AN UPDATE OF THE CURRENT STATUS OF THE RCRA METHODS DEVELOPMENT PROGRAM

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Introduction

This article updates our previous article in *Environmental Testing & Analysis* and will cover the activities of the OSW Methods Team in advancing the RCRA Methods Development Program over the past twelve months. We will discuss the new methods that we have either pursued or received since last year, and update some sections of last year's overview, as well as the status of the SW-846 Updates. We will provide an update on the progress in our major initiative to rewrite and update OSW's sampling guidance that we described in detail in the last article. We will also continue to provide a clarification of OSW's policy on flexibility in using and modifying SW-846 methods, and new developments on the status of its implementation of the Performance-Based Measurement System (PBMS).

As in previous years, OSW's primary focus has been on improved sample preparation and cost-effective screening methods, as well as keeping SW-846 current with state-of-the-art developments in analytical instrumentation and methodology. We have also made significant progress in updating the sampling issues in SW-846, with regard to both sampling strategy and sampling methods. This past year, we have made significant progress in using the Internet to provide useful and timely electronic information on the SW-846 Methods Program (www.epa.gov/SW-846).

Status of the Third Update

Update IIIA

The Freon Replacement Rule was published jointly as a Final Rule by the Office of Water (OW) and the Office of Solid Waste as a *Federal Register* Notice on May 14, 1999 (64 *FR* 26315). This rule replaces the use of Freon 113 with *n*-hexane in the two SW-846 methods (Method 9070A for aqueous matrices and Method 9071B for solid matrices) used to determine whether or not a waste being considered for delisting is an oily waste. Specifically the rule replaces Method 9070A with the OW Method 1664 for determining oil and grease in aqueous matrices while Method 9071B replaces Freon 113 with *n*-hexane as the solvent for determining whether a solid waste matrix exhibits the properties of an oily waste for the purpose of filing a delisting petition. See the Electronic Documents and Information section of this article for information on how to obtain electronic documents containing the Final Rule and Methods 9070A and 9071B.

Status of the Fourth Update

Update IVA

OSW published Update IVA as a Notice of Data Availability (NODA) in the *Federal Register* on May 8, 1998 (63 *FR* 25430). Details on the contents of the package are in last year's overview article and on the OSW Methods Team Homepage (which will be covered in greater

detail in the section on “Electronic Documents and Information”).

Update IVB

OSW is currently working on Update IVB, which includes primarily organic methods, to be published as another NODA. We expect to publish the Update IVB NODA in the *Federal Register* for public comment in either late 2000 or early 2001, depending on the availability of resources and the promulgation of the Methods Reinvention Rule. The Update could possibly contain more than 60 methods, approx. 30 new methods, approx. 30 revised methods, and 4 air methods to be incorporated by reference (see Table 1). However, resource limitations and the need for additional development time may cause some of the methods tentatively scheduled for Update IVB to be included in subsequent Updates. Draft Update IVB methods will be made available on the Methods Team Homepage on the Internet as they are completed. Announcements on the availability of these draft methods will be included in future issues of *Environmental Testing and Analysis* and on the Methods Team Homepage.

When and how the Update IV methods will be incorporated into SW-846, will depend on the progress of the Methods Reinvention Rule, which is nearing the proposal stage. This rule is intended to remove the unnecessary requirements to use SW-846 methods in the RCRA regulations and to allow OSW to issue Updates to SW-846 as guidance per the original intent of the document. We have included a more detailed discussion of this issue in the “PBMS Implementation” section of this article.

Method Flexibility and the Performance Based Measurement System (PBMS)

Historically, of all of EPA's regulatory program offices, OSW has allowed the most flexibility in methods selection and comes closest to implementing the PBMS for the RCRA Program. This allowance for methods flexibility and PBMS has been seriously misunderstood by both the regulators and the regulated community. We have attempted to clarify this allowable flexibility in Update III, and in a series of articles, presentations and training courses. We would like to briefly clarify OSW's policy on method flexibility and PBMS in this article. A more detailed discussion of these issues will be the topic of a future article.

Simply stated, OSW requires that the analyst must be able to demonstrate the ability to determine the analytes of concern in the matrix(ces) of concern at the [concentration] level of concern at an allowable uncertainty for any particular RCRA application. RCRA regulations basically specify "what" needs to be determined and leaves the "how" up to the analyst. "Any reliable analytical method" (58 *FR* 46040) may be used for this demonstration. In a few instances under the RCRA regulations, which are listed in the promulgation of Update II (60 *FR* 3089), the "reliable analytical method" must be included in SW-846. For all other RCRA applications, SW-846 methods are not required to be used, but are provided as guidance to the analyst. Therefore, OSW considers that SW-846 functions primarily as a guidance document setting forth acceptable, although not required, methods to be implemented by the user, as appropriate, in responding to RCRA-related sampling and analysis requirements.

