ENVIRONMENTAL PROTECTION AGENCY

[EPA-HQ-RCRA-2012-0072; FRL-9901-86-OSWER]

Waste Management System; Testing and Monitoring Activities; Update V of SW-846

AGENCY: Environmental Protection Agency (EPA).

ACTION: Notice.

SUMMARY: The Environmental Protection Agency (EPA or the Agency) is providing notice of the availability of "Update V" to the Third Edition of EPA publication SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods." Update V contains 23 new and revised analytical methods that the Agency has evaluated, and determined to be appropriate and which may be used for monitoring or complying with the Resource Conservation and Recovery Act (RCRA) hazardous and non-hazardous waste regulations. Because the analytical methods contained in Update V are not required by the RCRA hazardous waste regulations, EPA is issuing this update as guidance. In addition, the Agency is also taking comment on revisions to Chapters One through Five of EPA publication SW-846, an ORCR Policy Statement, and other guidance. The Agency is seeking public comment on Update V, and after consideration of the public comments, will place these new and revised methods, guidance, and chapters in the SW-846 methods compendium.

DATES: Comments must be received on or before **[INSERT DATE 90 DAYS AFTER PUBLICATION IN THE FEDERAL REGISTER**].

ADDRESSES: Submit your comments, identified by Docket ID No. **EPA-HQ-RCRA-2012-0072**, by one of the following methods:

- www.regulations.gov: Follow the on-line instructions for submitting comments.

- Email: RCRA-docket@epa.gov, Attention Docket ID No. **EPA-HQ-RCRA-2012-0072**.
- Fax: Fax comments to: 202-566-9744, Attention Docket ID No. **EPA-HQ-RCRA-2012-0072**.
- Mail: Send comments to: OSWER Docket, EPA Docket Center, Mail Code 28221T, Environmental Protection Agency, 1200 Pennsylvania Avenue N.W., Washington D.C. 20460, Attention Docket ID No. **EPA-HQ-RCRA-2012-0072**. Please include two copies of your comments.
- Hand Delivery: Deliver two copies of your comments to: Environmental Protection Agency, EPA Docket Center, Room 3334, 1301 Constitution Avenue, N.W., Washington D.C., Attention Docket ID No. **EPA-HQ-RCRA-2012-0072**. Such deliveries are only accepted during the docket's normal hours of operation and special arrangements should be made for deliveries of boxed information.

Instructions: Direct your comments to **EPA-HQ-RCRA-2012-0072**. EPA's policy is that all comments received will be included in the public docket without change and may be made available online at www.regulations.gov, including any personal information provided, unless the comment includes information claimed to be Confidential Business Information (CBI) or other information whose disclosure is restricted by statute. Do not submit information that you consider to be CBI or otherwise protected through www.regulations.gov or e-mail. The www.regulations.gov website is an "anonymous access" system, which means EPA will not know your identity or contact information unless you provide it in the body of your comment. If you send an e-mail comment directly to EPA without going through www.regulations.gov, your e-mail address will be automatically captured and included as part of the comment that is placed in the public docket and made available on the Internet. If you submit an electronic comment,

EPA recommends that you include your name and other contact information in the body of your comment and with any disk or CD-ROM you submit. If EPA cannot read your comment due to technical difficulties and cannot contact you for clarification, EPA may not be able to consider your comment. Electronic files should avoid the use of special characters, any form of encryption, and be free of any defects or viruses. For additional information about EPA's public docket, visit the EPA Docket Center homepage at http://www.epa.gov/epahome/dockets.htm. *Docket:* All documents in the docket are listed in the www.regulations.gov index. Although listed in the index, some information is not publicly available, e.g., CBI or other information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, will be publicly available only in hard copy. Publicly available docket materials are available either electronically in www.regulations.gov or in hard copy at the OSWER Docket, EPA/DC, EPA West, Room 3334, 1301 Constitution Ave., N.W., Washington DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is (202) 566-1744, and the telephone number for the OSWER Docket is (202) 566-0270.

FOR FURTHER INFORMATION CONTACT: Kim Kirkland, Materials Recovery and Waste Management Division, Office of Resource Conservation and Recovery, Office of Solid Waste and Emergency Response (5304P), Environmental Protection Agency, 1200 Pennsylvania Ave., NW, Washington, DC 20460; telephone number: 703-308-8855; fax number: 703-308-0522; email address: kirkland.kim@epa.gov.

SUPPLEMENTARY INFORMATION:

I. General Information

A. *Does this Action Apply to Me?*

This notice is directed to the public in general. It may, however, be of particular interest to you if you conduct waste sampling and analysis for RCRA-related activities. This might include any entity that generates, treats, stores, or disposes of hazardous or nonhazardous solid waste and is subject to RCRA subtitle C or D sampling and analysis requirements, and might also include any laboratory that conducts waste sampling and analyses for such entities.

B. *What Should I Consider as I Prepare My Comments for EPA?*

1. *Submitting CBI.* Do not submit this information to EPA through www.regulations.gov or e-mail. Clearly mark the part or all of the information that you claim to be CBI. For CBI information on a disk or CD ROM that you mail to EPA, mark the outside of the disk or CD ROM as CBI and then identify electronically within the disk or CD ROM the specific information that is claimed as CBI. In addition to one complete version of the comment that includes information claimed as CBI, a copy of the comment that does not contain the information claimed as CBI must be submitted for inclusion in the public docket. Information so marked will not be disclosed except in accordance with the procedures set forth in 40 CFR part 2.

