



## Project Summary

# Validation of SW-846 Methods 8010, 8015, and 8020

J.E. Gebhart, S.V. Lucas, S.J. Naber, A.M. Berry, T.H. Danison,  
and H.M. Burkholder

**A hierarchical approach is being implemented for the development and validation of analytical methods for the determination of the over 400 RCRA Appendix VIII and Michigan List compounds in wastes. The first phase of this approach involved testing GC/MS methods for the detection and measurement of these compounds. Next, semivolatile compounds determined to be amenable to GC/MS were used to evaluate the performance of SW-846 Method 3510. In the study described in the full report, volatile organic compounds determined to be amenable to GC/MS were used to evaluate the performance of SW-846 Method 5030.**

**The performance of Method 5030 was evaluated in conjunction with SW-846 Methods 8010, 8015, and 8020. In these studies, purge-trap-desorb sample introduction techniques were used for synthetic aqueous and solid samples, and direct liquid injection was used for synthetic nonaqueous liquid wastes. The results of these studies are presented, including purging efficiencies and estimated method detection limits for compounds in aqueous samples and method detection limits for compounds in nonaqueous liquid wastes.**

*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 specifies over 300 toxic organic compounds in its Appendix VIII to 40 CFR 261 listing that 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<sup>1</sup> by adding over 100 other organic compounds to give a total of over 400 organic constituents. Various gas chromatographic (GC) methods for determining Appendix VIII compounds in wastes are given in SW-846, *Test Methods for Evaluating Solid Wastes*<sup>2</sup>. In many cases, these methods are modifications of procedures cited under the Clean Water Act for determining some, but not all, of Appendix VIII and Michigan List compounds in wastewater. The EPA is currently attempting to validate the appropriate SW-846 analytical methods for as many of the 400 plus target compounds as possible. A hierarchical approach to these validation efforts is being pursued.

A schematic illustration of the hierarchical approach to the development and validation of analytical methods for the determination of over 400 organic compounds in wastes is presented in Figure 1. The first phase of this approach was conducted under Work Assignment 4 of EPA Contract Number 68-03-3224<sup>3</sup> and involved identifying volatile and semivolatile compounds that are amenable to GC separation and mass spectrometric (MS) detection. Next, the semivolatile compounds determined to be amenable to GC/MS were then used to evaluate the performance of SW-846 Method 3510<sup>4</sup>. This work focussed on the recovery from water and aqueous stability of the semivolatile compounds

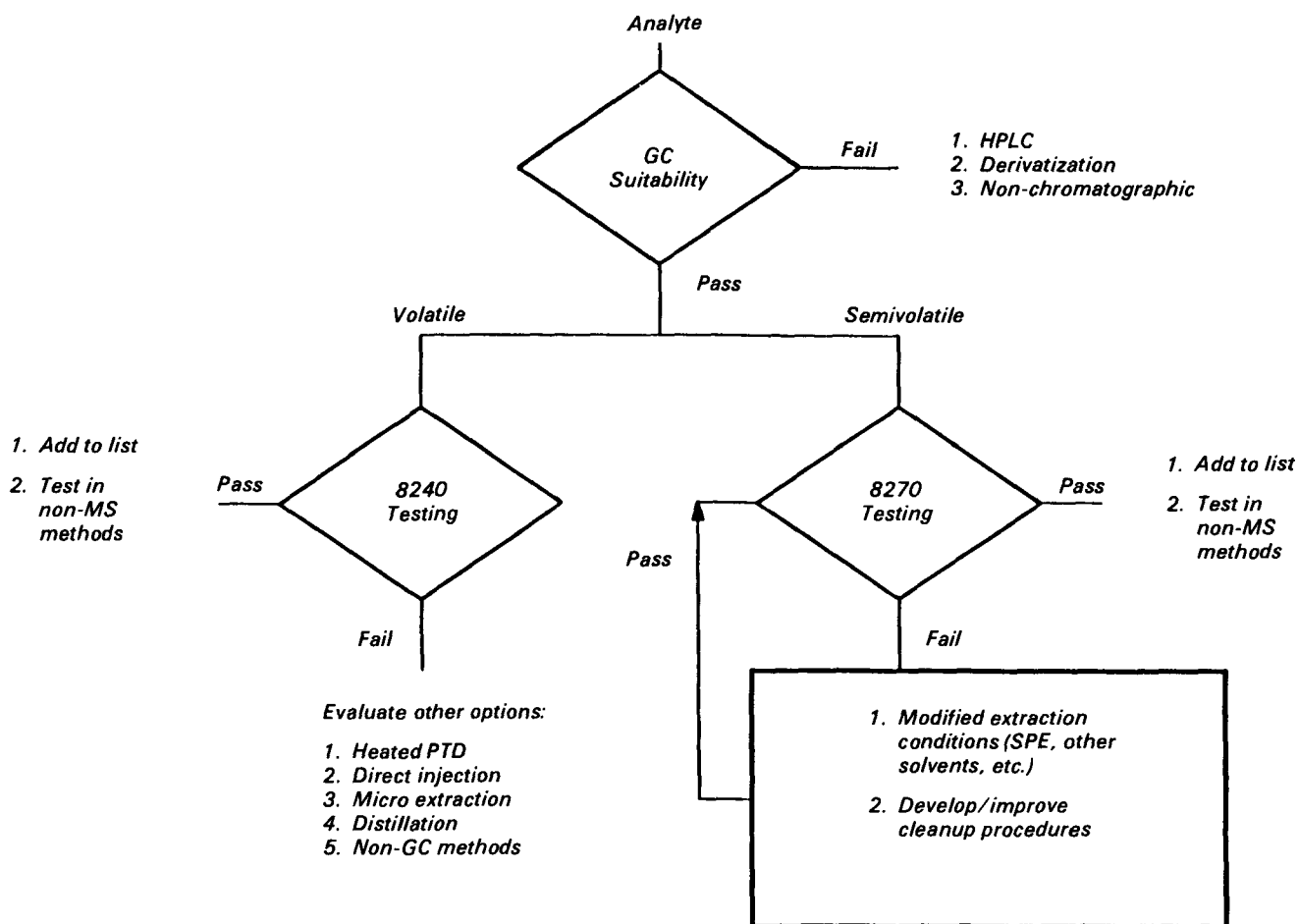


Figure 1. Hierarchical approach for analytical method development for organic RCRA analytes.

using standardized storage and extraction procedures. These experiments were conducted under Work Assignment 8 of EPA Contract Number 68-03-3224. In the study described in the full report, volatile compounds determined to be amenable to GC separation were used to evaluate SW-846 Method 8010, 8015, and 8020. Evaluating these methods was one of the major objectives of this Work Assignment. These experiments which comprised these evaluations and the results obtained are presented in the full report. Recommendations for further effort in the evaluation of methods for the determination of volatile organic compounds (VOCs) in waste samples are also provided in the report. The other major objective of this Work Assignment was to use the results of Methods 8010, 8015, and 8020 testing to formulate recommendations for including specific compounds in the scope of Method 5030 for the validation of Method 8240. These recommendations are made based on

the recovery and precision of the determination of these analytes using procedures specified in Methods 8010, 8015, and 8020.