Currently, the "Disclaimer" and "Preface and Overview" sections of SW-846 provide allowances for flexibility in methods selection and modification, and Section 2.1 of Chapter Two provides more specific guidance on how to implement this flexibility. The following language from the Preface and Overview should help clarify the intent of the SW-846 manual:

"The procedures described in this manual are meant to be comprehensive and detailed,

coupled with the realization that the problems encountered in sampling and analytical situations require a certain amount of flexibility. The solutions to these problems will depend, in part, on the skill, training, and experience of the analyst. For some situations, it is possible to use this manual in rote fashion. In other situations, it will require a combination of technical abilities, using the manual as guidance rather than in a step-by-step, word-by-word fashion. Although this puts an extra burden on the user, it is unavoidable because of the variety of sampling and analytical conditions found with hazardous wastes."

SW-846 methods are written so that they may be used as quantitative trace analytical methods to demonstrate that a waste "does not contain" constituents that require it to be managed as a hazardous waste. If particular RCRA applications do not require this rigor, looser analytical criteria may be applied, provided that they satisfy the data quality requirements for the particular application. Since data quality needs are project-specific in the RCRA Program, in order to successfully perform analyses it is necessary to address data quality issues prior to initiating any analyses. Good science dictates that, at a minimum, the following questions should be asked before beginning any analyses:

1) What is the purpose of this analysis? (Why are we doing this analysis?)

2) How (for what action) is the data generated from this analysis to be used?

3) What are the data quality needs for this project, i.e., how good does the data have to be to be useful for its intended purpose? (Including regulatory drivers, target analytes, matrices, concentration levels, statistical confidence levels, etc.)

The Agency once again emphasizes that the ultimate responsibility for producing reliable analytical results lies with the entity subject to the Federal, State, or local regulation. Thus, members of the regulated community are advised to refer to the information in Chapter Two and to consult with knowledgeable laboratory personnel when choosing the most appropriate suite of analytical methods for any particular RCRA application. The regulated community is further advised that the methods in SW-846 or from other sources need only be used for those specific analytes of concern that are subject to regulation or other monitoring requirements.

Many of the determinative methods in SW-846 include performance data. These data are only intended as guidance on the performance that may be achieved in typical matrices and may be used by the analyst to select the appropriate method for the intended application. These performance data are not intended to be used as absolute QC acceptance criteria. Rather, each laboratory should develop actual performance criteria as described in Chapter Two and elsewhere in the manual for their particular applications. All methods used, either existing SW-846 methods, modified SW-846 methods, or methods from other sources should be documented in appropriate Sampling and Analysis Plans.

In summary, the methods included in SW-846 provide guidance to the analyst and the regulated community in making judgments necessary to generate data that meet the data quality needs or objectives for the intended use of the results.

PBMS Implementation for the RCRA Program

As we have mentioned previously, OSW has been operating in the PBMS mode for the most part, since its inception in the early 1980s. Because of the wide variety of the types of

RCRA matrices, and many site-specific and project-specific applications, flexibility and the application of the PBMS approach in the use of analytical methods has been a necessity. As part of the overall Agency directive from the Agency's Deputy Administrator to implement PBMS in all of its programs, OSW coordinated preparation of the PBMS Implementation Plan for the entire Office of Solid Waste and Emergency Response (OSWER). The OSWER plan was completed and submitted to the Deputy Administrator in April, 1998. See the Electronic Documents and Information section of this article for instructions on how to obtain a copy on-line.

As stated in the OSWER PBMS Implementation Plan, OSW has initiated two major actions to remove some existing restrictions on method selection to improve method flexibility and to complete its move toward a more definitive PBMS approach to its regulatory policy. These two initiatives are 1) to remove the regulatory requirements for using SW-846 methods where they are not necessary, i.e, for analyses which do not require the use of method-defined parameters; and 2) to include data quality requirements directly in RCRA regulations. By method-defined parameters, we mean methods that define a regulation and must be followed exactly to insure regulatory compliance, such as Method 1311-Toxicity Characteristic Leaching Procedure or Method 9095-Paint Filter Test for Free Liquid Determination).

Currently, there are approximately 14 citations in the RCRA Subtitle C regulations which require the use of SW-846 methods for compliance. Several of these regulations require the use of SW-846 methods for determining method-defined parameters, i.e., where the method is itself the regulation. These are not subject to change nor are they applicable to a PBMS approach. However, the Agency is working to remove the unnecessarily rigid requirements in other regulations which currently require the use of SW-846 methods, such as filing delisting petitions and compliance with the Boiler, Incinerator and Furnace (BIF) Rules. The completion of this "Methods Reinvention Rule" will allow the Agency to put out SW-846 methods as guidance, with the exception of the previously-mentioned method-defined parameters. In anticipation of the expected promulgation of this rule, OSW is issuing the Update IV methods as guidance. OSW expects to propose the Methods Reinvention Rule in the *Federal Register* by the end of December, 1999.

