicted inability to be determined by either method.

Sources for the selected analytes were identified in the following order of priority: 1) the EPA repositories of reference compounds and pesticides (EMSL - Las Vegas and RTP), 2) the EPA repository of certified solutions (EMSL - Cincinnati), and 3) commercial suppliers. GC-MS suitability studies utilized analyte mixtures prepared after consideration of chemical reactivity.

For most of the analytes, individual analyte concentrations in the volatile mixtures were 200 μg/mL, for a few analytes predicted to exhibit lower response factors; concentrations were 400 μg/mL. Injections of volatile analytes provided a minimum of 300 ng of analyte on column. The concentrations of individual semivolatile analytes in the injection standards were 40 μg/mL. For analytes not detected on the first attempt higher concentrations were employed ranging from 50-400 μg/mL. Injections of semivolatile analytes provided a minimum of 80 μg of analyte to the splitless injection evaporator cavity. The usual packed GC column, 1 percent SP1000/Carbopack B (Supelco), was used for volatile compounds, and a 30 meter × 0.25 mm ID fused silica coated with 0.25 micron immobilized methyl phenyl silicone (J&W DB-5) was used for semivolatile compounds.

Internal standards specified in the CLP for both volatile and semivolatile analyses were used to provide measures for both GC relative retention indices and MS detection response factors. Surrogate standards specified in the CLP were included in volatile analyte mixtures but not in the semivolatile mixtures. In the latter case, data interpretation would be more difficult with little increase in usefulness of results. In all cases, the CLP GC and MS analysis conditions and MS quality control checks on ion source tuning were used.

## Results and Discussion

### *Selection and Procurement of Analytes to be Tested*

After elimination of redundancies, the combined Appendix VIII and Michigan analyte sets contain 440 compounds, 112 of which are already thoroughly characterized EPA priority pollutants. Thus, 328 substances qualified for the present study. Each substance was classified according to its amenability to analysis by Method 8240 (volatile analytes), Method 8270 (semivolatile analytes), or by neither method. Eight of the compounds are classified as both volatile and semivolatile since, after failing to elute in the volatile compound testing, they were carried into the semivolatile compound testing.

Table 1 lists 58 analytes that are omitted *a priori* from GC-MS suitability testing. Reasons for *a priori* omission and suggestions for future method development can be classified as follows:

- Acids — 14 Compounds. Typically, these acids are carboxylic acids (or of comparable strength) and require derivatization to achieve acceptable GC performance.

- Nonvolatile — 12 compounds. These analytes are so polar and/or of such high molecular weight that there is essentially no possibility of their elution from a GC column. Typical of these compounds are macrolytic antibiotics and dye molecules for which HPLC, rather than a derivatization-GC approach is appropriate for most cases.

- Hydrolytically or otherwise unstable — 9 compounds. Since groundwater and wet soils and sludges are sample matrices of interest, there is no need to demonstrate analysis capability for compounds which decompose rapidly in these sample types.

- Aldehydes — 6 compounds. Special GC conditions have been used successfully for a number of these analytes but, generally, the associated sample workup procedures are not fully successful in preserving these reactive analytes prior to their detection. Hence derivatization followed by HPLC with UV detection, which has been shown to be sensitive and reproducible for formaldehyde, is clearly the favored analytical