Methods 8010, 8015, and 8020 provide packed-column GC conditions for the determination of certain VOCs. Waste samples are analyzed using these Methods in conjunction with purge-trap-desorb (PTD), Method 5030; direct liquid injection (DLI); or headspace sampling, Method 5020, sample introduction techniques. Temperature programs are used in the GC to separate organic compounds. Detection is achieved by halogen specific detector for Method 8010, a flame ionization detector for Method 8015, and a photoionization detector for Method 8020.

These Methods were evaluated using procedures described in the *Single Laboratory Method Validation Protocol (SLMVP)*<sup>5</sup> which was developed under Work Assignment 1 of EPA Contract Number 68-03-3224. While the SLMVP specifies six steps for full

method validation, only the first two steps, Instrumentation Range Determination and Preliminary Method Evaluation were used in these evaluations. This approach was taken because EPA anticipated that many laboratories would soon have the capability to conduct PTD analysis using capillary column GC. Consequently, full validation of packed column methods was not considered necessary or appropriate. Research results provided in the full report are intended to define the scope of the three packed-column methods and establish a basis for testing of capillary column based methods for the determination of VOCs in waste samples.

### Experimental Approach

Compounds initially considered for inclusion in the Methods 8010, 8015, and 8020 testing are listed in Table 1. Based on preliminary evaluations, a number of these analytes were excluded from these experiments because of poor purging efficiency, poor chromatographic

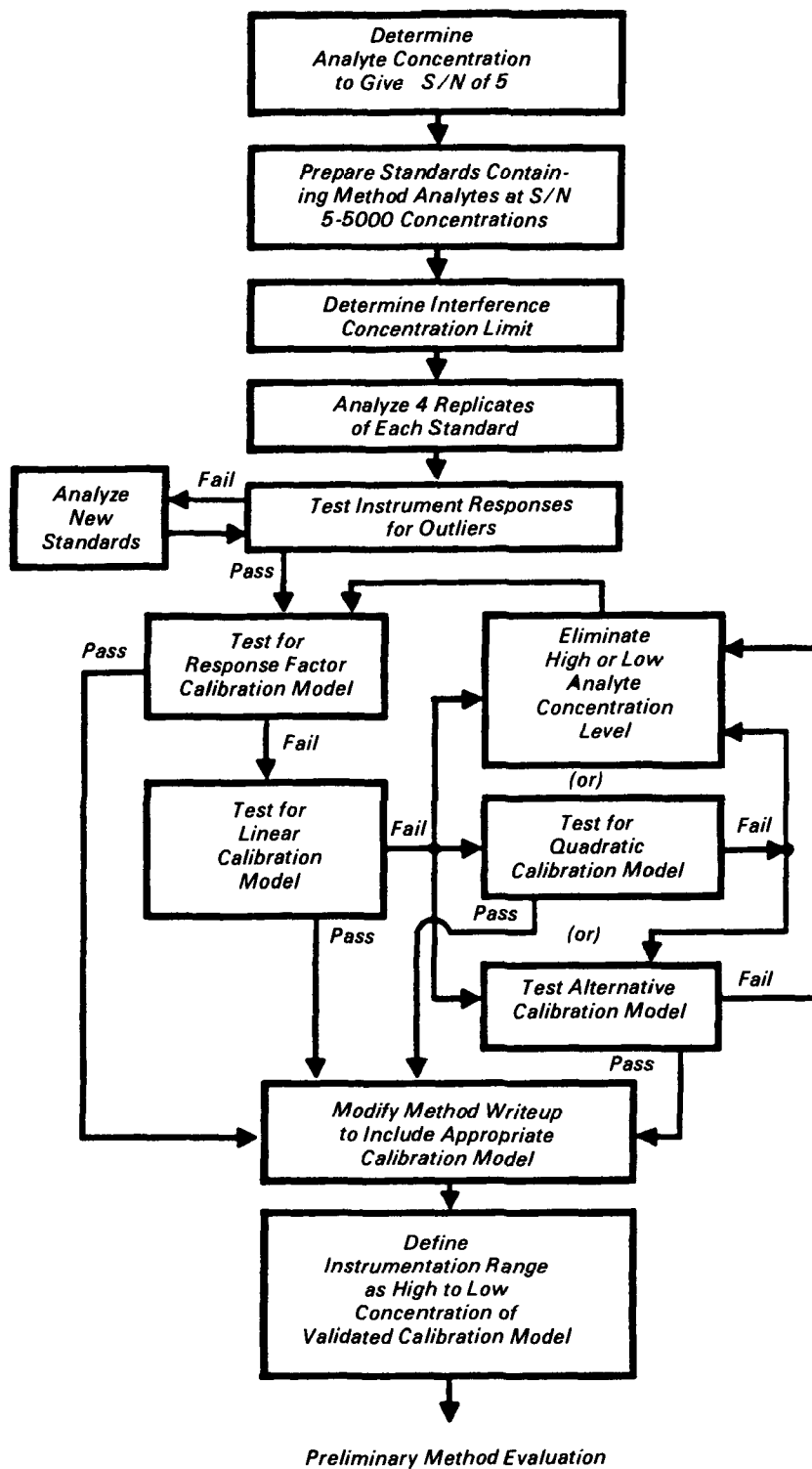


Figure 2. Instrumentation range determination.

behavior, or lack of pure standard material. These compounds are listed in Table 2 along with the reason for elimination from testing.