2. *Tips for Preparing Your Comments.* When submitting comments, remember to:

- Identify the rulemaking by docket number and other identifying information (subject heading, Federal Register date and page number).
- Follow directions - The agency may ask you to respond to specific questions or organize comments by referencing a Code of Federal Regulations (CFR) part or section number.
- Explain why you agree or disagree; suggest alternatives and substitute language for your requested changes.

- Describe any assumptions and provide any technical information and/or data that you used.
- If you estimate potential costs or burdens, explain how you arrived at your estimate in sufficient detail to allow for it to be reproduced.
- Provide specific examples to illustrate your concerns, and suggest alternatives.
- Explain your views as clearly as possible, avoiding the use of profanity or personal threats.
- Make sure to submit your comments by the comment period deadline identified.

C. *How Can I Get Copies of Update V and the Third Edition of SW-846 as amended by its Final Updates?*

Update V is available in the RCRA docket and the final version will be available on-line after all comments have been addressed. The Third Edition of SW-846, as amended by Final Updates I, II, IIA, IIB, III, IIIA, IIIB, IVA, and IVB, is available in portable document format (PDF) on EPA's Office of Resource Conservation and Recovery (ORCR) web page at: http://www.epa.gov/SW-846.

D. *How Is the Rest of This Notice Organized?*

The rest of this Notice includes the following sections:

II. What is the Subject and Purpose of this Notice?

The Agency is announcing the availability of and inviting public comment on Update V to "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, " EPA Publication SW-846. Update V of SW-846 contains analytical methods that the Agency has evaluated, and/or revised and determined to be appropriate and may be used for monitoring or complying with the RCRA hazardous waste regulations. Because the analytical methods contained in Update V are not required by the RCRA hazardous waste regulations, EPA is issuing this update as guidance. This guidance does not add or change the RCRA regulations, and does not have any impact on existing rulemakings associated with the RCRA program. To date, the Agency has finalized Updates I, II, IIA, IIB, III, IIIA, IIIB, IVA, and IVB to the SW-846 manual, which can be found on the EPA's ORCR web page at: http://www.epa.gov/SW-846.

III. Why Is the Agency Releasing Update V to SW-846?

The Agency revises the content of SW-846 over time as new information and data become available. We continually review advances in analytical instrumentation and techniques and periodically incorporate such advances into SW-846 as method updates by adding new methods to the manual, and replacing existing methods with revised versions of the same method. These updates improve analytical method performance and cost effectiveness. Since the publication of the Methods Innovation Rule (MIR) (70 FR 34537, June 14, 2005), the Agency no longer needs to use a rulemaking process for publication of an update to SW-846, as

long as the update does not contain a method required by the RCRA regulations (e.g., Method-Defined Parameter (MDP), such as the Toxicity Characteristic Leaching Procedure (TCLP) (Method 1311)), see 40 CFR 260.11. The Agency instead can make an SW-846 update available to the public more efficiently through a Federal Register notice announcing its availability and inviting public comment on the update.

In addition, the MIR allows flexibility in method selection and use for meeting the analytical needs of the RCRA program, with the exception of those methods specifically required by the RCRA regulations. This approach is consistent with the Agency's commitment to fully implement a performance-based measurement system (PBMS), whereby the analytical focus is on measurement objectives and performance rather than specific measurement technologies. Furthermore, the Agency's PBMS approach has evolved resulting in the Agency adopting the new "Flexible Approaches to Environmental Measurement – The Evolution of the Performance Approach" as developed by the Forum on Environmental Measurements (FEM) at the direction of EPA's Science Policy Council (i.e., now the Science and Technology Policy Council (STPC)). One of the main goals of the Performance Approach is to increase flexibility in choosing sampling and analytical approaches to meet regulatory requirements for measurements. For more information on the Performance Approach, see: http://www.epa.gov/fem/approach.htm.

In using the SW-846 methods, the regulated entity need only demonstrate that an analytical method generates data that meet the project-specific data quality objectives (DQOs) and performance acceptance criteria. The Agency finds this flexible approach to be particularly useful, and sufficient in most cases, during the characterization of the complex matrices of RCRA-related wastes. Thus, a method user can modify an SW-846 method (provided it is not one specifically required by regulation, e.g., 40 CFR 260.11), in order to best meet a waste

matrix-specific analytical need, as long as the modifications meet the project-specific DQOs and performance acceptance criteria. The public should note that in some cases the method established certain requirements (e.g., conducting a calibration curve, using specific reagents, analyzing a Quality Control (QC) check sample to demonstrate precision and accuracy). While these standard principles are not regulatory requirements, they are necessary to yield data of acceptable quality as intended and are called for by sound science. (The public can obtain more information about the MIR and PBMS at the Agency's website dedicated to SW-846 and the testing of RCRA-regulated wastes: http://www.epa.gov/SW-846.)