**Table 1.** *Analytes Omitted A Priori From GC-MS Suitability Testing*

| No. | Substance | List(a) | CAS No. |
|---|---|---|---|
| 1 | Acetaldehyde(b) | 8 | 75-07-0 |
| 2 | Acetyl chloride | 8 | 75-36-5 |
| 3 | Actinomycin D | M | 50-76-0 |
| 4 | Aflatoxins(b) | 8 | 98-05-5 |
| 5 | Aniline hydrochloride | M | 142-04-1 |
| 6 | O-Anisidine hydrochloride | M | 134-29-2 |
| 7 | Azaserine | 8 | 115-02-6 |
| 8 | Benzal chloride | 8 | 98-87-3 |
| 9 | Benzenearsonic acid | 8 | 98-05-5 |
| 10 | Benzidine sulfate | M | 531-86-2 |
| 11 | Benzotrichloride | 8 | 98-07-7 |
| 12 | Chlorambucil | 8 | 305-03-3 |
| 13 | Chloroacetaldehyde(b) | 8 | 107-20-0 |
| 14 | Coal tars | 8 | 20830-81-3 |
| 15 | Creosote | 8 | 8001-58-9 |
| 16 | Crotonaldehyde(b) | 8 | 123-73-9 |
| 17 | Cycasin | 8 | 14901-08-7 |
| 18 | 2,4-D | 8 | 94-75-7 |
| 19 | Daunomycin | 8 | 20830-81-3 |
| 20 | Dimethylcarbamoyl chloride | 8 | 79-44-7 |
| 21 | Diphenylamine(c) | 8 | 62-74-8 |
| 22 | 5,5-Diphenylhydantoin monosodium salt | M | 630-93-3 |
| 23 | 2,4-Dithiobuiret | 8 | 541-53-7 |
| 24 | Endothal | 8 | 129-67-9 |
| 25 | Epinephrine | 8 | 51-43-4 |
| 26 | Ethylene bis-dithiocarbamates | 8 | 142-59-6 |
| 27 | Fluoroacetic acid, sodium salt | 8 | 62-74-8 |
| 28 | Formaldehyde(b) | 8 | 50-00-0 |
| 29 | Formic acid | 8 | 64-18-6 |
| 30 | Glycidaldehyde (b) | 8 | 765-34-4 |
| 31 | Hydrazine | 8 | 302-01-2 |
| 32 | Ketene | M | 463-51-4 |
| 33 | Lasiocarpine | 8 | 303-34-4 |
| 34 | Malachite green | M | 569-64-2 |
| 35 | Melphalan | 8 | 148-82-3 |
| 36 | Methyl chlorocarbonate | 8 | 79-22-1 |
| 37 | Mitomycin C | 8 | 50-07-7 |
| 38 | Monocrotaline | M | 315-22-0 |
| 39 | Nitrogen mustard N-oxide | 8 | 302-70-5 |
| 40 | N-Nitrososarcosine | 8 | 13256-22-9 |
| 41 | Paraldehyde(b) | 8 | 123-63-7 |
| 42 | Peroxyacetic acid | M | 79-21-0 |
| 43 | Phenesterin | M | 3546-10-9 |
| 44 | Phenyl dichloroarsine | 8 | 696-28-6 |
| 45 | Polybrominated biphenyls(b) | M | 59536-65-1 |
| 46 | Polychlorinated biphenyls(b) | 8 | 1336-36-3 |
| 47 | Reserpine | 8 | 50-55-5 |
| 48 | Semicarbazide | M | 57-56-7 |
| 49 | Silvex | 8 | 93-72-1 |
| 50 | Streptozotocin | 8 | 18883-66-4 |
| 51 | 2,4,5-T | 8 | 93-76-5 |
| 52 | 2,3,7,8-TCDD(b) | 8 | 1746-01-6 |
| 53 | Thiosemicarbazide | 8 | 79-19-6 |
| 54 | Thiourea | 8 | 62-56-6 |
| 55 | Thiram(d) | 8 | 137-26-8 |
| 56 | Trichloromethanesulfenyl chloride | 8 | 594-42-3 |
| 57 | Trypan blue | 8 | 72-57-1 |
| 58 | Ziram | M | 137-30-4 |

*(a) 8 = Appendix VIII; M = Michigan List.*

*(b) Partially or fully demonstrated method for selected matrices already exists.*

*(c) Diphenylamine has been well characterized by GC-MS since the priority pollutant N-nitrodiphenyl-amine has been thoroughly demonstrated to quantitatively decompose to diphenylamine upon GC injection.*

*(d) This family of analytes have been successfully determined in aqueous media by GC-Hall/Sulfur analysis of carbon disulfide decomposition product.*

approach for these compounds. Thus, there was no need to include them in GC method testing.

- Redundancies — 5 compounds. Typically, these compounds are salts of a free base also listed as an analyte. Diphenylamine was considered redundant with the priority pollutant, N-nitrosodiphenylamine since the latter is known to decompose quantitatively to the former in GC injectors.
- Inappropriate for inclusion in the present scope for miscellaneous reasons — 12 substances. Some analytes were considered inappropriate because they can hardly be considered organic analytes. Examples of this category include hydrazine, thiosemicarbizide, semicarbizide, thiourea and dithiobiuret. Others are currently, or expected to be addressed by other specialized methods. Examples of this category include tetrachlorodibenzodioxin and polychlorinated and polybrominated biphenyls. Two substances, coal tars and creosote, are too heterogeneous for inclusion in Method 8270. Epinephrine had to be omitted since it could not be dissolved at sufficiently high concentration in any reasonable GC injection solvent.

Some 32 analytes could not be obtained in time to be included in this work and are listed in Table 2. One of these, chloral, is available as the hydrate which was included in the volatile analyte testing. Thus, the summary listings show 31 analytes as "unavailable."

## Results of Volatile Analyte Testing

Table 3 lists 54 volatile analytes tested with Method 8240 GC conditions. The status of each analyte is indicated: satisfactorily detected (S); detected with a response factor versus benzene-$D_6$ below 0.02 (LR), or not detected (ND). Thirty three analytes were satisfactorily detecte and six were detected with low respons factors. The very low response factor will probably result in unacceptably hig minimum detection limit (MDL) value for Method 8240.

Table 3 also lists 15 volatile analyte that were not detected under Metho 8240 conditions. All of these analyte were analyzed at least twice, with th repeat analysis usually at 2- to 5-fol higher levels than the original 300 n level. Three of these 15 analytes, hexa chloropropene, tetranitromethane, an thiophenol, were thought to have faile to elute due to boiling points and/o polarities that were too high for th SP1000/Carbopack B column, and thes compounds were retested using th Method 8270 (semivolatile analyte conditions.

Non-detection of the hydrazines an aziridines (6 analytes) was probably du to extreme GC peak tailing on th SP1000/Carbopack B column. Five o these nitrogen bases were also teste with the semivolatile analytes. The sixth N(2-hydroxyethyl)ethyleneimine, was no tested due to its extreme polarity.