The first phase of method evaluation was the Instrumentation Range Determination step of the SLMVP. These experiments involved replicate analysis of aqueous calibration standards using the PTD sample introduction technique and of nonaqueous liquid calibration standards using the DLI sample introduction method. In these studies, at least four replicate standards were analyzed at each of seven concentration levels. The concentrations were selected to cover a three-orders-of-magnitude range. Results obtained in this validation step establish a basis for determining the test concentrations and the calibration function to be used in later steps of the validation. A flow diagram illustrating specific activities of the Instrumentation Range Determination step is shown in Figure 2.

The second phase of the method testing was the Preliminary Method Evaluation step of the SLMVP. These studies involved the analysis of eight replicates of synthetic aqueous and solid samples that had been fortified with the known amounts of the compounds of interest. In these experiments, only the PTD sample introduction technique was used. The synthetic aqueous samples consisted of reagent water which was spiked as a 500 mL batch, divided into 40 mL aliquots, and stored in Teflon-lined septum screw cap vials overnight at 4°C. The synthetic solid sample was composed of equal parts of Celite 503 and Kaolin. Two gram aliquots of this sample were spiked, mixed thoroughly, and stored overnight at 4°C. This step of the validation is conducted to determine if the method performs adequately for specified analytes before actual validation begins. This preliminary evaluation ensures that no major technical difficulties are inherent in the method, that reasonable results can be obtained for method analytes, and that the time, effort, and cost of a validation study will not be spent on an unsatisfactory method. A flow diagram illustrating specific activities of the Preliminary Method Evaluation step is shown in Figure 3.

Analyses of all standards and samples in these studies were conducted exactly according to the procedures presented in Method 5030. The chromatographic columns and conditions used for the analyses were those described in Method 8010, 8015, and 8020.

## Results and Discussion

A summary of the results obtained from the evaluations of Methods 8010, 8015, and 8020 is provided in Table 3 which presents the compounds for which each of these Methods was found to be suitable. These results are discussed below.

### Method 8010

A total of 53 compounds were originally considered for inclusion in the evaluations of Method 8010. Preliminary experiments were conducted to evaluate purging efficiencies and chromatographic behavior of these compounds. Based on these experiments a number of analytes were excluded from the PTD and/or the DLI portions of Method 8010 testing. Chloroacetaldehyde was excluded from all testing because a commercial source could not be identified. Bis (2-chloroisopropyl) ether was excluded from all experiments because the standard material obtained was not pure and another batch could not be obtained in time for use in these studies. Of the remaining 51 compounds, seven were excluded from the PTD portion of the experiments because of poor purging efficiencies. These compounds including bis (2-chloroethoxy) methane; bromoacetone; 2-chloroethanol; 2-chloroethyl vinyl ether; chloromethyl methyl ether; 1,3-dichloro-2-propanol; and epichlorohydrin were used in the DLI portion of the method performance testing. Four analytes including bis(2-chloroethyl) sulfide; chloral; 3-chloropropionitrile; and pentachloroethane were excluded from both the PTD and the DLI portions of these studies because of poor chromatographic behavior under the Method 8010 conditions. Chloroprene was excluded from the DLI experiments because of apparent decomposition in the injector. This analyte was included in the PTD portion of these studies in which this effect was not observed. Based on the results of these preliminary experiments, a total of 40 compounds, including 26 priority pollutant compounds, was included in the PTD portions of these studies. Forty-six compounds, including 27 priority pollutants were used in the DLI portion of the Method 8010 evaluations.

Based on the results of the Instrumentation Range Determination and the Preliminary Method Evaluation experiments, Method 8010 was determined to be suitable for the

determination of 36 of the 40 test compounds used in the PTD portion of these studies. The compounds for which the performance of this Method was considered unacceptable included methyl iodide, benzyl chloride, 4-chlorotoluene, and dichlorodifluoro-methane. For the determination of these analytes in aqueous and solid matrices, Method 8010 did not achieve the criteria for recovery and precision that were established with the priority pollutants which were used as reference compounds throughout these studies.

Based on the results of the Instrumentation Range Determination experiments, the performance of Method 8010 was considered to be acceptable for all 46 of the analytes used in the PTD portion of these studies.

### Method 8015

A total of 21 compounds were originally considered for inclusion in the evaluations of Method 8015. Due to the results of preliminary experiments, two compounds including acrylamide and hydroxypropionitrile were excluded from both the PTD and the DLI portions of these studies because of poor chromatographic behavior under the Method 8015 conditions. A number of compounds were excluded from the PTD experiments because of poor purging efficiencies. These analytes include acetonitrile; allyl chloride; carbonyl disulfide; 1,2,3,4-diepoxybutane; 1,4-dioxane; ethylene oxide; isobutane; malononitrile; methyl mercaptan; paraaldehyde; propargyl alcohol; propiolactone; and propionitrile. These compounds were included in the DLI experiments of Method 8015 testing. Based on the results of these preliminary experiments, six analytes were used in the PTD portion of Method 8015 testing and 19 compounds were used in the DLI experiments. None of these compounds were priority pollutants.

Based on the Instrumentation Range Determination and the Preliminary Method Evaluation experiments, Method 8015 was considered to perform acceptably for five of the six compounds used in the PTD portions of the testing. Methyl isobutyl ketone was eliminated from these experiments when the performance of the Method for this compound was not found to be sufficiently reproducible for reliable instrument calibration. Based on the Instrumentation Range Determination experiments, Method 8015 was