In order to move towards a completely PBMS-based approach to regulations per Agency policy, OSW has begun to include data quality requirements directly in new RCRA regulations on a case-by-case basis. The first regulation promulgated under this PBMS approach was the Comparable Fuels Rule, which was published in the *Federal Register* in June, 1998. Others have followed including promulgation of the combustion or Maximum Achievable Control Technology (MACT) Rule and proposal of the Chlorinated Solvents and Dyes and Pigments Listings in 1999. The direct incorporation of data quality requirements into the regulations should greatly assist both the regulators and the regulated community in facilitating methods selection for specific applications.

Training is of prime importance to the implementation of PBMS. OSW is heavily involved with the Agency's Environmental Monitoring Management Council (EMMC) in helping to develop Agency-wide PBMS training modules for a variety of target audiences. In addition, for the past five years, OSW has been actively providing program-specific training for EPA Headquarters, EPA Regional, and State personnel involved in RCRA regulatory activities that involve sampling and analysis, as well as the general public. We have developed two training modules. OSW presents the first module, "Analytical Strategy for the RCRA Program: A Performance-Based Approach", annually, in July, at the Waste Testing and Quality Assurance Symposium (WTQA) in Washington, DC and at a variety of other sites around the country. OSW also presents the second training module, "Basic RCRA Analytical Methods", a module designed for non-chemists who must deal with analytical issues, at various sites around the country. OSW is working on training

modules on sampling and has completed a training course on the basics of chromatography in collaboration with partners from the Corps of Engineers and industry. The Basic Chromatography course was first presented at the 1999 WTQA Symposium.

Electronic Documents and Information

Since last year, the OSW Methods Team has continued its efforts to produce documents in electronic form so that the public can have easier access to them. The two types of electronic documents that the Methods Team currently maintains are the "Monitoring Science in the RCRA Program" Internet site (www.epa.gov/SW-846) and the SW-846 CD-ROM.

Internet

After last year's successful uploading of SW-846 methods through final Update III and proposed Update IVA, pre-release versions of sixteen methods from Draft Update IVB are now available on-line with more to follow in the next few months. The methods currently online on the Methods Team Website are identified by a double asterisk (**) after the method number in Table 1. The next group of methods scheduled for posting on the website are tagged in Table 1 with a triple asterisk (***) following the method number. These methods can be accessed on the Methods Team Homepage by logging on to the basic "Monitoring Science in the RCRA Program" address and clicking on the "In the News" prompt.

In addition, the following documents can also be accessed on-line on the "Monitoring Science in the RCRA Program" Website:

* the Office of Solid Waste and Emergency Response (OSWER) plan for implementing a Performance-Based Measurement System (PBMS) approach,
* the program information for the 1999 Waste Testing and Quality Assurance (WTQA) symposium,
* a description of new methods under development, including their use and application
* a link to the Waste Policy Institute (WPI) "On-line Environmental Methods Database" which will help people quickly obtain up-to-date information about method or technology performance,
* two OSW guidance documents, one on methods development and evaluation and the other on submittal of new immunoassay screening methods,
* a table of immunoassay test kits that have been validated by OSW,
* the new MICE e-mail address for leaving method-related questions and comments on technical issues regarding SW-846 (mice@cpmx.saic.com),
* new GPO pricing information for ordering hard copies of SW-846 ($367.00 per domestic subscription and $458.75 for foreign mailing),
* revised Methods 9070 and 9071A that replaced the CFC Freon 113 with n-hexane as the extraction solvent for determination of oil and grease, issued as Update IIIA and published in the Federal Register on May 14, 1999.

In the future, we plan to update the design of the site to make it more user friendly, including adding a task bar with buttons to facilitate navigation.

CD-ROM

Adding to its collection of SW-846 CD-ROMs, OSW put out draft Update IVA methods on

CD-ROM in March of this year. We also plan to produce a similar CD-ROM containing the Draft Update IVB Methods in the near future and eventually incorporate both updates into Version 3.0 of the main SW-846 CD-ROM.

The Update IVA disk contains only the draft IVA methods and includes all the text and figures found in the hard copies of the methods. Users can search by the chemical and common name of an analyte of interest, its CAS number, the number of the method, the analytical technique, or a variety of keywords. Users can also print the material including the diagrams and figures, and cut and paste the material in order to develop or update their own laboratory's standard operating procedures (SOPs). The system is compatible with both Windows and Macintosh operating systems and is available in both single user ($50) and LAN ($130) forms from NTIS.