In the injector, 2-Butanone peroxide apparently quantitatively decomposed t 2-butanone. Methyl mercaptan apparentl coelutes with methanol on the SP1000/ Carbopack B column and would be substantially lost at the jet separator due t the presence of the methanol vapor displacement of the helium carrier. The remaining four undetected volatile compounds, two haloethers, methyl isocyanate, and 2-methyllactonitrile, were not repeated in the semivolatile set since they were both too volatile to be recovered in a Kuderna-Danish (KD) distillation of extraction solvent and were also known to be chemically and/or hydrolytically labile.

## Results of Semivolatile Analyte Testing

Table 4 lists the 185 semivolatile analytes and the eight volatile analytes to be retested with Method 8270 conditions. The status of each analyte is indicated; satisfactorily detected (S), expected to be satisfactory for GC-MS determination based on other information (ES), detected with a response factor less than 0.02 versus phenanthrene-$D_{10}$ (LR), or not detected (ND). One hundred and twenty eight analytes were detected with satisfactory response factors and nine analytes were detected with low response factors.

**Table 2.** *Volatile and Semivolatile Analytes Not Obtainable in Time for Inclusion*

| No. | Substance | List(a) | CAS No. |
|---|---|---|---|
| 1 | 1-Amino-2-methylanthraquinone | M | 82-28-0 |
| 2 | 5-(Aminomethyl)-3-isoxazolol | 8 | 2763-96-4 |
| 3 | Azinophos-ethyl | M | 2642-71-9 |
| 4 | Benz(c)acridine | 8 | 225-51-4 |
| 5 | 7,8-Benzfluoranthene | 8 | 205-82-3 |
| 6 | 4-(Butylnitrosoamino)-1-butanol | M | 3817-11-6 |
| 7 | Chloral(b) | 8 | 75-87-6 |
| 8 | Chloronaphazine | 8 | 494-03-1 |
| 9 | 1-(2-Chlorophenyl)thiourea | 8 | 5344-82-1 |
| 10 | Citrus Red No. 2 | 8 | 6358-53-8 |
| 11 | 1,2:5,6-Dibenzacridine | 8 | 226-36-8 |
| 12 | 3,4:5,6-Dibenzocarbazole | 8 | 194-59-2 |
| 13 | Dibenzo(a,i)pyrene(c) | 8 | 189-55-9 |
| 14 | Dibenzo(a,h)pyrene | 8 | 129-67-9 |
| 15 | Diethylarsine | 8 | 692-42-2 |
| 16 | 1,2-Diethylhydrazine | 8 | 1615-80-1 |
| 17 | Diisopropyl fluorophosphate | 8 | 55-91-4 |
| 18 | 3,3-Dimethyl-1-(p-chlorophenyl)triazene | M | 7203-90-9 |
| 19 | O,O-Dimethyl-S-methyl phosphorodithioate | 8 | 3288-58-2 |
| 20 | Furathiazole | M | 531-82-8 |
| 21 | Hexaethyl tetraphosphate | 8 | 757-58-4 |
| 22 | 4,4'-Methylenebis(2-methylaniline) | M | 838-88-0 |
| 23 | 2-Methyl-1-nitroanthraquinone | M | 129-15-7 |
| 24 | Nifurthiazol | M | 3570-75-0 |
| 25 | Niridazole | M | 61-57-4 |
| 26 | Nithiazide | M | 139-94-6 |
| 27 | N-Nitrosomethylvinylamine | 8 | 4549-40-0 |
| 28 | N-Nitrosonornicotine | 8 | 16543-55-8 |
| 29 | Phosacetim | M | 4104-14-7 |
| 30 | 4,4'-Thiodianiline | M | 139-65-1 |
| 31 | Tris(1-azridinyl)phosphine sulfide | 8 | |
| 32 | Uracil mustard | 8 | 66-75-1 |

(a) 8 = Appendix VIII; M = Michigan list.
(b) Chloral hydrate was substituted for chloral.
(c) Judged cost prohibitive for this program.