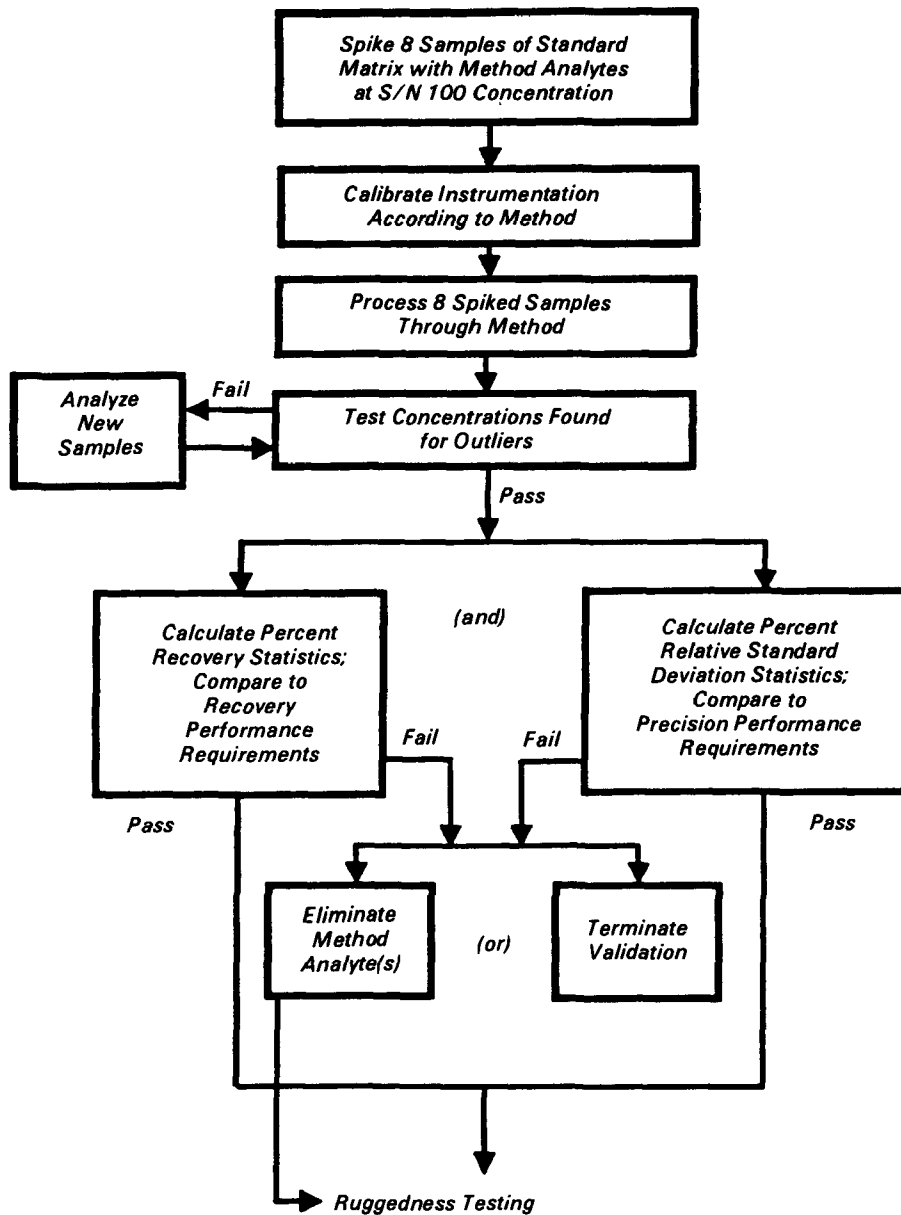


Figure 3. Preliminary method evaluation.

considered to be suitable for 16 of the 19 compounds used in the DLI portions of these studies. The performance of this Method appeared to be unsuitable for the determination of carbon disulfide, malononitrile, and  $\beta$ -propiolactone in nonaqueous liquid matrices primarily because of the very low response of the flame ionization detector used in this Method for these analytes.

### Method 8020

A total of 14 analytes were initially considered for inclusion in the evaluation of Method 8020. Based on the results of

preliminary experiments, pyridine and thiophenol were eliminated from both the PTD and the DLI portions of these studies due to poor chromatographic behavior under the conditions specified by Method 8020. In addition, 2-picoline was eliminated from the PTD experiments because of poor purging efficiency. This compound was included in the DLI studies. Data obtained during these preliminary experiments resulted in the use of 11 compounds in the PTD portion of Method 8020 testing and 12 analytes in the DLI studies. In each case, seven of the compounds used were priority pollutants.

Based on the results of the Instrumentation Range Determination and the Preliminary Method Evaluation steps of method testing, Method testing was considered to be suitable for the determination of all 11 compounds in aqueous and solid matrices. These experiments involved the use of the PTD sample introduction technique and the criteria for acceptable method performance was based on results obtained for the seven priority pollutant compounds. Method 8020 was considered to be suitable for the determination of all 12 of the analytes

used in the DLI portion of these experiments.

## Conclusions and Recommendations

Based on the studies described and the results presented in the full report, the following conclusions are drawn:

- Using the PTD sample introduction technique, Method 8015 was found to be suitable for the determination of five of the 21 test compounds in aqueous and solid samples. This Method, in combination with DLI sample introduction, was demonstrated to be successful for the determination of 19 of the 21 analytes in nonaqueous liquid samples.
- Method 8020 was determined to be suitable for the determination of 11 of the 14 test compounds in aqueous and solid samples using PTD sample introduction. Using DLI sample introduction, this Method was demonstrated to be successful in the determination of 12 of the 14 compounds in nonaqueous liquid samples.
- Poor purging efficiency and poor chromatographic behavior for a number of test compounds prevented Methods 8010, 8015, and 8020 from performing successfully for these analytes.

Table 1 lists the compounds for which these Methods were determined to be suitable based on the experiments conducted during these studies. Table 4 lists the compounds for which the performance of these Methods was found to be unacceptable. This table also provides a brief comment of the difficulties encountered with each of these compounds.

Based on the experiments described and the results presented in the full report, the following recommendations are made.

- Pending further method suitability testing, the compounds listed in Table 1 should be included in the scopes of Methods 8010, 8015, and 8020 as indicated.
- A total of 51 compounds was used to evaluate Method 8010. This Method was determined to be suitable for the determination of 36 of these analytes in aqueous and solid samples using the PTD sample introduction technique. When the DLI sample introduction technique was used, Method 8010 was found to be suitable for the determination of 46 of the test

compounds in nonaqueous liquid samples.

- At this time, compounds listed in Table 4 should be excluded from the scopes of Methods 8010, 8015, and 8020.
- Further method suitability testing should involve the use of capillary columns and should include those analytes excluded from this study on the basis of poor chromatographic behavior.
- Further evaluations of these Methods should include analysis of actual waste samples, rigorous determination of method detection limits for all analytes, and the conduct of the referee validation step of the SLMVP.
- Compounds listed in Table 5 have been determined to purge with acceptable efficiency and precision from aqueous samples. These compounds should be included in performance testing of SW-846 Method 8240.
- For future studies involving these and other methods for the determination of volatile compounds, more reliable procedures for the preparation of spiked aqueous and solid samples should be developed and implemented. Emphasis should be placed on minimizing analyte losses during the preparation of replicate samples.