The subject of today's notice, Update V to SW-846, contains 23 new and revised analytical methods and revises Chapters One through Five of SW-846. After the comment period, and based on the Agency's evaluation of the comments received, the new and revised methods and revised chapters will be formally included in the SW-846 methods compendium. Most of the Update V methods previously resided under the heading "New Methods" at EPA's SW-846 website as either revised versions of existing SW-846 methods or as new methods that the Agency planned to add to SW-846. Although these methods were not yet part of an official update to any edition of the SW-846 manual at the time of their posting on the website, the Agency wanted to make these Agency-evaluated methods available for use and comment as soon as possible. The Agency believed that public access to these new and revised methods, for guidance purposes, would assure that reliable and innovative methods are provided to the regulated community in a timely and cost-effective manner. Therefore, these methods could be used for any RCRA applications, other than one specifically required by regulation, for which their performance could be demonstrated to be appropriate and meet project-specific DQOs, and

thus be consistent with implementation and promotion of a flexible and performance-based approach to RCRA-related analyses.

The Agency is also responding to concerns expressed by the Environmental Laboratory Advisory Board (ELAB), a Federal Advisory Committee Act (FACA) committee that advises the Agency on measurement, monitoring, and laboratory science issues, who contacted EPA's FEM with several issues regarding the use of SW-846. The ELAB specifically contacted EPA regarding which version of a revised method is recommended. Historically, as noted above, the Agency has posted new and revised methods on the SW-846 website under the "New Test Methods Online" (at: http://www.epa.gov/epawaste/hazard/testmethods/sw846/new_meth.htm), for use by the laboratory community, the States, and the regulated community pending publication of these methods in the Federal Register. The Agency was subsequently contacted by the ELAB, who identified several concerns regarding the process for updating and posting updates on the "New Test Methods Online" link on the SW-846 website.

ELAB requested that EPA clarify those issues that caused some confusion with some entities of the user community. Specifically, confusion existed when a method had multiple versions available on the web. For example, Method 8000C, on the "New Test Methods Online" link has quality control (QC) guidelines that differ from Method 8000B (the official version) in the SW-846 compendium. The public was confused by the difference in QC guidelines in the two available versions of the method. The Agency subsequently decided that the revisions to Method 8000C were more significant than those previously posted, and has decided to replace Method 8000C with Method 8000D, and is issuing Method 8000D as part of Update V.

In response to ELAB's concerns, ORCR prepared a Policy Statement that identifies the status of methods (e.g., validated methods, final methods, etc.), and provides the rationale for

identifying when changes to methods are significant, through a letter designation and by noting the date the method was revised by ORCR. For more information on the ORCR Policy Statement, see section IV of this Notice.

Finally, the Agency is requesting public comment on the Update V methods and the other relevant updated materials presented in this Notice for inclusion in the SW-846 manual (i.e., Table of Contents and Chapters One through Five). See the ADDRESSES: section of this notice for the procedure for submitting comments. The Agency will consider public comments submitted on or before the comment period deadline and subsequently finalize Update V as an official part of SW-846. In addition, the EPA SW-846 web site contains an updated version of the "Method Status Table for SW-846," which identifies the update history for each document in SW-846.

The Agency strongly recommends the use of the latest version of an SW-846 method, especially for new analyte monitoring situations. The Agency, however, is not imposing restrictions on the use of earlier versions of non-required SW-846 methods or precluding the use of previous guidance, if such use is appropriate. For example, earlier versions of an SW-846 method may be more appropriate for regulatory purposes (e.g., for compliance with an existing permit or consent decree), or when new method versions may be more costly than necessary for meeting project-specific objectives. In the future, the Agency plans to make electronic copies of earlier versions of SW-846 methods available through a separate hyperlink from the SW-846 website.

The Agency hopes that the posting of this information on the website for immediate public access will mitigate any remaining confusion regarding the use of SW-846 methods. In addition, the public can also access the Methods Information Communication Exchange (MICE)

for answers to their questions or concerns regarding SW-846 methods. MICE can be accessed by phone at (703) 818-3238, by fax at (703) 818-8813, or by e-mail at mice@techlawinc.com.

IV. What Does Update V Contain?

Update V contains 23 new and revised analytical methods, revised versions of Chapters One through Five of EPA publication SW-846, the ORCR Policy Statement, and other guidance (e.g., quality assurance/quality control (QA/QC) guidance on lower limit of quantitation (LLOQ), relative standard error (RSE), initial demonstration of proficiency (IDP), etc.), each dated October 2012 and identified as "Update V" in the document footer. For the convenience of the reader, EPA has identified key areas of interest in the sections below, but all the methods and other information for which the Agency is seeking comments are contained in the docket for this Notice. Table 1 (included at the end of this Notice) provides a listing of the five revised chapters and twenty-three methods (eight new and fifteen revised methods) in Update V. After consideration of comments received from publication of this Notice, Update V, including the revised versions of Chapters One through Five, will be incorporated into the SW-846 methods compendium.

A. OSWER/ORCR Policy Statement

In 2008, ELAB requested that ORCR describe their plan for releasing Updates to SW-846, as well as clarify the status of deleted, obsolete, previous versions or revised methods, and a statement regarding the status of previous versions of methods. In addition, ELAB raised the following additional concerns and suggestions:

- Clarification is needed regarding which method is the final version in SW-846.
- Many states are not adopting the final version of new methods.