**Table 3.** *GC-MS Suitability Testing Results for Volatile Analytes*

| No. | Substance | List(a) | | CAS No. | RCRA Number | Status Code(b) |
|---|---|---|---|---|---|---|
| 1 | Acetonitrile | 8 | | 75-05-8 | U003 | S |
| 2 | Allyl alcohol | 8 | | 107-18-6 | P005 | S |
| 3 | Allyl chloride | 8 | M | 107-05-1 | U317 | S |
| 4 | Benzyl chloride | 8 | | 100-44-7 | P028 | S |
| 5 | Bis-(2-chloroethyl) sulfide | 8 | M | 505-60-2 | P158 | LR |
| 6 | Bis(chloromethyl) ether | 8 | | 542-88-1 | | ND |
| 7 | Bromoacetone | 8 | | 598-31-2 | P017 | S |
| 8 | 2-Butanone peroxide | 8 | | 1338-23-4 | U160 | ND |
| 9 | 2-Butanone | 8 | | 78-93-3 | | S |
| 10 | Carbon disulfide | 8 | | 75-15-0 | | S |
| 11 | Chloral hydrate | 8 | | 75-87-6 | U034 | LR |
| 12 | 2-Chloroethanol | | M | 107-07-3 | P133 | LR |
| 13 | Chloromethyl methyl ether | 8 | | 107-30-2 | U046 | ND |
| 14 | Chloroprene | 8 | M | 126-99-8 | U276 | S |
| 15 | 3-Chloropropionitrile | 8 | | 542-76-7 | P027 | S |
| 16 | 1,2-Dibromo-3-chloropropane | 8 | | 96-12-8 | | S |
| 17 | Dibromomethane | 8 | | 74-95-3 | | S |
| 18 | 1,4-Dichloro-2-butene | 8 | | 764-41-0 | U074 | S |
| 19 | Dichlorodifluoromethane | 8 | | 75-71-8 | | |
| 20 | 1,3-Dichloro-2-propanol | 8 | | 96-23-1 | | S |
| 21 | 1,2,3,4-Diepoxybutane | 8 | | 1464-53-5 | U085 | S |
| 22 | 1,1-Dimethylhydrazine | 8 | | 57-14-7 | U098 | ND |
| 23 | 1,2-Dimethylhydrazine | 8 | | 540-73-8 | U099 | ND |
| 24 | 1,4-Dioxane | 8 | | 123-91-1 | U108 | S |
| 25 | Epichlorohydrin | 8 | | 106-89-8 | | S |
| 26 | Ethylene dibromide | 8 | | 106-93-4 | | S |
| 27 | Ethylene oxide | 8 | | 75-21-8 | U115 | S |
| 28 | Ethylenimine | 8 | | 151-56-4 | P054 | ND |
| 29 | Ethyl methacrylate | 8 | | 97-63-2 | U118 | S |
| 30 | Hexachloropropene | 8 | | 1888-71-7 | U243 | ND |
| 31 | N-(2-Hydroxyethyl)ethyleneimine | | M | 1072-52-2 | U289 | ND |
| 32 | 2-Hydroxypropionitrile | | M | 78-97-7 | | LR |
| 33 | Isobutyl alcohol | 8 | | 78-83-1 | | S |
| 34 | Malononitrile | 8 | | 109-77-3 | U149 | S |
| 35 | Methacrylonitrile | 8 | | 126-98-7 | U152 | S |
| 36 | 2-Methylaziridine | 8 | | 75-55-8 | P067 | ND |
| 37 | Methylhydrazine | 8 | | 60-34-4 | P068 | ND |
| 38 | Methyl iodide | 8 | | 74-88-4 | U138 | S |
| 39 | Methyl isocyanate | 8 | | 624-83-9 | P064 | ND |
| 40 | 2-Methyllactonitrile | 8 | | 75-86-5 | P069 | ND |
| 41 | Methyl mercaptan | 8 | | 74-93-1 | | ND |
| 42 | Methyl methacrylate | 8 | | 80-62-6 | U162 | S |
| 43 | Pentachloroethane | 8 | | 76-01-7 | | S |
| 44 | 2-Picoline | 8 | | 109-06-8 | U191 | S |
| 45 | Propargyl alcohol | 8 | | 107-19-7 | P102 | LR |
| 46 | β-Propiolactone | | M | 57-57-8 | U302 | S |
| 47 | Propionitrile | 8 | | 107-12-0 | P101 | S |
| 48 | N-Propylamine | 8 | | 107-10-8 | U194 | LR |
| 49 | Pyridine | 8 | | 110-86-1 | U196 | S |
| 50 | Styrene | | M | 100-42-5 | U323 | S |
| 51 | 1,1,1,2-Tetrachloroethane | 8 | | 630-20-6 | | S |
| 52 | Tetranitromethane | 8 | | 509-14-8 | P112 | ND |
| 53 | Thiophenol | 8 | | 108-98-5 | P104 | ND |
| 54 | 1,2,3-Trichloropropane | 8 | | 96-18-4 | | S |

(a) 8 = Appendix VIII; M = Michigan list.
(b) LR: low response factor
S: suitable for GC-MS analysis
ND not detected in GC-MS data.

All of these latter nine analytes are highly polar and are expected to be sensitive to thermal decomposition in the injection port.

Table 4 contains 11 analytes with the status "ES." All 11 of these analytes are suitably analyzed in Work Assignment 2-08 which extracts analytes from spiked aqueous standards, concentrated and analyzed by fused silica capillary GC-FID. Thus, although MS data was not obtained in the WA 2-08 work, their non-detection in the present work is anomalous. Except for the two organophosphates, these "ES" analytes in Table 4 are strongly basic molecules; a possible explanation for their non-detection is that the GC column used was somewhat acidic, precluding satisfactory elution.