## References

1. Federal Register, 49, No. 247, December 21, 1984, pp 49784-49793.
2. *Test Methods for Evaluating Solid Waste*, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, SW-846, Third Edition, November, 1986.
3. *GC-MS Suitability Testing*, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory - Cincinnati, EPA Contract Number 68-03-3224, Work Assignment 1-04.
4. *Screening of Semivolatile Organic Compounds for Extractability and Aqueous Stability by SW-846 Method 3510*, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory - Cincinnati, EPA Contract Number 68-03-3224, Work Assignment 2-08.
5. *Development of a Single Laboratory Method Validation Protocol*, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory - Cincinnati, EPA

Contract Number 68-03-3224, Work Assignment 1-01.

**Table 1. Compounds Considered for Inclusion in the Suitability Testing of Methods 8010, 8015, and 8020**

Compound	CAS Number	List <sup>(a)</sup>	Source
<b>Method 8010</b>			
Allyl chloride	107-05-1	8, 9, M	Aldrich Chemical Company
Benzyl chloride	100-44-7	8	Fisher Scientific Company
Bis(2-chloroethoxy)methane	111-91-1	8, 9	Pfaltz and Bauer, Inc.
Bis(2-chloroethyl)sulfide	505-60-2	8, M	Chem Services, Inc.
Bis(2-chloroisopropyl)ether	108-60-1	8	Chem Services, Inc.
Bromoacetone	598-31-2	8	Chem Services, Inc.
Bromobenzene	108-86-1	--	Fluka AG Chemical Company
Bromodichloromethane	75-27-4	PP, 8, 9	Aldrich Chemical Company
Bromoform	75-25-2	PP, 8, 9	Eastman Organic Chemical Products
Bromomethane	74-83-9	PP, 8, 9	Matheson Gas Products
Carbon tetrachloride	56-23-5	PP, 8, 9	Fluka AG Chemical Company
Chloroacetaldehyde	107-20-0	8	No commercial source
Chloral	75-87-6	8	Fisher Scientific Company
Chlorobenzene	106-90-7	PP, 8, 9	Matheson, Coleman, and Bell
Chloroethane	75-00-3	PP, 9	Chem Services, Inc.
2-Chloroethanol	107-07-3	M	Eastman Organic Chemical Products
Chloroform	67-66-3	PP, 8, 9	Burdick and Jackson Laboratories
1-Chlorohexane	544-10-5	--	Fluka AG Chemical Company
2-Chloroethyl vinyl ether	100-75-8	PP, 8	Aldrich Chemical Company
Chloromethane	74-87-3	PP, 8, 9	Matheson Gas Products
Chloromethyl methyl ether	107-30-2	8	Sigma Chemical Company
Chloroprene	126-99-8	8, 9, M	Alfa Products
3-Chloropropionitrile	542-76-7	8	Aldrich Chemical Company
4-Chlorotoluene	106-43-4	--	Chem Services, Inc.
Dibromochloromethane	124-48-1	PP, 9	Alfa Products
1,2-Dibromo-3-chloropropane	96-12-8	8, 9	Chem Services, Inc.
Dibromomethane	74-95-3	8	Analabs
1,2-Dichlorobenzene	95-50-1	PP, 8, 9	Aldrich Chemical Company
1,3-Dichlorobenzene	541-73-1	PP, 8, 9	Aldrich Chemical Company
1,4-Dichlorobenzene	106-46-7	PP, 8, 9	Aldrich Chemical Company
1,4-Dichloro-2-butene	764-41-0	8, 9	Aldrich Chemical Company
Dichlorodifluoromethane	75-71-8	8, 9	Matheson Gas Products
1,1-Dichloroethane	75-34-3	PP, 8, 9	Aldrich Chemical Company
1,2-Dichloroethane	107-06-2	PP, 8, 9	Burdick and Jackson Laboratories
1,1-Dichloroethylene	75-35-4	PP, 8, 9	Fluka AG Chemical Company
Trans-1,2-dichloroethylene	156-60-5	PP, 8, 9	Fluka AG Chemical Company
Dichloromethane	75-09-2	PP, 8, 9	Burdick and Jackson Laboratories
1,2-Dichloropropane	78-87-5	PP, 8, 9	Aldrich Chemical Company
1,3-Dichloro-2-propanol	96-23-1	8	Aldrich Chemical Company
Cis-1,3-dichloropropylene	10061-01-5	PP	Fluka AG Chemical Company
Epichlorohydrin	106-89-8	8	Aldrich Chemical Company
Ethylene dibromide	106-93-4	8	Fluka AG Chemical Company
Methyl iodide	74-88-4	8, 9	Aldrich Chemical Company
Pentachloroethane	76-01-7	8, 9	Aldrich Chemical Company
1,1,2,2-Tetrachloroethane	79-34-5	PP, 8, 9	J. T. Baker Chemical Company
1,1,1,2-Tetrachloroethane	630-20-6	8, 9	Aldrich Chemical Company
Tetrachloroethylene	127-18-4	PP, 8, 9	Aldrich Chemical Company
1,1,1-Trichloroethane	71-55-6	PP	Fisher Scientific Company
1,1,2-Trichloroethane	79-00-5	PP, 8, 9	Aldrich Chemical Company
Trichloroethylene	79-01-6	PP, 8, 9	Aldrich Chemical Company

(Continued)