Revisions to OSW's Sampling Guidance

The initial and perhaps most critical element in a program designed to evaluate the physical and chemical properties of a solid waste is the plan for sampling the waste. It is understandable that analytical studies, with their sophisticated instrumentation and high cost, are often perceived as the dominant element in a waste characterization program. Yet, despite that sophistication and high cost, analytical data generated by a scientifically defective sampling plan have limited utility, particularly in the case of regulatory proceedings. Chapters Nine and Ten of SW-846 address the development and implementation of a scientifically credible sampling plan for analyzing a solid waste. The principles to be considered in a performance-based sampling plan are delineated in these two chapters.

These chapters have been revised, expanded, and combined into a single guidance document that covers the great diversity of samples that are encountered in the RCRA program. The guidance examines the overall sampling plan including the data quality objective (DQO) process, the optimization of sampling design, and the data quality assessment (DQA) process. Specific sampling strategies and techniques, e.g., drums, waste piles, building debris, sampling tools, etc. are also covered in detail. These chapters were published in 1986 and have not been updated since, despite the enormous increase in knowledge on the subject. The revision includes the latest information on the applied statistics necessary for designing a scientifically credible sampling plan for determining the hazard potential of wastes.

Sampling and analysis of waste material is not an end in itself, rather it is a component of a larger regulatory framework in which data collection, data quality and data interpretation are linked. This interrelationship is the key to the understanding and development of useful sampling guidance for OSW. Environmental data collection for regulatory decision making and industry compliance must be of sufficient quality and quantity to support defensible decision making. This document imparts to our users a sound understanding of the DQO process, modern sampling theory, applied statistics, and how existing and developing regulations drive the technical requirements of sampling and analysis guidance.

Consistent with the goals enumerated by OMB Circular A-119 and Public Law #104-113, the "Technology Transfer and Improvements Act of 1995", which requires Federal agencies to use consensus standards whenever possible, OSW is working with ASTM to develop improved sampling standards and methods for waste characterization, site assessment, and environmental monitoring. These standards along with current EPA sampling, analysis, and quality assurance documents form a nucleus from which OSW developed updated sampling guidance for inclusion in SW-846.

Technical review of the guidance by EPA and ASTM was completed in the spring of 1999. A final draft was prepared in July. Since then, a number of internal "policy issues" have arisen which has prevented OSW from distributing the guidance to the regulated community. We hope to resolve these issues by the end of 1999 and to distribute the guidance electronically via our Methods Team website www.epa.gov/SW-846 shortly thereafter.

Organic Methods Update

The primary focus of the RCRA Organic Methods Development Program over the past two years has continued to be on the use of innovative technologies for improving sample preparation methods and for developing cost-effective screening procedures. In this section, we will only cover additional developments to methods discussed in the last article and the new additions.

We expect that the methods listed in Table 1 and described in both this section and the Inorganic Methods Update section of this article will be completed in time to be included in Update IVB. However, due either to budgetary constraints or unforeseen developmental difficulties, some of these methods may not be completed in time to make Update IV and will be included in subsequent Updates.

The convention used in SW-846 organic methods dictates that performance data for analytical methods be included in the determinative method with a reference back to the sample preparatory method used. The revised determinative methods in Update IVB (see Table 1) were revised based on the addition of performance data from the new or revised sample preparative methods, Methods 3546, 3535A and 3545A.

Advances in immunoassay technology will be represented with some new types of immunoassay techniques. Here is an update on the status of the immunoassay methods discussed in the last article. The validation of the two immunosensor methods developed at the Naval Research Laboratory for TNT- and RDX-type explosives have been completed for water matrices and are now completing their validations for soil matrices. This additional validation work to expand the scope of Methods 4655 (Flow Cell Detection) and 4656 (Fiberoptic Detection) is the reason for the delay in putting them up on the Methods Team Homepage as completed draft methods. Another new type of immunoassay method, Method 4425-Screening Extracts of Environmental Samples for Planar Organic Compounds (PAHs, PCBs, Dioxins/ Furans) by a Reporter Gene on a Human Cell Line, developed by Columbia Analytical Services of Carlsbad, CA, which utilizes a reporter gene to do a gross site screening for general compound classes of contaminants has been completed and has been placed on the Methods Team Homepage. Method 4025-Dioxin in Water and Soil by Immunoassay, developed by Cape Technologies, is in the final stages of validation and we expect to have it out within the next few months. A new immunoassay method, Method 4026, also developed by Cape Technologies for the analysis of coplanar PCBs is in its early stages of development and may be completed in time for inclusion in Update IVB.

Another new immunoassay technique, Method 4430-Dioxin TEQ by DELFIA Immunoassay, utilizes a cellular protein known as the "Ah receptor" and dissociation-enhancement lanthanide fluoroimmunoassay to measure the TEQ of dioxin and dioxin-like compounds in environmental samples. The interaction of dioxin-like compounds with Ah receptor represents the first step in a series of events that ultimately alters normal physiological processes. The method, developed by Hybrizime, is very sensitive to dioxin TEQ and unlike the other types of immunoassay methods used to determine small molecules in environmental media, is non-competitive in nature. We expect to have the validation completed and the method available on the Homepage within the next few months.