Also listed in Table 4, are the 45 analytes for which non-detection in the GC-MS data cannot be classified as anomalous. Generally, these analytes are highly polar, or labile to decomposition before or during chromatography. Four of these 45 analytes are aromatic diamines, 1,2- and 1,3-phenylenediamine, 2,4-diaminoanisole, and 1,5-naphthalendiamine. These four analytes probably can be analyzed by fused silica GC if special precautions are taken to ensure good performance for basic materials. Ethylene thiourea (ETU) has been shown in previous work at Battelle to be amenable to GC analysis using special conditions. For another six analytes (acrylamide, cycloheximide, 2-fluoroacetamide, niclosamide, oxydemeton-methyl, and thioacetamide) polarity, volatility and lability considerations apparently do not account for the non-detection, and, therefore, a more thorough attempt to develop GC-based methods might be successful. For the remaining 34 analytes, the causes of non-detection can be classified as one or more of the following: exceptionally high polarity, thermal or chemical lability, or insufficient volatility. Recommendations for further method development for these analytes focus on HPLC techniques, especially ion chromatography or post column derivatization methods.

## Conclusions and Recommendations

After the elimination of redundancies, the Appendix VIII and Michigan list compounds include 440 organic compounds. The classification of these compounds and results of testing them for suitability for inclusion in present volatile and semivolatile analysis methods are summarized as follows:

Analytes Considered

| | | | | |
|---|---|---|---|---|
| Tested for GC-MS Suitability | | | | 239* |
| Omitted Priority Pollutant | | | | 112 |
| Omitted A Priori | | | | 58 |
| Not obtainable | | | | 31 |
| Total considered | | | | 440 |
| Volatile Analyte Testing | | | | |
| Suitably Detected | 33 | | | |
| Detected with Low Response Factor | | 6 | | |
| Not Detected | | 15 | | |
| Sub Total | 33 | + 21 | = | 54* |
| Semivolatile Analyte Testing | | | | |
| Suitably Detected | 128 | | | |
| Expected to be Suitable | 11 | | | |
| Detected with Low Response Factor | | 9 | | |
| Not Detected | | 45 | | |
| Sub Total | 139 | + 54 | = | 193* |
| Total Suitable | 172 | | | |
| Total Not Suitable | | 75 | | |
| Total Tested | | | | 247* |

* Eight of the analytes that failed volatile testing were carried into the semivolatile testing. Two of these eight analytes were suitably detected in semivolatile testing.

For the 39 volatile and 137 semivolatile compounds which were detected by GC-MS analyses, key GC performance and mass spectral data were obtained.

The general recommendation arising from the data and results generated in the studies presented in this report is to continue implementation of the hierarchical research strategy. The following specific recommendations are made:

- The 39 volatile compounds detected using the GC conditions of Method 8240 should be included in an evaluation of the Method 5030 PTD sample introduction.
- Extractability studies of the 137 semivolatile compounds detected using the GC conditions of Method 8270 should be conducted.
- The 11 compounds which were anomalously not detected should receive more detailed examination.
- Twelve additional analytes that were not detected should also receive further direct GC investigation.