**Table 1. (Continued)**

Compound	CAS Number	List(a)	Source
<b>Method 8010 (Continued)</b>			
Trichlorofluoromethane	75-69-4	PP, 8, 9	Aldrich Chemical Company
1,2,3-Trichloropropane	96-18-4	8, 9	Aldrich Chemical Company
Vinyl chloride	75-01-4	PP, 8, 9	Matheson Gas Products
<b>Method 8015</b>			
Acetonitrile	75-05-8	8	Burdick and Jackson Laboratories
Allyl alcohol	107-18-6	8	Aldrich Chemical Company
Acrylamide	79-06-1	8	Aldrich Chemical Company
Carbon disulfide	75-15-0	8, 9	Matheson, Coleman, and Bell
1,2,3,4-Diepoxybutane	1464-53-5	8	Sigma Chemical Company
Diethyl ether	60-29-7	--	Burdick and Jackson Laboratories
1,4-Dioxane	123 -91-1	8	Burdick and Jackson Laboratories
Ethylene oxide	75-21-8	8	Matheson Gas Products
Ethyl methacrylate	97-63-2	8, 9	Aldrich Chemical Company
2-Hydroxypropionitrile	78-97-7	M	Aldrich Chemical Company
Isobutanol	78-83-1	8	Aldrich Chemical Company
Malononitrile	109-77-3	8	Aldrich Chemical Company
Methacrylonitrile	126-98-7	8	Aldrich Chemical Company
Methyl ethyl ketone	78-93-3	8, 9	Burdick and Jackson Laboratories
Methyl isobutyl ketone	108-10-1	--	Aldrich Chemical Company
Methyl mercaptan	74-93-1	8	Matheson Gas Products
Methyl methacrylate	80-62-6	8, 9	Matheson Gas Products
Paraldehyde	123-63-7	8	Sigma Chemical Company
Propargyl alcohol	107-19-7	8	Aldrich Chemical Company
$\beta$ -propiolactone	57-57-8	M	Sigma Chemical Company
Propionitrile	107-12-0	8	Aldrich Chemical Company
<b>Method 8020</b>			
Benzene	71-43-2	PP, 8, 9	Burdick and Jackson Laboratories
Chlorobenzene	106-90-7	PP, 8, 9	Matheson, Coleman, and Bell
1,2-Dichlorobenzene	95-50-1	PP, 8, 9	Aldrich Chemical Company
1,3-Dichlorobenzene	541-73-1	PP, 8, 9	Aldrich Chemical Company
1,4-Dichlorobenzene	106-46-7	PP, 8, 9	Aldrich Chemical Company
Ethyl benzene	100-41-4	PP, 9	Poly Science Corporation
2-Picoline	109-06-8	8, 9	Aldrich Chemical Company
Pyridine	110-86-1	8, 9	Aldrich Chemical Company
Styrene	100-42-5	9, M	Chem Services, Inc.
Thiophenol	108-98-5	8	Aldrich Chemical Company
Toluene	108-88-3	PP, 8, 9	Burdick and Jackson Laboratories
o-Xylene	95-47-6	9	Burdick and Jackson Laboratories
m-Xylene	1477-55-0	9	Chem Services, Inc.
p-Xylene	106-42-3	9	Matheson, Coleman, and Bell

(a) PP = Priority Pollutant; 8 = Appendix VIII; 9 = Appendix IX; M = Michigan List; -- = not on any list.



**Table 2. Compounds Not Included in Evaluations of Methods 8010, 8015, and 8020**

<i>Compound</i>	<i>Reasons for Exclusion</i>	<i>Portion of Study From Which Compound Excluded</i>	
		<i>PTD</i>	<i>DLI</i>
<b>Method 8010</b>			
<i>Bis(2-chloroethoxy)methane</i>	<i>Poor purging efficiency</i>	X	
<i>Bis(2-chloroethyl)sulfide</i>	<i>Poor chromatographic behavior</i>	X	X
<i>Bis(2-chloroisopropyl)ether</i>	<i>Standard impure</i>	X	X
<i>Bromoacetone</i>	<i>Poor purging efficiency</i>	X	
<i>Chloroacetaldehyde</i>	<i>Standard not available</i>	X	X
<i>Chloral</i>	<i>Poor chromatographic behavior</i>	X	X
<i>2-Chloroethanol</i>	<i>Poor purging efficiency</i>	X	
<i>Chloroethyl vinyl ether</i>	<i>Poor purging efficiency</i>	X	
<i>Chloromethyl methyl ether</i>	<i>Poor purging efficiency</i>	X	
<i>Chloroprene</i>	<i>Poor chromatographic behavior</i>		X
<i>3-Chloropropionitrile</i>	<i>Poor chromatographic behavior</i>	X	X
<i>1,3-Dichloropropanol</i>	<i>Poor purging efficiency</i>	X	
<i>Epichlorohydrin</i>	<i>Poor purging efficiency</i>	X	
<i>Pentachloroethane</i>	<i>Poor chromatographic behavior</i>	X	X
<b>Method 8015</b>			
<i>Acetonitrile</i>	<i>Poor purging efficiency</i>	X	
<i>Allyl alcohol</i>	<i>Poor purging efficiency</i>	X	
<i>Acrylamide</i>	<i>Poor chromatographic behavior</i>	X	X
<i>Carbon disulfide</i>	<i>Poor purging efficiency</i>	X	
<i>1,4-Dioxane</i>	<i>Poor purging efficiency</i>	X	
<i>Ethyl oxide</i>	<i>Poor purging efficiency</i>	X	
<i>2-Hydroxypropionitrile</i>	<i>Poor chromatographic behavior</i>	X	X
<i>Isobutanol</i>	<i>Poor purging efficiency</i>	X	
<i>Malononitrile</i>	<i>Poor purging efficiency</i>	X	
<i>Methyl mercaptan</i>	<i>Poor purging efficiency</i>	X	
<i>Paraldehyde</i>	<i>Poor purging efficiency</i>	X	
<i>Propargyl alcohol</i>	<i>Poor purging efficiency</i>	X	
<i>β-Propiolactone</i>	<i>Poor purging efficiency</i>	X	
<i>Propionitrile</i>	<i>Poor purging efficiency</i>	X	
<b>Method 8020</b>			
<i>2-Picoline</i>	<i>Poor purging efficiency</i>	X	
<i>Pyridine</i>	<i>Poor chromatographic behavior</i>	X	X
<i>Thiophenol</i>	<i>Poor chromatographic behavior</i>	X	X