- States may not have the resources to certify multiple versions of final methods.
- Some of the regulated community doesn't know how the method revision varied.

EPA has engaged in several face-to-face meetings with the ELAB at national conferences to address their requests and resolve their concerns and suggestions. As a result of those meetings, ORCR developed a policy statement intended to clarify the basic terminology used in SW-846 regarding the status of methods and how the SW-846 Methods program develops and releases methods to the public. That policy statement, entitled "USEPA Office of Resource Conservation and Recovery Policy on the Use of *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (SW-846)" provides background on SW-846, general guidance on the procedures for adopting methods into SW-846, and defines key terms used to identify the status of methods in SW-846. Below is the ORCR Policy Statement, a copy of which has also been placed in the docket associated with this Federal Register Notice:

USEPA Office of Solid Waste and Emergency Response/Office of Resource Conservation and Recovery Policy on the Use of "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" (SW-846)

The United States Environmental Protection Agency's (EPA) Office of Resource Conservation and Recovery (ORCR) provides analytical and sampling methods to assist the regulated and regulatory community and others in implementing the Resource Conservation and Recovery Act (RCRA). These methods are published in the *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (SW-846) and are available on the ORCR web site (www.epa.gov./epawaste/hazard/testmethods/index.htm). With the exception of those particular methods which are promulgated in the regulations to implement RCRA (see 40 CFR 260.11), the remaining methods are considered guidance, and users may select any scientifically appropriate

method when conducting analyses to comply with the RCRA regulatory program.

The Methods Innovation Rule (MIR) published on June 14, 2005 (70 FR 34538), reemphasized the flexible approach in method selection, when appropriate, when testing for compliance, under RCRA. Since the publication of this rule, ORCR no longer uses a formal rulemaking process for publication of method updates to SW-846. EPA informs the regulated and regulatory community of new methods and updates to SW-846 and solicits comments on them through a Notice of Availability published in the Federal Register. This approach is consistent with ORCR's commitment to fully implement the Agency's performance-based measurement system (PBMS) approach to regulation.

A **new** effort was developed and approved to reinvigorate the goals of PBMS with the versatility of each of our program's needs. It is called the Flexible Approaches to Environmental Measurements – The Evolution of the Performance Approach which the Science and Technology Policy Council (STPC) approved on February 15, 2008. In 2009, ORCR subsequently adopted the new "Performance Approach" as defined by the Forum on Environmental Measurements (FEM). The FEM is a standing committee of senior EPA Environmental Protection managers established to develop policies to guide the Agency's measurement community in: validating and disseminating methods for sample collection and analysis; for ensuring that monitoring studies are scientifically rigorous, statistically sound, and yield representative measurements; and for employing a quality systems approach that ensures that the data gathered and used by the Agency are of known and documented quality.

After shortening the name of the PBMS effort to the "Performance Approach," the FEM's Performance Approach Action Team took a look at the issues surrounding the lack of the program's progress with the ultimate conclusion that the "one size fits all" approach does not

work for the diversely different programs and authorities each of our major program offices (i.e., air, pesticides, waste, and water) has in carrying out their work. To avoid the proliferation of terminology, ORCR has adopted the "Flexible Approach" which is consistent with ORCR's approach to environmental management, based on the goals and statutes of EPA program offices.

Under the PBMS approach for RCRA, when labs conducted regulatory required monitoring, the regulated community had to either employ a scientifically appropriate method published in SW-846 or use any other scientifically appropriate method from another reliable source. This is still true under the Flexible Approach. However, when choosing a reliable alternative source, the focus should be on measurement objectives, rather than on measurement technologies. In all cases, the user must demonstrate the method selected generates data that are appropriate for the intended use. Although both approaches are applicable for RCRA, ORCR had dropped the term PBMS, and strongly supports the use of the new Flexible Approach to be consistent with the Agency's new guidance that allows each program to determine program specific flexibility when addressing waste analysis issues.

ORCR strongly recommends that persons use the latest version of a SW-846 method whenever possible, especially in new monitoring situations, since updated versions of the methods EPA publishes generally are in the Agency's view less subject to misinterpretation, yield improved precision and/or bias, or provide for the use of newer and, often, more cost-effective technologies. In situations where it may not be appropriate to use the latest method in SW-846, earlier versions may be used. These situations may include, but are not limited to, those where an earlier version of a method is required for existing permits, consent decrees, waste analysis plans or sampling analysis plans. In addition, laboratories, especially small laboratories, may find a previous version of a SW-846 method appropriate if it is more cost-

effective in meeting the project-specific objectives. The Agency is not imposing restrictions on the use of earlier versions of non-required methods contained in SW-846 or precluding the use of previous guidance. Nonetheless, the adoption of the latest method version is recommended and should be accomplished as soon as possible, as appropriate. When methods are employed, it is the responsibility of the user to ensure that the method yields data of a quality appropriate for the particular application for which it is being used.