Method 3815-Screening Procedure for Determination of Use of High or Low-Level VOA Procedures, developed by Alan Hewitt of CRREL, has been completed and is on the Methods Team Homepage. It utilizes a portable photoionization detector (PID) to determine whether a high or low concentration of the principal site contaminants are present. Use of Method 3815 will help a sampler or analyst to determine whether a high- or low-concentration sample preparative procedure for Method 5035 (Closed-System Purge-and-Trap) or Method 5021 (Headspace) should be used. Knowledge of the site contaminants is necessary to effectively use this technique and Method 3815 must *not* be used to decide that a sample or site is free of VOC contamination, or to decide that samples need not be collected for quantitative VOC analysis.

Another new screening method that should be very useful for groundwater monitoring is Method 8535-Quick Method for Determination of TCE and Other Halogenated Aliphatic Hydrocarbons in Water. It is a semiquantitative colorimetric screening method for trichloroethylene and other halogenated aliphatics in water and was developed by Envirol, Inc. The method was approved by the SW-846 organic Methods Workgroup in July. Method 8535 is in the final stages of editing Workgroup comments and will be put up on the Methods Team Homepage in the next few months.

A new sample preparation method described in the last article that has now been completed and is on the Methods Team Homepage is Method 3546-Microwave Extraction. Method 3546 was developed by Environment Canada and is intended to be used as another rigorous rapid extraction technique analogous to PFE (Method 3545). Multilaboratory studies on analytes exhibiting the potential to undergo solvent-analyte interactions, i.e., phenols, organophosphorus pesticides, and phenoxyacid herbicides demonstrated that these analytes did not react under the extraction conditions of Method 3546.

Another FTIR method for measuring volatile contaminants in ambient air, Method 8455-Fourier-Transform Infrared (FTIR) Spectrometry for Volatile Organic Compounds (VOCs) in Ambient Air is being developed by the Department of Energy's Carlsbad, NM site for monitoring the air in the Waste Isolation Pilot Plant (WIPP) disposal facility for volatile organic contaminants. The method is still in the developmental stages and may or may not make it into Update IVB.

The last of the new methods that have materialized this year is Method 8135-Amines by GC . Dennis Gere of Hewlett Packard has taken the lead in the development and validation of this method. He is working with some academic colleagues to validate the RCRA-regulated target amines, which are a relatively difficult group of compounds to chromatograph well using existing general-purpose columns. There are now some very good specialty columns on the market for the chromatography of amines. The validation is being performed using these types of columns. If anyone out there is interested in participating in a Method 8135 multilaboratory validation study next year (2000), please let us know (email: lesnik.barry@epa.gov).

Most of the modifications to the determinative methods are due to addition of performance data from the expansion of scope of the two revised Update III extraction methods, Method 3535A-Solid Phase Extraction Disk Method (SPE) (Rev 1A) and Method 3545A-Pressurized Fluid Extraction (PFE) (Rev. 1A). Specifically the change to Method 3535A from the Update IVA version was to move the CRREL SPE sample prep method from Method 8095,while the change to Method 3545A was to add data on petroleum hydrocarbons generated by Dionex. However, four determinative methods had their target analyte lists expanded to include an expanded list of carbamates from an OSW carbamate project. These methods were Method 8141B-Organophosphorus Compounds by Gas Chromatography (Rev 1A), Method 8270D-Semivolatile Organic Compounds by GC/MS (Rev 4A), Method 8318A-N-Methyl Carbamates by High

Performance Liquid Chromatography (HPLC), and Method 8321B-Solvent-Extractable Non-volatile Compounds by High Performance Liquid Chromatography/ Thermospray/Mass Spectrometry or Ultraviolet (UV) Detection (Rev 2A). Method 8015C-Nonhalogenated Volatile Organics by GC/FID is included because of the addition of triethylamine as a target analyte from the carbamate project and additional TPH data from Method 3545A.

Although development of new organic methodology was relatively slow for a few years for a variety of reasons, it has rapidly picked up its pace over the past two years. OSW expects to continue to prioritize its focus on new methods development activities. OSW solicits your input on new technologies for environmental monitoring that can improve upon existing SW-846 methods or expand the scope of the manual. We are particularly interested in those technologies which are faster, more cost effective, and create less waste than existing methods, yet with comparable precision and accuracy. If you are interested in working with us on a new organic methods project, please contact us at the above e-mail address.