**Table 3. Compounds Recommended for Inclusion in the Scopes of Methods 8010, 8015, and 8020**

Compound	List(a)	Sample Matrix for Which Method Was Found to Be Suitable	
		Aqueous/Solids Sample Matrices(b)	Nonaqueous Sample Matrices(c)
<b>Method 8010</b>			
<i>Allyl chloride</i>	8,9,M	X	X
<i>Benzyl chloride</i>	8	(d)	X
<i>Bis(2-chloroethoxy)methane</i>	8,9	(e)	X
<i>Bromoacetone</i>	8	(e)	X
<i>Bromobenzene</i>	--	X	X
<i>Bromodichloromethane</i>	PP,8,9	X	X
<i>Bromoform</i>	PP,8,9	X	X
<i>Bromomethane</i>	PP,8,9	X	X
<i>Carbon tetrachloride</i>	PP,8,9	X	X
<i>Chlorobenzene</i>	PP,8,9	X	X
<i>Chloroethane</i>	PP,9	X	X
<i>2-Chloroethanol</i>	M	(e)	X
<i>Chloroform</i>	PP,8,9	X	X
<i>1-Chlorohexane</i>	--	X	X
<i>2-Chloroethyl vinyl ether</i>	PP,8	(e)	X
<i>Chloromethane</i>	PP,8,9	X	X
<i>Chloromethyl methyl ether</i>	8	(e)	X
<i>Chloroprene</i>	8,9,M	X	(f)
<i>4-Chlorotoluene</i>	--	(d)	X
<i>Dibromochloromethane</i>	PP,9	X	X
<i>1,2-Dibromo-3-chloropropane</i>	8,9	X	X
<i>Dibromomethane</i>	8	X	X
<i>1,2-Dichlorobenzene</i>	PP,8,9	X	X
<i>1,3-Dichlorobenzene</i>	PP,8,9	X	X
<i>1,4-Dichlorobenzene</i>	PP,8,9	X	X
<i>1,4-Dichloro-2-butene</i>	8,9	X	X
<i>Dichlorodifluoromethane</i>	8,9	(d)	X
<i>1,1-Dichloroethane</i>	PP,8,9	X	X
<i>1,2-Dichloroethane</i>	PP,8,9	X	X
<i>1,1-Dichloroethylene</i>	PP,8,9	X	X
<i>Trans-1,2-dichloroethylene</i>	PP,8,9	X	X
<i>Dichloromethane</i>	PP,8,9	X	X
<i>1,2-Dichloropropane</i>	PP,8,9	X	X
<i>1,3-Dichloro-2-propanol</i>	8	(e)	X
<i>Cis-1,3-dichloropropylene</i>	PP	X	X
<i>Epichlorhydrin</i>	8	(e)	X
<i>Ethylene dibromide</i>	8	X	X
<i>Methyl iodide</i>	8,9	(e)	X
<i>1,1,1,2-Tetrachloroethane</i>	PP,8,9	X	X
<i>1,1,1,2-Tetrachloroethane</i>	8,9	X	X

(Continued)

Table 3. (Continued)

Compound	List <sup>(a)</sup>	Sample Matrix for Which Method Was Found to Be Suitable	
		Aqueous/Solids Sample Matrices <sup>(b)</sup>	Nonaqueous Sample Matrices <sup>(c)</sup>
<b>Method 8010 (Continued)</b>			
Tetrachloroethylene	PP,8,9	X	X
1,1,1-Trichloroethane	PP	X	X
1,1,2-Trichloroethane	PP,8,9	X	X
Trichloroethylene	PP,8,9	X	X
Trichlorofluoromethane	PP,8,9	X	X
1,2,3-Trichloropropane	8,9	X	X
Vinyl chloride	PP,8,9	X	X
<b>Method 8015</b>			
Acetonitrile	8	(e)	X
Allyl alcohol	8	(e)	X
1,2,3,4-Diepoxybutane	8	(e)	X
Diethyl ether	--	X	X
1,4-Dioxane	8	(e)	X
Ethylene oxide	8	(e)	X
Ethyl methacrylate	8,9	X	X
Isobutanol	8	(e)	X
Methacrylonitrile	8	X	X
Methyl ethyl ketone	8,9	X	X
Methyl isobutyl ketone	--	(d)	X
Methyl mercaptan	8	(e)	X
Methyl methacrylate	8,9	X	X
Paraldehyde	8	(e)	X
$\beta$ -Propiolactone	M	(e)	X
Propionitrile	8	(e)	X
<b>Method 8020</b>			
Benzene	PP,8,9	X	X
Chlorobenzene	PP,8,9	X	X
1,2-Dichlorobenzene	PP,8,9	X	X
1,3-Dichlorobenzene	PP,8,9	X	X
1,4-Dichlorobenzene	PP,8,9	X	X
Ethyl benzene	PP,9	X	X
2-Picoline	8,9	(d)	X
Styrene	9,M	X	X
Toluene	PP,8,9	X	X
<i>o</i> -Xylene	9	X	X
<i>m</i> -Xylene	9	X	X
<i>p</i> -Xylene	9	X	X

(a) PP = Priority Pollutant; 8 = Appendix VIII; 9 = Appendix IX; M = Michigan List; -- = not on any list.

(b) Method testing with aqueous and solid samples involved the use of PTD sample introduction.

(c) Method testing with nonaqueous liquid samples involved the use of DLI sample introduction.

(d) Method determined to be unsuitable for determination of this compound in the sample matrix indicated.

(e) Compound not included in this portion of testing due to poor purging efficiency.

(f) Chloroprene not included in this portion of testing due to poor chromatographic behavior with DLI sample introduction under conditions specified in method. See text for discussion.

**Table 4. Compounds Recommended for Exclusion from the Scopes of Methods 8010, 8015, and 8020**

Compound	List <sup>(a)</sup>	Sample Matrix for Method was Found to be Unsuitable		Comments
		Aqueous Solid <sup>(b)</sup>	Nonaqueous Liquid <sup>(c)</sup>	
<b>Method 8010</b>				
Benzyl chloride	8	X	(d)	Method not suitable <sup>(f)</sup>
Bis(2-chloroethoxy)methane	8,9	X	(d)	Poor purging efficiency
Bis(2-chloroethyl)sulfide	8,M	X	X	Poor chromatographic behavior
Bis(2-chloroisopropyl)ether	8	X	X	Standard impure
Bromoacetone	8	X	(d)	Poor purging efficiency
Chloroacetaldehyde	8	X	X	Standard not available
Chloral	8	X	X	Poor chromatographic behavior
2-Chloroethanol	M	X	(d)	Poor purging efficiency
Chloroethyl vinyl ether	PP,8	X	(d)	Poor purging efficiency
Chloromethyl methyl ether	8	X	(d)	Poor purging efficiency
Chloroprene	8,9,M	(e)	X	Poor chromatographic behavior
3-Chloropropionitrile	8	X	X	Poor chromatographic behavior
Chlorotoluene	--	X	(d)	Method not suitable <sup>(f)</sup>
Dichlorodifluoromethane	8,9	X	(d)	Method not suitable <sup>(f)</sup>
1,3-Dichloropropanol	8	X	(d)	Poor purging efficiency
Epichlorohydrin	8	X	(d)	Poor purging efficiency
Methyl iodide	8,9	X	(d)	Method not suitable <sup>(f)</sup>
Pentachloroethane	8,9	X	X	Poor chromatographic behavior
<b>Method 8015</b>				
Acetonitrile	8	X	(d)	Poor purging efficiency
Allyl alcohol	8	X	(d)	Poor purging efficiency
Acrylamide	8	X	X	Poor chromatographic behavior
Carbon disulfide	8,9	X	(d)	Poor purging efficiency
1,4-dioxane	8	X	(d)	Poor purging efficiency
Ethyl oxide	8	X	(d)	Poor purging efficiency
2-hydroxypropionitrile	M	X	X	Poor chromatographic behavior
Isobutanol	8	X	(d)	Poor purging efficiency
Malononitrile	8	X	(d)	Poor purging efficiency
Methyl mercaptan	8	X	(d)	Poor purging efficiency
Paraldehyde	8	X	(d)	Poor purging efficiency
Propargyl alcohol	8	X	(d)	Poor purging efficiency
$\beta$ -Propiolactone	M	X	(d)	Poor purging efficiency
Propionitrile	8	X	(d)	Poor purging efficiency
<b>Method 8020</b>				
2-Picoline	8,9	X	(d)	Poor purging efficiency
Pyridine	8,9	X	X	Poor chromatographic behavior
Thiophenol	8	X	X	Poor chromatographic behavior