**Table 4.** *GC-MS Suitability Testing Results for Semivolatile Analytes*

| No. | Substance | List(a) | CAS No. | RCRA Number | Status Code(b) |
|---|---|---|---|---|---|
| 1 | Acetophenone | 8 | 98-86-2 | U004 | S |
| 2 | 2-Acetylaminofluorene | 8 | 53-96-3 | U005 | S |
| 3 | 1-Acetyl-2-thiourea | 8 | 591-08-2 | P002 | LR |
| 4 | Acrylamide | 8 | 79-06-1 | | ND |
| 5 | Aldicarb | 8 | 116-06-3 | | ND |
| 6 | 2-Aminoanthraquinone | M | 117-79-3 | U264 | S |
| 7 | Aminoazobenzene | M | 60-09-3 | U257 | S |
| 8 | 4-Aminobiphenyl | 8 M | 92-67-1 | U274 | S |
| 9 | 3-Amino-9-ethylcarbazole | M | 132-32-1 | U253 | ES |
| 10 | Amitrole | 8 | 61-82-5 | U011 | ND |
| 11 | Anilazine | M | 101-05-3 | U333 | S |
| 12 | Aniline | 8 | 62-53-3 | | ES |
| 13 | o-Anisidine | M | 90-04-0 | U260 | S |
| 14 | Aramite | 8 M | 140-57-8 | U326 | S |
| 15 | Auramine | 8 | 492-80-8 | U014 | ND |
| 16 | Azinphos-methyl | M | 86-50-0 | P151 | S |
| 17 | Barban | M | 101-27-9 | U280 | LR |
| 18 | Benomyl | M | 17804-35-2 | U271 | ND |
| 19 | p-Benzoquinone | 8 | 106-51-4 | U197 | S |
| 20 | Bromoxynil | M | 1689-84-5 | U272 | S |
| 21 | Brucine | 8 | 357-57-3 | P018 | ND |
| 22 | Captafol | M | 2425-06-1 | U285 | S |
| 23 | Captan | M | 133-06-2 | U266 | S |
| 24 | Carbaryl | M | 63-25-2 | U279 | S |
| 25 | Carbofuran | M | 1563-66-2 | U127 | S |
| 26 | Carbophenothion | M | 786-19-6 | U148 | S |
| 27 | Chlorfenvinphos | M | 470-90-6 | P143 | S |
| 28 | 4-Chloroaniline | 8 | 106-47-8 | | S |
| 29 | Chlorobenzilate | 8 | 510-15-6 | U038 | S |
| 30 | 5-Chloro-2-methylaniline | M | 95-79-4 | U329 | S |
| 31 | 3-(Chloromethyl)pyridine hydrochloride | M | 6959-48-4 | U319 | S |
| 32 | 4-Chloro-1,3-phenylenediamine | M | 5131-60-2 | U305 | ES |
| 33 | 4-Chloro-1,2-phenylenediamine | M | 95-83-0 | U306 | ES |
| 34 | Coumaphos | M | 56-72-4 | P130 | S |
| 35 | p-Cresidine | M | 120-71-8 | U262 | S |
| 36 | Crotoxyphos | M | 7700-17-6 | U238 | S |
| 37 | Cupferron | M | 135-20-6 | U290 | ND |
| 38 | Cycloheximide | M | 66-81-9 | P134 | ND |
| 39 | 2-Cyclohexyl-4,6-dinitrophenol | 8 | 131-89-5 | P034 | LR |
| 40 | Cyclophosphamide | 8 | 50-18-0 | U058 | ND |
| 41 | Demeton | M | 8065-48-3 | P155 | S |
| 42 | Diallate | 8 | 2303-16-4 | U062 | S |
| 43 | 2,4-Diaminoanisole sulfate | M | 39156-41-7 | U307 | ND |
| 44 | 2,4-Diaminotoluene | 8 M | 95-80-7 | U327 | S |
| 45 | Diazinon | M | 333-41-5 | U313 | ES |
| 46 | 1,2:7,8-Dibenzacridine | 8 | 224-42-0 | | S |
| 47 | 1,2:4,5-Dibenzopyrene | 8 | 192-65-4 | | S |
| 48 | Dichlone | M | 117-80-6 | U299 | S |
| 49 | 2,6-Dichlorophenol | 8 | 87-65-0 | | S |
| 50 | Dichlorovos | M | 62-73-7 | P144 | S |
| 51 | Dicrotophos | M | 141-66-2 | P146 | S |
| 52 | Diethylstilbestrol | 8 | 56-53-1 | U086 | S |
| 53 | Diethyl sulfate | M | 64-67-5 | U325 | LR |
| 54 | Dihydrosafrole | 8 | 56312-13-1 | U090 | ND |
| 55 | Dimethoate | 8 | 60-51-5 | | S |
| 56 | 3,3'-Dimethoxybenzidine | 8 | 119-90-4 | U091 | LR |
| 57 | 1,4-Dimethylaminoazobenzene | 8 | 60-11-7 | U093 | S |
| 58 | 7,12-Dimethylbenz(a)anthracene | 8 | 57-97-6 | U094 | S |
| 59 | 3,3'-Dimethylbenzidine | 8 | 119-93-7 | U095 | S |
| 60 | 1,1-Dimethylhydrazine | 8 | 57-14-7 | U098 | ND |
| 61 | 1,2-Dimethylhydrazine | 8 | 540-73-8 | U099 | ND |
| 62 | αα-Dimethylphenethylamine | 8 | 122-09-8 | P046 | S |
| 63 | 1,2-Dinitrobenzene | 8 | 99-65-0 | | S |
| 64 | 1,3-Dinitrobenzene | 8 | 528-29-0 | | S |
| 65 | 1,4-Dinitrobenzene | 8 | 100-25-4 | | S |
| 66 | Dinocap | M | 39300-45-3 | U284 | S |

- For 33 non-detected analytes, HPLC (including ion chromatography or post-column derivatization) should be investigated.
- No recommendations can be made at this time for 7 analytes not detected in this study.