Inorganic Methods Update

The focus of the RCRA Inorganic Methods Development Program over the past year has been on the validation of characteristic methods for oxidizers and self-heating substances, on the development of screening procedures, and on the speciation of highly toxic inorganic analytes.

The addition of new inorganic and characteristic analytical technologies to SW-846 has progressed rapidly. Update IVB will contain five new methods and will revise three existing methods and one chapter (See Table 1). X-ray diffractometry is a proven technology introduced into EPA's environmental monitoring procedures for solid waste by Update IVB.

Method 1040-Test Method for Oxidizing Solids, and Method 1050-Test Method to Determine Substances Liable to Spontaneous Combustion, two of the new methods, give testing procedures which are useful guidance but not required methods for the characteristics. Both methods are adopted from the Department of Transportation regulations and the United Nations "Recommendations on the Transportation of Dangerous Goods." We provided them to assist the regulated community in classifying and managing their waste.

Method 9058-Determination of Perchlorate by Ion Chromatography, adds a new analyte to the IC technology which is becoming important to monitor at selected sites. Ammonium perchlorate, a key ingredient in solid rocket propellants, has recently been found in drinking water wells in regions of the U.S. where aerospace material, munitions and fireworks were developed, tested, or manufactured. To date, perchlorate has been found in ground and surface waters in California, Nevada, Utah, Texas, New York, Maryland, Arkansas and West Virginia. The presence of perchlorate in drinking water poses a considerable health risk, even at trace levels. Howard Okamoto and others at the California Department of Health Services developed an ion chromatographic (IC) method for the analysis of trace perchlorate in 1997. The technology was validated and extended to lower concentrations with better separation by Peter Jackson of Dionex Corporation. Method 9058 is applicable to a variety of environmental samples, including drinking water, ground and surface waters, soils and contaminated wastes.

Mercury continues to be a "hot" topic for OSW. Because of the widely varying toxicities of different chemical species of mercury, a method is needed to distinguish between mercury's different chemical forms. Method 3200-Determination of Mercury Species in Soil by Selective Solvent Extraction and Acid Digestion, is a separation and analysis scheme for the speciation of mercury (organic, inorganic, and elemental) that fills this void. Preliminary work was done in this

area by EPA's Office of Research and Development. Their work is being greatly expanded, validated, and drafted into a standard laboratory method by Dr. Skip Kingston and his students at Duquesne University. They are designing and validating an analysis scheme using selective solvent extraction and acid digestion to separate the different chemical species of mercury. Analysis of the separated fractions will be by Method 7473.

There are no standardized methods for mercury speciation. Development of Method 3200 will fill this void. Presently total mercury is measured regardless of its form. Thus, for site remediation and clean up we have no information on the species of mercury present - organic, inorganic, or elemental. Knowledge of the mercury species present at a site or in a waste will help us (1) trace its source, (2) identify its routes of transport, (3) determine its fate, and (4) guide us in the best methods of treatment or removal. The method will fill a monitoring niche for which there are currently no applicable methods published in the environmental area. Mercury is a common contaminant at many Superfund sites so the benefits of the technology should be immediate.

The chemical and physical properties of mercury are unique among the elements. Method 7473-Analysis of Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry exploits the unique chemistry of mercury to create a robust, highly specific and extremely sensitive technique for quantitative mercury analysis. Work by Helen Boylan of Duquesne University in Pittsburgh, PA under the direction of Dr. Skip Kingston continues to extend the capabilities of the method to other matrices such as coal. Due to the severe toxicity of mercury and to its pervasive pre-RCRA mismanagement, EPA envisions widespread use of this technology especially in field applications. Method 7473 is receiving much attention both inside and outside of the Agency.

Method 7473 has a dynamic range that spans approximately four orders of magnitude with an instrumental detection limit of 0.01 ng total mercury. The method is designed for the determination of mercury in solids, aqueous samples, and digested solutions in both the laboratory and field environments. The stability of the instrument is such that field and laboratory results are statistically identical. Integration of thermal decomposition sample preparation, mercury separation by amalgamation and thermal desorption from a gold collector, and quantitation by atomic absorption spectrophotometry into one instrument reduces the total analysis time of most samples to less than five minutes in either the laboratory or field setting.

Method 6300-X-ray Diffraction for the Determination of Crystalline Phases in Environmental Samples is a technology long overdue for inclusion in EPA's arsenal of environmental monitoring methods. X-ray Diffraction (XRD) is a well known highly developed technology for the identification of crystalline materials. It has been used for many years in industry and by the geological community. It has many unique features that make it an attractive technology for environmental monitoring. Knowing the exact molecular form of a waste is often more important than knowing the concentration of each individual element. For example, proving chromium is present in a waste as chromite ($FeCr_2O_4$) where the chromium is insoluble, trivalent, and is not bioavailable rather than as chromium trioxide (CrO_3) where the chromium is soluble, hexavalent, bioavailable, and highly toxic and the compound is corrosive and a powerful oxidizing agent, is an extremely important piece of information in determining the management of the waste. David Frank of EPA Region 10, Doug Kendall of NEIC, and Dave Bartenfelder of OSW have expertise in XRD and are working with us on the development of this method along with input from instrument vendors.