(a) PP = Priority Pollutant; 8 = Appendix VIII; 9 = Appendix IX; M = Michigan List; -- = not on any list.

(b) Method testing with aqueous and solid samples involved the use of PTD sample introduction.

(c) Method testing with nonaqueous liquids involved the use of DLI sample introduction.

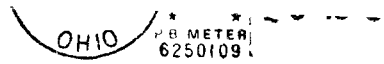
(d) Method suitable for this compound in nonaqueous liquids using DLI sample introduction.

(e) Method suitable for this compound in aqueous and solid samples using PTD sample introduction. See text for discussion.

(f) See Section 5 for detailed discussions.

**Table 5.** Compounds Recommended for Inclusion in Method 8240 Performance Testing

Compound	CAS Number	Retention Time (minutes)	Purging Efficiency (percent)	Estimated Detection Limit (µg/L)
Allyl chloride	107-05-1	10.17	88	0.272
Benzene	73-41-2	2.59	77	0.0554
Bromobenzene	108-86-1	29.05	81	0.278
Bromodichloromethane	75-27-4	15.44	107	0.138
Bromoform	75-25-2	21.12	65	0.951
Bromomethane	74-83-9	2.90	77	0.850
Carbon Tetrachloride	56-23-5	14.58	81	0.111
Chlorobenzene	106-90-7	25.49	51	0.701
Chloroethane	75-00-3	5.18	85	0.755
Chloroform	67-66-3	12.62	88	0.123
1-Chlorohexane	544-10-5	26.26	76	0.283
Chloromethane	74-87-3	1.40	73	0.258
Chloroprene	126-99-8	15.60	90	2.50
Dibromochloromethane	124-48-1	18.22	109	0.488
1,2-Dibromo-3- chloropropane	96-12-8	28.09	14	1.66
Dibromomethane	74-95-3	13.83	78	0.900
1,2-Dichlorobenzene	95-50-1	37.96	83	1.59
1,3-Dichlorobenzene	541-73-1	36.88	82	0.274
1,4-Dichlorobenzene	106-46-7	38.64	80	0.362
1,4-Dichloro-2-butene	764-41-0	23.45	30	0.488
1,1-Dichloroethane	75-34-3	11.21	86	0.164
1,2-Dichloroethane	107-06-2	13.14	103	0.129
1,1-Dichloroethylene	75-35-4	10.04	78	0.180
Trans-1,2-Dichloroethylene	156-60-5	11.97	107	0.897
Dichloromethane	75-09-2	7.56	86	2.93
1,2-Dichloropropane	78-87-5	16.69	90	0.300
Cis-1,3-Dichloropropylene	10061-01-5	17.00	100	0.317
Diethyl ether	60-29-7	11.24	90	0.013
Ethyl benzene	100-41-4	8.12	94	0.0957
Ethyl methacrylate	97-63-2	23.98	55	0.389
Ethylene dibromide	106-93-4	19.59	71	0.645
Methacrylonitrile	126-98-7	13.09	37	2.53
Methyl ethyl ketone	78-93-1	12.93	14	0.189
Methyl methacrylate	80-62-6	20.22	55	0.064
Styrene	100-42-5	11.60	86	0.118
1,1,2,2-Tetrachloroethane	79-34-5	23.12	102	0.140
1,1,1,2-Tetrachloroethane	630-20-6	21.10	85	0.117
Tetrachloroethylene	127-18-4	23.05	51	0.402
1,1,1-Trichloroethane	71-55-6	14.48	97	0.082
1,1,2-Trichloroethane	79-00-5	18.27	83	0.049
Trichloroethylene	79-01-6	17.40	85	0.124
Trichlorofluoromethane	75-69-4	9.26	82	0.191
1,2,3-Trichloropropane	96-18-4	22.95	50	0.346
Toluene	108-88-3	5.14	99	0.0867
Vinyl chloride	75-01-4	3.25	81	0.733
o-Xylene	95-47-6	10.54	92	0.0326
m-Xylene	1477-55-0	9.77	99	0.125
p-Xylene	106-42-3	9.18	98	0.0759



J. E. Gebhart, S.V. Lucas, S.J. Naber, A.M. Berry, T.H. Danison, and H.M. Burkholder are with Battelle Columbus Division, Columbus, OH 43201-2693.

**James E. Longbottom** is the EPA Project Officer (see below).

The complete report, entitled "Validation of SW-846 Methods 8010, 8015, and 8020," (Order No. PB88-161 567/AS; Cost: \$14.95, subject to change) will be available only from:

National Technical Information Service  
 5285 Port Royal Road  
 Springfield, VA 22161  
 Telephone: 703-487-4650

The EPA Project Officer can be contacted at:  
 Environmental Monitoring and Support Laboratory  
 U.S. Environmental Protection Agency  
 Cincinnati, OH 45268

United States  
 Environmental Protection  
 Agency

Center for Environmental Research  
 Information  
 Cincinnati OH 45268

BULK RATE  
 POSTAGE & FEES PAID  
 EPA  
 PERMIT No. G-35

Official Business  
 Penalty for Private Use \$300

EPA/600/S4-88/006

00Q0329 PS

- U S ENVIR PROTECTION AGENCY  
 REGION 5 LIBRARY  
 230 S DEARBORN STREET  
 CHICAGO IL 60604