EPA views the methods in the SW-846 compendium as tools for the user to employ in developing individual standard operating procedures to meet the goals and objectives of specific projects. This approach enables the user to optimize and modify methods for effective performance on unique projects. The SW-846 methods are for most applications considered as guidance with the exception of those methods required by the RCRA regulations (i.e., Method-Defined Parameters (MDPs), see 40 CFR 260.11).

In situations where the user is not certain whether the selected method or method modification is appropriate, EPA recommends regulated entities contact and seek approval as needed from the appropriate regulatory agency (e.g., Federal or State/local government) before applying any method on a specific project, including situations where the method is used verbatim.

EPA may publish new methods, revise existing methods, or withdraw methods from the SW-846 compendium whenever it deems it appropriate. For example, methods may be updated in order to reflect new advancements in technology, to reflect the addition of new performance data, or to clarify areas of the procedure that experience indicates may be misunderstood. Methods may also be revised to reflect new EPA policy regarding the use of certain chemicals and reagents. In other cases, methods are removed if the technology is no longer available or

applicable. ORCR has developed specific procedures for releasing updates, revisions, or withdrawing methods, which are designed to minimize disruption to regulatory processes. Specific definitions for the terms associated with a method's status, which support the change procedures, have been developed and provided below.

The Agency will only post the most recent version of a final SW-846 method on the ORCR web page as part of the SW-846 methods compendium (www.epa.gov./epawaste/hazard/testmethods/index.htm). Prior versions of methods formerly contained in SW-846 and still considered appropriate for use will be available through a separate hyperlink in the future. EPA's objective is to identify and make available on the Agency's SW-846 website the latest information regarding the methodologies that generate effective data at minimum costs in response to new technological or scientific advancements, while, at the same time, making available earlier versions for those situations where such methods may be needed or appropriate (e.g., to determine how a particular analysis had been performed, to determine how to comply with a specific permit requirement, etc.).

SW-846 Methods Status Definitions

Analytical methods are officially made a part of the SW-846 manual through a rigorous process of technical evaluation both within the Agency and through external review. Methods are also revised as needed after a formal evaluation process by analytical experts (e.g., SW-846 work and focus groups) and an announcement of method availability and request for public comment in the Federal Register as a Notice of Availability. During the method development/evaluation process, the methods go through various stages of review and revision. The methods are officially included as part of an update to the most current edition of SW-846 at the conclusion of this process.

ORCR employs a specific naming convention (i.e., method number and letter suffix) when publishing methods. The naming convention is intended to minimize confusion within the user community regarding a method's developmental status. The method number designates the underlying technology (e.g., 8000 series methods designate determinative procedures for organic compounds). A revision to a method where the underlying technology does not change is indicated by continued use of the same method number and letter, but with a new issuance date. If the revision retains the underlying technology, but does not affect the precision and/or accuracy of the data, the revision is considered to be minor or nonsignificant and the method number and letter is not changed or sequenced.

If, on the other hand, the revision retains the underlying technology, but changes the precision and/or accuracy of the data, the change is considered to be significant and is indicated by a subsequent letter suffix (e.g., changes from 8270C to 8270D) and a new issuance date. For example, if the quality control recommendations are changed in a manner that improves the bias or precision of the method, but does not change the underlying technology (e.g., a tightening of the calibration acceptance criteria), the method number stays the same, but the letter suffix is sequenced to the next letter. The differences between the earlier and later versions of a method are detailed in the method summary section of the revised version regardless of the type of change.

Examples of changes that may be considered minor or nonsignificant include, but are not limited to: language added to a method to provide increased clarity or guidance; expansion of lists of acceptable instrumentation, applicability of the method to a matrix not previously referenced, adding new compounds to the list of applicable compounds, or changes to instrument specifications which do not result in an existing acceptable instrument being rendered

unacceptable; or formatting and editorial changes that are designed to improve readability or correct spelling or grammatical errors.

ORCR has defined a "significant change" as a change that results in improved analytical results (e.g., changes that result in reducing analytical bias or improving data precision). Examples of significant changes may include, but are not limited to: a change in the operating parameter which reduces analytical flexibility; a change in instrumentation specification which minimizes interference and/or optimizes instrument performance (if the use of such interference reduction technique or performance enhancement is required); a change in calibration guidance which results in more restrictive recommendations; a change that institutes tighter QC recommendations; or a change in the reagents that are required by the method.

ORCR understands revisions are sometimes necessary to either enhance the performance of the method or to allow flexibilities due to the complexity of sample matrices. In situations where the user is not certain whether the selected method, method modification or modification to their plan is appropriate, EPA recommends the regulated community seek approval from the appropriate regulatory agency (e.g., Federal or State/local government, client) before their use of a revised method; amend their plan (e.g., Project Plan, Quality Assurance Project Plan (QAPP), Sampling and Analysis Plan (SAP), Standard Operating Procedure (SOP)); and properly document the change when reporting analytical results.

The following method status definitions reflect the current method development process and have been developed to add clarity for the method users. ORCR uses these definitions and the terms may vary for other program offices.

Final Method – A method that has been formally adopted into the most recent version of the SW-846 compendium. Before a method becomes final, the validated version would have been

made available for public review and comment in a Notice of Availability (NOA) or a proposed rulemaking, as appropriate.