When dealing with unknown solid materials, the number one question is "What is it?". In environmental analysis of solids, no other technology currently exists that can answer this question

as rapidly, completely, or unambiguously as X-ray diffraction especially coupled with qualitative chemical information. Unknown solids are a common occurrence at Superfund sites. Thus, the CERCLA program should find widespread application and benefit from this technology. Knowing the composition of solid phases present at a site or in a waste will help us (1) trace its source, (2) identify its routes of transport, (3) determine its fate, and (4) guide us in the best methods of treatment or removal. This method will fill a monitoring niche for which there are currently no applicable methods published in the environmental area. The technology is well developed for geological applications and industrial quality control and analysis. XRD is a prime candidate to adapt for environmental analytical applications.

Method 6010C-Inductively Coupled Plasma-Atomic Emission Spectroscopy was revised to incorporate improved quality control criteria which is in conformance with the Update IVA revisions to the AA and GFAA methods. Joe Solsky with the U.S. Army Corp of Engineers provided extensive expert help and guidance to OSW in this work. The Corps' hands on experience with all of our major methods, gives them a unique perspective for improving the quality and clarity of SW-846 procedures for all required users of RCRA methods.

Method 9056A-Determination of Inorganic Anions by Ion Chromatography was revised to correct some inconsistences in QC guidance and to incorporate improvements in IC technology that have occurred over the six years since its initial proposal. Method 9210A- Potentiometric Determination of Nitrate in Aqueous Samples with Ion-Selective Electrode was revised to correct a problem with calibration standards. SW-846 Chapter Three was revised to add additional information to the definitions section.

Future Directions

General directions for the RCRA program include: continued development of cost effective screening methods, continued improvement of sample preparation methods, with particular emphasis on reduction of the use of hazardous substances (i.e., the so-called "green methods"), and continued development of cost effective analytical methods that can be used directly on site.

Specific directions for the RCRA methods development program are being charted by government regulatory needs, analytical technology development, and basic research coming from academia. New technologies that address our needs and that are well validated for the analytes and matrices of interest will find enthusiastic acceptance into OSW's method development program.

Some specific organic projects include continued development of Fourier Transform Infrared (FTIR) methods for headspace determination of volatile organics, continued development of new types of immunoassay products including quantitative techniques, immunosensors and affinity chromatography, development of new HPLC methods, and evaluation of capillary zone electrophoresis (CZE) for RCRA organic target analytes. Three specific projects which are funded in FY2000 are 1) development and validation of CZE methods for carbamates, phenols, phenoxyacid herbicides, and amines; 2) Evaluation of electrospray/ ATP interfaces for HPLC/MS, specifically targeting the Method 8321 analytes; and 3) development of new immunosensor methods for PCBs, PAHs, TCE and other target analytes. If anyone is interested in participating in these studies, please contact us by e-mail at the address above.

Future inorganic methods for SW-846 may incorporate the following emerging technologies and applied research: laser ablation spectrophotometry, immunoassay for other inorganic analytes, new reactive cyanide and sulfide procedures, new procedures for speciating

the various complex forms of cyanide, field-portable potentiometric stripping analyzers, chemical reactivity with water (evolution of ignitable gases), and laboratory-based x-ray fluorescence spectrometry.

OSW actively solicits your input on new technologies for environmental monitoring that can improve upon existing SW-846 methods or expand the scope of the manual. We are particularly interested in those technologies which are faster, more cost effective, and create less waste than existing methods, yet with comparable precision and accuracy.

TABLE 1: UPDATE IVB

NEW METHODS[a]

Method 0015:	Acetonitrile from Stack Emissions by GC
Method 1040: **	Test Method for Oxidizing Solids
Method 1050:**	Test Method to Determine Substances Liable to Spontaneous Combustion
Method 3200:	Determination of Mercury Species in Soil by Selective Solvent Extraction and Acid Digestion
Method 3511:	Microextraction Technique for Aqueous Matrices
Method 3546:**	Microwave Extraction
Method 3570:	Microextraction Technique for Soils and Solid Matrices
Method 3815:**	Screening Procedure for Determination of Use of High or Low-Level VOA Procedures
Method 4025:	Dioxin in Water and Soil by Immunoassay
Method 4026:	Coplanar PCBs in Water and Soil by Immunoassay
Method 4425:**	Screening Extracts of Environmental Samples for Planar Organic Compounds (PAHs, PCBs, Dioxins/ Furans) by a Reporter Gene on a Human Cell Line
Method 4430:	Dioxin TEQ by DELFIA Immunoassay
Method 4655:	Explosives Analysis in Soil and Water Using Environmental Immunosensors
Method 4656:	Explosives Analysis in Soil and Water Using Fiber-Optic Immunosensors
Method 6300:	X-ray Diffraction for the Determination of Crystalline Phases in Environmental Samples
Method 8085:**	Pesticides by GC/AED
Method 8095:**	Nitroaromatics and Nitramines by GC (CRREL)
Method 8135:	Amines by GC (Dennis Gere, Hewlett Packard)
Method 8261:**	Volatile and Semivolatile Organic Compounds by Closed System Vacuum Distillation with Cryogenic Condensation and Mass Spectrometric Determination
Method 8265:	Volatiles in Water by Direct Sampling Ion Trap Mass Spectrometry (ORNL)