*Table 4. (continued)*

| | | | | | | |
|---|---|---|---|---|---|---|
| 67 | Dinoseb | 8 | | 88-85-7 | | S |
| 68 | Dioxathion | | M | 78-34-2 | P153 | S |
| 69 | 5,5-Diphenylhydantoin | | M | 57-41-0 | | S |
| 70 | 1,2-Diphenylhydrazine | 8 | | 122-66-7 | U109 | ND |
| 71 | Disulfoton | 8 | | 298-04-4 | | S |
| 72 | EPN | | M | 2104-64-5 | P141 | S |
| 73 | Ethion | | M | 563-12-2 | P154 | S |
| 74 | Ethyl carbamate | 8 | | 51-79-6 | U238 | S |
| 75 | Ethylenimine | 8 | | 151-56-4 | P054 | ND |
| 76 | Ethylene thiourea | 8 | | 96-45-7 | | ND |
| 77 | Ethyl methanesulfonate | 8 | | 62-50-0 | U119 | S |
| 78 | Famphur | 8 | | 52-85-7 | P097 | S |
| 79 | Fensulfothion | | M | 115-90-2 | P156 | S |
| 80 | Fenthion | | M | 55-38-9 | | S |
| 81 | Fluchloralin | | M | 33245-39-5 | U330 | S |
| 82 | 2-Fluoroacetamide | 8 | | 640-19-7 | P057 | ND |
| 83 | Hexachlorophene | 8 | | 70-30-4 | U132 | S |
| 84 | Hexachloropropene | 8 | | 1888-71-7 | U243 | S |
| 85 | Hexamethyl phosphoramide | | M | 680-31-9 | U312 | S |
| 86 | Hydroquinone | | M | 123-31-9 | | S |
| 87 | Isodrin | 8 | | 465-73-6 | P060 | S |
| 88 | Isonicotinic acid hydrazide | | M | 54-85-3 | | ND |
| 89 | Isosafrole | 8 | | 120-58-1 | U141 | S |
| 90 | Kepone | 8 | | 143-50-0 | | S |
| 91 | Leptophos | | M | 21609-90-5 | P140 | S |
| 92 | Malathion | | M | 121-75-5 | U324 | S |
| 93 | Maleic anhydride | 8 | | 108-31-6 | U147 | S |
| 94 | Maleic hydrazide | 8 | | 123-33-1 | | ND |
| 95 | Mestranol | | M | 72-33-3 | U301 | S |
| 96 | Methapyrilene | 8 | | 91-80-5 | U155 | S |
| 97 | Methomyl | 8 | | 16752-77-5 | P066 | ND |
| 98 | p,p'-Methoxychlor | 8 | | 72-43-5 | | S |
| 99 | 2-Methylaziridine | 8 | | 75-55-8 | P067 | ND |
| 100 | 3-Methylcholanthrene | 8 | | 56-49-5 | U157 | S |
| 101 | 4,4'-Methylenebis(2-chloroaniline) | 8 | | 101-14-4 | U158 | LR |
| 102 | 4,4'-Methylenebis(N,N-dimethylaniline) | | M | 101-61-1 | U255 | ES |
| 103 | Methylhydrazine | 8 | | 60-34-4 | P068 | ND |
| 104 | Methyl methanesulfonate | 8 | | | | S |
| 105 | N-Methyl-N-nitro-N-nitrosoguanidine | 8 | | 70-25-7 | U163 | ND |
| 106 | Methyl parathion | 8 | | 298-00-0 | | S |
| 107 | 2-Methylphenol | 8 | | 95-48-7 | | S |
| 108 | 3-Methylphenol | 8 | | 108-39-4 | | S |
| 109 | 4-Methylphenol | 8 | | 106-44-5 | | S |
| 110 | Methylthiouracil | 8 | | 56-04-2 | U164 | ND |
| 111 | Mevinphos | | M | 7786-34-7 | P131 | S |
| 112 | Mexacarbate | | M | 315-18-4 | P128 | S |
| 113 | Mirex | | M | 2385-85-5 | U297 | S |
| 114 | Monocrotophos | | M | 6923-22-4 | P147 | S |
| 115 | Naled | | M | 300-76-5 | U309 | S |
| 116 | 1,5-Naphthalenediamine | | M | 2243-62-1 | U298 | ND |
| 117 | 1,4-Naphthoquinone | 8 | | 130-15-4 | U166 | S |
| 118 | 1-Naphthylamine | 8 | | 134-32-7 | U167 | S |
| 119 | 2-Naphthylamine | 8 | | 91-59-8 | U168 | ES |
| 120 | 1-Naphthyl-2-thiourea | 8 | | 86-88-4 | P072 | ND |
| 121 | Niclosamide | | M | 50-65-7 | U321 | ND |
| 122 | Nicotine | 8 | | 54-11-5 | P075 | S |
| 123 | 5-Nitroacenaphthene | | M | 602-87-9 | U250 | S |
| 124 | 4-Nitroaniline | 8 | | 100-01-6 | | S |
| 125 | 5-Nitro-o-anisidine | | M | 99-59-2 | U263 | S |
| 126 | 4-Nitrobiphenyl | | M | 92-93-3 | U275 | S |
| 127 | Nitrofen | | M | 1836-75-5 | U288 | S |
| 128 | Nitrogen mustard | 8 | M | 51-75-2 | P132 | ND |
| 129 | Nitroglycerine | 8 | | 55-63-0 | P081 | ND |
| 130 | 5-Nitro-o-toluidine | 8 | | 99-55-8 | U181 | S |
| 131 | 4-Nitroquinoline-1-oxide | 8 | | 56-57-5 | | S |
| 132 | N-Nitrosodibutylamine | 8 | | 924-16-3 | | S |
| 133 | N-Nitrosodiethanolamine | 8 | | 1116-54-7 | U173 | ND |
| 134 | N-Nitrosodiethylamine | 8 | | 55-18-5 | | S |
| 135 | p-Nitrosodiphenylamine | | M | 156-10-5 | U287 | ES |