Validated Method – A method that has undergone development and technical review by EPA, but has not been formally adopted into the SW-846 method compendium and published through a Federal Register Notice. Since this review includes technical work group approval and/or inter-laboratory validation, validated methods are included on the Agency website for evaluation and use by the public and as a means of soliciting comment from the broader scientific community. The public may use a validated method prior to its inclusion in the SW-846 compendium, provided that the users demonstrate that it generates data that are appropriate for the intended use.

Revised Method – A method included in SW-846 that has been updated to reflect changes that may be editorial in nature and do not impact data or performance comparability, that broaden the method to introduce new technologies that may increase productivity, but do not change the fundamental technology, or that change the quality control requirements to increase bias or precision.

The number of a method that has been revised does not change, but the method may receive a subsequent letter suffix. If the revision is a significant one (as defined above) then both the letter suffix and the issuance date are updated. If, on the other hand, the revision is editorial in nature, or consists of the addition of new performance data, then only the issuance date is changed. Previous versions are not precluded from being used provided that the users demonstrate that it generates data that are appropriate for the intended use.

Draft Method – A new method that is being evaluated for possible inclusion into SW-846. It represents the latest innovative technological advancements in scientific methodology, but has

not completed technical review by EPA nor been subject to notice and comment in the Federal Register.

Superseded Method – A superseded method is an earlier version of an SW-846 method or other guidance that is no longer included in the SW-846 compendium and has been replaced by a newer version. Revised versions of ***Superseded*** methods should be viewed as the preferred method. Methods in this category are removed from the compendium, but remain available on line and are not precluded for use where required for existing projects or where an adequate justification for use exists. The term "***Superseded***" is documented in the method title as listed on the EPA website for prior versions of final methods followed by the date it was superseded.

Withdrawn Method – A method or other guidance that EPA strongly recommends not be used, (e.g., cyanide and sulfide reactivity guidance withdrawn, June 14, 2005). EPA has determined that such procedures or methods, for the use or technical objectives for which they were originally published, are technically inadequate and/or no longer meet such use or technical objectives. This does not mean, however, that there would be no situations under which the procedures or methods may be appropriate. In any situation in which a person may believe that the withdrawn method is appropriate, we strongly encourage consultation with applicable regulatory agencies at the state or federal level. The prospective user of the method will need to demonstrate the old method is, indeed, appropriate. Any use of these methods, without any such consultation and demonstration, will be done at the user's risk.

The Agency understands that earlier versions of the SW-846 methods that aren't required may still be in use to meet project specific criteria (e.g., permits, sampling plans, Consent Decrees, etc.). Permits and other plans formally approved by regulatory authorities that specify the use of particular methods for required analysis continue in effect unless they are changed.

However, the Agency encourages the regulated community to use the *latest version* of SW-846, when applicable. EPA will continue to update the Methods Status Table to inform the public as to the status of methods in SW-846 and the Policy Statement will be added to the SW-846 methods compendium when the Update V package is finalized.

[end of policy statement]

B. Changes to Chapters One through Five and QA/QC Guidance (Chapter One and individual methods) in SW- 846

In general, EPA's revisions to Chapters One through Five to EPA publication SW-846 reflects the new method style guide format and added all the Update V methods and new letters/version to the appropriate related method sections. Specifically:

- **Chapter One** of SW-846 was revised to make it more user friendly and to be more consistent with the Agency's official guidance on QA/QC implementation and procedures (e.g., Quality Assurance Project Plans (QAPPS), Data Quality Objectives (DQOs), and the Flexible Approach to Environmental Measurement).
- **Chapter Two** now includes a Table of Contents to make finding the information easier. In addition, a typographical error was found for bis(2-chloroisopropyl) ether and was corrected to bis(2-chloro-1-methylethyl) ether in Tables 2-1, 2-4, 2-15, 2-22, and 2-34. Furthermore, Table 2-40(A) was revised to reflect the current sample preservation guidance for styrene and vinyl chloride in aqueous samples (i.e., deletion of previously recommended practice of collecting a second set of samples without acid preservatives and analyze immediately, if styrene and vinyl chloride are analytes of interest) and Table 2-40(B) was revised to include Mercury Speciation hold times in addition to totals.

- **Chapter Three** was revised so that the definition for instrument detection limit (IDL) is consistent with the revised methods 6010D and 6020B. In addition, the term "accuracy" was replaced by "bias" where appropriate; the definition for linear range was revised to be consistent with methods 6010D and 6020B; the definition of interference check sample (ICS) was replaced with the spectral interference check (SIC) solution to be consistent with methods 6010D and 6020B; and the definition of "laboratory control sample" was revised to recommend the use of a spiking solution from the same source as the calibration standards. Also, the collision/reaction cell technology was added to Sections 3.6 and 3.7 as an effective method for removing isobaric interferences when analyzing by ICP-MS and a minimum collection mass of 100 g was added to Table 3-2 for solid samples collected for sulfide analysis.
- **Chapter Four** (see Table 4-1) was reformatted and updated by removing the recommendation to collect a second set of samples without adding an acid preservative and analyze in a shorter time frame if vinyl chloride and styrene are analytes of concern for aqueous samples.
- **Chapter 5** had no significant changes outside of general ones specified above (e.g., updated format changes and method reference to chapters).