Method 8450: Determination of Volatile Organic Compounds in Gaseous Samples by Fourier Transform Infrared (FT-IR) Spectroscopy (INEL)

Method 8455: Fourier-Transform Infrared (FTIR) Spectrometry for Volatile Organic Compounds (VOCs) in Ambient Air

Method 8510: Field Method for the Determination of RDX in Soil

Method 8530: Field Method for Determination of THMs in Water

Method 8535: Quick Method for Determination of TCE and Other Halogenated Aliphatic Hydrocarbons in Water

Method 8540: Pentachlorophenol (PCP) in Soil and Water by UV-Induced Colorimetry

Method 9058:*** Determination of Perchlorate by Ion Chromatography

[a] Absence of a suffix following the Method No. indicates Revision 0

** Completed draft methods on OSW Methods Team Homepage (Click on "In the News")

*** Next group of methods to be posted on Homepage (Click on "In the News")

REVISED METHODS[b]

Chapter 3: Revise Definitions

Method 3500C:** Organic Extraction and Sample Preparation

Method 3535A:** Solid Phase Extraction Disk Method (SPE) (Rev 1A),

Method 3545A: Pressurized Fluid Extraction (PFE) (Rev. 1A)

Method 3550C:** Ultrasonic Extraction

Method 3620C:** Florisil Cleanup

Method 5000A: Sample Preparation for Volatile Organic Compounds

Method 5021A: Volatile Organic Compounds in Solid Matrices Using Automated Static Headspace Apparatus

Method 5030C: Volatile Organic Compounds in Aqueous Matrices by the Purge and Trap Procedure

Method 5032A: Volatile Organic Compounds by Vacuum Distillation

Method 5035A: Volatile Organic Compounds in Solid Matrices by the Purge-and-Trap Procedure

Method 6010C:*** Inductively Coupled Plasma-Atomic Emission Spectroscopy

Method 8000C: Chromatography

Method 8015C: Nonhalogenated Volatile Organics by GC/FID

Method 8021C: Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors

Method 8041A: Phenols by Gas Chromatography

Method 8081B:** Organochlorine Pesticides by Gas Chromatography

Method 8082B:** Polychlorinated Biphenyls (PCBs) by Gas Chromatography

Method 8100A: Polynuclear Aromatic Hydrocarbons by Gas Chromatography

Method 8141B:** Organophosphorus Compounds by Gas Chromatography: Capillary Column Technique (Rev. 2A)

Method 8151B: Chlorinated Herbicides by Gas Chromatography: Capillary Column Technique

Method 8260C: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Method 8270D: Semivolatile Organic Compounds by GC/MS: Capillary Technique (Rev. 4A),

Method 8290A: Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) (Rev. 1A)

Method 8310A: Polynuclear Aromatic Hydrocarbons by HPLC

Method 8318A:** N-Methyl Carbamates by High Performance Liquid Chromatography (HPLC)

Method 8321B: Solvent-Extractable Non-volatile Compounds by High Performance Liquid Chromatography/Thermospray/Mass Spectrometry or Ultraviolet (UV) Detection (Rev 2A)

Method 9056A:*** Determination of Inorganic Anions by Ion Chromatography

Method 9210A:*** Potentiometric Determination of Nitrate in Aqueous Samples with Ion-Selective Electrode

[b] Suffixes following the Method No. indicate that a method has been revised and reissued:

A indicates Revision 1
B indicates Revision 2

C indicates Revision 3, etc.

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*** Next group of methods to be posted on Homepage (Click on “In the News”)

METHODS INCORPORATED BY REFERENCE

Method 25D:	Determination of the Volatile Organic Content of Waste Samples (40 CFR 60, Appendix A)
Method 25E:	Determination of Vapor Phase Organic Concentration in Waste Samples (40 CFR 60, Appendix A)
Method 207-1:	Sampling Method for Isocyanates
Method 207-2:	Analysis for Isocyanates by High Performance Liquid Chromatography (HPLC)