*Table 4. (continued)*

| | | | | | |
|---|---|---|---|---|---|
| 136 | N-Nitroso-N-ethylurea | 8 | | 759-73-9 | U176 | ND |
| 137 | N-Nitrosomethylethylamine | 8 | | 10595-95-6 | | S |
| 138 | N-Nitroso-N-methylurea | 8 | | 684-93-5 | U177 | ND |
| 139 | N-Nitroso-N-methylurethane | 8 | | 615-53-2 | U178 | ND |
| 140 | N-Nitrosomorpholine | 8 | | 59-89-2 | | ES |
| 141 | N-Nitrosoplperidine | 8 | | 100-75-4 | U176 | S |
| 142 | N-Nitrosopyrrolidine | 8 | | 930-55-2 | | S |
| 143 | Octamethylpyrophosphoramide | 8 | | 152-16-9 | P085 | LR |
| 144 | Oxydemeton-methyl | | M | 301-12-2 | P157 | ND |
| 145 | 4,4'-Oxydianiline | | M | 101-80-4 | U303 | S |
| 146 | Parathion ethyl | 8 | | 56-38-2 | | S |
| 147 | Pentachlorobenzene | 8 | | 608-93-5 | | S |
| 148 | Pentachloronitrobenzene | 8 | | 82-68-8 | | S |
| 149 | Phenacetin | 8 | | 62-44-2 | U187 | S |
| 150 | Phenazopyridine hydrochloride | | M | 136-40-3 | U320 | ND |
| 151 | Phenobarbital | | M | 50-06-6 | U268 | S |
| 152 | 1,2-Phenylenediamine | 8 | | 95-54-5 | | ND |
| 153 | 1,3-Phenylenediamine | 8 | | 108-45-2 | | ND |
| 154 | 1,4-Phenylenediamine | 8 | | 106-50-3 | | S |
| 155 | N-Phenylthiourea | 8 | | 103-85-5 | P093 | ND |
| 156 | Phorate | 8 | | 298-02-2 | | S |
| 157 | Phosalone | 8 | | 2310-17-0 | | S |
| 158 | Phosmet | | M | 732-11-6 | | S |
| 159 | Phosphamidon | | M | 13171-21-6 | P145 | S |
| 160 | Phthalic anhydride | 8 | | 85-44-9 | U190 | S |
| 161 | Piperonyl sulfoxide | | M | 120-62-7 | U270 | S |
| 162 | Pronamide | 8 | | 23950-58-5 | | S |
| 163 | 1,3-Propane sultone | 8 | | 1120-71-4 | U193 | ND |
| 164 | Propylthiouracil | 8 | M | 51-52-5 | U334 | LR |
| 165 | Resorcinol | 8 | | 108-46-3 | | S |
| 166 | Rotenone | | M | 83-79-4 | U273 | ND |
| 167 | Saccharin | 8 | | 81-07-2 | U202 | ND |
| 168 | Safrole | 8 | | 94-59-7 | U203 | S |
| 169 | Strychnine | 8 | | 57-24-9 | | S |
| 170 | Sulfallate | | M | 95-06-7 | U277 | S |
| 171 | Terbufos | | M | 13071-79-9 | P149 | S |
| 172 | 1,2,4,5-Tetrachlorobenzene | 8 | | 95-94-3 | | S |
| 173 | 2,3,4,6-Tetrachlorophenol | 8 | | 58-90-2 | | S |
| 174 | Tetrachlorvinphos | | M | 961-11-5 | U308 | S |
| 175 | Tetraethyl dithiopyrophosphate | 8 | | 3689-24-5 | P109 | ES |
| 176 | Tetraethyl pyrophosphate | 8 | | 107-49-3 | | S |
| 177 | Tetranitromethane | 8 | | 509-14-8 | P112 | ND |
| 178 | Thioacetamide | 8 | | 62-55-5 | U128 | S |
| 179 | Thiofanox | 8 | | 39196-18-4 | P045 | ND |
| 180 | Thionazine | 8 | | 297-97-2 | P040 | S |
| 181 | Thiophenol | 8 | | 108-98-5 | P104 | S |
| 182 | Toluene diisocyanate | 8 | | 584-84-9 | U223 | S |
| 183 | o-Toluidine | 8 | M | 95-53-4 | U328 | S |
| 184 | Trichlorform | | M | 52-68-6 | P139 | ND |
| 185 | 2,4,5-Trichlorophenol | 8 | | 95-95-4 | | S |
| 186 | O,O,O-Triethyl phosphorothioate | 8 | | 126-68-1 | | ES |
| 187 | Trifluralin | | M | 1582-09-8 | U332 | S |
| 188 | 2,4,5-Trimethylaniline | | M | 137-17-7 | U259 | S |
| 189 | Trimethyl phosphate | | M | 512-56-1 | U310 | S |
| 190 | 1,3,5-Trinitrobenzene | 8 | | 99-35-4 | U234 | S |
| 191 | Tris(2,3-dibromopropyl) phosphate | 8 | | 126-72-7 | U235 | LR |
| 192 | Tri-p-tolyl phosphate(c) | | M | 78-32-0 | | S |
| 193 | Warfarin | 8 | | 81-81-2 | P001 | ND |

(a) 8 = Appendix VIII; M = Michigan list.

(b) S - apparently suitable for GC-MS analysis
LR - low response; response factor, versus phenanthrene-D10, less than 0.02
ND - not detected
ES - expected to be suitable for GC-MS analysis but not detected in this study.

(c) Substituted for the non-specific mixture, tricresyl phosphate.

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***J. E. Longbottom** is the EPA Project Officer (see below).*
*The complete report, entitled "GC-MS Suitability Testing of RCRA Appendix VIII and Michigan List Analytes," (Order No. PB 87-227 674/AS; Cost: $13.95, subject to change) will be available only from:*
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*U.S. Environmental Protection Agency*
*Cincinnati, OH 45268*

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