In addition, EPA is incorporating three new and revised QC features in Chapter One and the Update V methods, where appropriate, for RCRA compliance monitoring which warrant further discussion here. A summary of changes to chapters in SW-846 are provided in Appendix A of each chapter.

The new and revised features that have been added to Chapter One *(Quality Control)* and individual methods (where appropriate) are:

- *Lower Limit of Quantitation (LLOQ)* – References to the Method Detection Limit (MDL) have been replaced with the LLOQ. It is recommended to establish the LLOQ as the lowest point of quantitation, which, in most cases, is the concentration of the lowest calibration standard in the calibration curve that has been adjusted for the preparation mass and/or volume. The LLOQ value is a function of both the analytical method and the sample being evaluated.

Why is MDL removed and replaced by LLOQ for SW-846?

ORCR has removed references to the MDL procedure (i.e., 40 CFR 136, Appendix B) beginning with Update IV and from the revised and new Update V methods and has recommended establishing the LLOQ. We continue to refine the procedure for establishing the LLOQ. The refined procedure considers sample matrix effects; provides a provision to verify the reasonableness of the reported quantitation limit (QL); and recommends a frequency of LLOQ verification (found in Chapter One and each method) to be balanced between rigor and practicality. (Note: The agency understands that previous versions of methods published in SW-846 may contain the MDL reference. However, as methods are updated, EPA will remove the reference to the MDL, and will remove the reference in older methods that have not yet been updated, as time and resources allow. Therefore, ORCR recommends that LLOQ be used, as appropriate, for the methods that have not yet been updated. See the Section 9.8 in Method 6020B for inorganic analytes and Section 9.7 in Method 8000 for organic analytes on LLOQ for further information on implementation.)

ORCR understands that other EPA programs may continue to use MDLs to meet their program use and needs (e.g., the National Pollutant Discharge Elimination System

(NPDES) permit program). However, ORCR has found that the procedure in 40 CFR 136, Appendix B, for the determination of MDLs, developed for the Clean Water Act (CWA) program uses a clean matrix (e.g., reagent water for preparing "spiked" samples, or samples with known constituent concentrations). Analytical laboratories often have difficulty demonstrating they can meet the MDL established using Part 136 when evaluating complex matrices, such as wastes. The procedure outlined in Part 136 is generally not suitable for RCRA wastes or materials because the MDL approach generally yields unrealistic and/or unachievable method detection limits for the complex matrices (e.g., soils, sludges, wipes, and spent materials) encountered under the RCRA program. The MDLs are normally calculated from analysis of a sample that does not cause matrix interferences (typically determined using spiked reagent water). However, most wastes evaluated for compliance with RCRA consist of complex matrices. The LLOQ considers the effect of sample matrix (e.g., components of a sample other than the analyte) by taking the sample through the entire analytical process, including sample preparation, clean up (to remove sample interferences), and determinative procedures. Also, if method users choose, the LLOQ sample can be included at the end of the run to see if it meets the established acceptance criteria. Lastly, results above the LLOQ are quantifiable within an acceptable precision and bias. Thus, the LLOQ approach better suits the needs of the RCRA program, because it provides reliable and defensible results, especially at the lower level of quantitation, and can be reported with a known level of confidence for the complex matrices being evaluated.

SW-846 methods are being used by various programs in implementing various statutes , including RCRA, the Comprehensive Environmental Response, Compensation

and Liability Act (CERCLA), the Toxic Substances Control Act (TSCA), and Homeland Security Presidential Directives, for waste and materials characterization, compliance testing, site/incident characterization and risk assessment for protection of human health and the environment, and better management and use of wastes and materials, for a wide range of difficult matrices. ORCR believes that the LLOQ approach is an important improvement, and supports the essential need to provide data that are verified to meet the precision and accuracy requirements of the Agency's program needs.

Establishing LLOQ for Inorganic analytes:

When performing methods for *inorganic* analyses, the LLOQ should be verified by the analysis of at least seven replicate samples (prepared in a clean matrix or control material) and spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation (RSD) of these samples provide an initial statement of precision and bias at the LLOQ. In most cases, the mean recovery should be ±35% of the true value and the RSD should be $\leq$ 20%. Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix free of target compounds. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements. For more information, please see the individual methods (e.g., Methods 6010 and 6020) and Chapter One of SW-846.

Establishing LLOQ for organic analytes:

When performing methods for *organic* analyses, the LLOQ should be verified

using either a clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix free of target compounds. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

For *organic* analyses, the acceptable recovery ranges of target analytes will vary more than for other types of analyses, such as inorganics. The recovery of target analytes in the LLOQ check sample should be within established limits, or other such project-required acceptance limits, for precision and bias to verify the data reporting limits. Until the laboratory has sufficient data to determine acceptance limits statistically, a limit of 20% +/- the Laboratory Control Sample (LCS) criteria may be used for the LLOQ acceptance criteria. This approach acknowledges the poorer overall response at the low end of the calibration curve. Historically based LLOQ acceptance criteria should be determined as soon as practical once sufficient data points have been acquired.

In-house limits for bias (e.g., % Recovery) and precision (e.g., Relative Percent Difference, %RPD) of the LLOQ for a particular sample matrix may be calculated when sufficient data points exist. The laboratory should have a documented procedure for establishing its in-house acceptance ranges. Sometimes the laboratory instrument and/or analyst performance vary or test samples cause problems with the detector (e.g., samples may have interferences; may clog the instruments cells, wall or tube; may cause contamination; etc.). Therefore, the limits of acceptance (for precision and bias) are established by a lab with sufficient data to demonstrate that they can report down to the LLOQ with a certain level of confidence. The acceptance limits (for precision and bias) for LLOQ may be established by the laboratory or at the project level through the data

quality objectives in a quality assurance project plan. The frequency of the LLOQ check is not specified for organic analytes.

Note: The LLOQ check sample should be spiked with the analytes of interest at the predicted LLOQ concentration levels and carried through the same preparation and analysis procedures as environmental samples and other QC samples. For more information, please see individual methods (e.g., Method 8000) and Chapter One of SW-846.

How is LLOQ used?

The RCRA program deals with complex wastes and materials that are managed or used in many different ways (e.g., landfilling, land application, incineration, recycling). The thresholds (e.g., action or remediation levels) for data users (e.g., engineers or risk assessors) to make their decisions, therefore, vary. Method users will need to properly plan their analytical strategy to ensure the LLOQs for targeted analytes are lower than the thresholds needed to generate data used to determine how waste or materials can be properly managed or used.

- *Initial Demonstration of Performance (IDP)* – The laboratory must make an initial demonstration of ability to generate results with acceptable accuracy and precision for each preparation and determinative method they perform. This demonstration should be performed prior to independently analyzing real sample matrices by each analytical method and should be repeated if other changes occur (e.g., significant change in procedure, new staff are trained, etc.). Documentation of the IDP should be maintained by the Quality Assurance Manager. Each laboratory should have a training program documenting that a new analyst is capable of performing the method or portion of the

method for which the analyst is responsible. This demonstration should document that the new analyst is capable of successfully following the standard operating procedure (SOP) based on the laboratory's IDP policy.

For Update V, changes to the IDP have been specified in the individual Update V methods where appropriate (e.g., screening method where there is not a quantitative reporting limit such as a bioassay method). The IDP changes allow laboratories to use their time and resources effectively, especially for the organic analyses.

Key Changes in the IDP for the Determination of Organic Analytes:

The IDP section was expanded to describe two situations:

When a significant change to instrumentation or procedure occurs:

Reliable performance of the methods is dependent on careful adherence to the instructions in the written method, and aspects of the method are mandatory to ensure that the method performs as intended. Therefore, if a major change to the sample preparation procedure is made (e.g., a change of solvent), the IDP must be repeated for that preparation procedure to demonstrate the laboratory technician's continued ability to reliably perform the method. EPA considers conducting IDPs as part of good laboratory practice procedures and has already included these procedures in EPA's laboratories practices.

Alterations in instrumental procedures only (e.g., changing Gas Chromatograph (GC) temperature programs or High Performance Liquid Chromatography (HPLC) mobile phases or the detector interface), require a new calibration, but not a new IDP because the preparation procedure is unchanged.

When new staff members are trained:

A new analyst needs to be capable of performing the method, or portion of the method, for which the analyst is responsible. For example, when analysts are trained for a subset of analytes for an 8000 series method, the new sample preparation analyst should prepare reference samples for a representative set of analytes (e.g., the primary analyte mix for Method 8270, or a mix of Aroclor 1016 and 1260 for Method 8082) for each preparation method the analyst will be performing. The instrument analyst being trained will need to analyze prepared samples (e.g., semi-volatile extracts).

- *Relative Standard Error (RSE)* – ORCR evaluated and included, as the analytical community recommended, RSE as an option (in addition to calculation of the % error) in SW-846 for the determination of the acceptability for a linear or non-linear calibration curve. RSE refits the calibration data back to the calibration model and evaluates the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of Relative Standard Error (RSE - expressed as %)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x'_i - x_i}{x_i}\right]^2 / (n - p)}$$

where:

x_i = True amount of analyte in calibration level i, in mass or concentration units

x'_i = Measured amount of analyte in calibration level i, in mass or concentration units

p = Number of terms in the fitting equation

(average = 1, linear = 2, quadratic = 3, cubic = 4)

n = Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit in the determinative method. If the RSD limit is not defined in the determinative method, the RSE limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

V. Summary

EPA believes that these changes in Update V will assist the method users to demonstrate method competency and generate better quality data. For the convenience of the analytical community, the Agency will revise the OSWER Methods' Team homepage on EPA's website with updated information to better communicate new policy and analytical procedures, and will include Update V and selected documents at that website after Update V is finalized.

Please see the website: http://www.epa.gov/epawaste/hazard/testmethods/index.htm for more information. Table 1 provides a listing of the five chapters and twenty-three methods (eight new and fifteen revised methods) in Update V.

Table 1. Update V (Methods, Chapters and Guidance)

Analytical Method No.	Method or Chapter Title
--	Table of Contents
--	Chapter One – Quality Control
--	Chapter Two – Choosing the Correct Procedure
--	Chapter Three – Inorganic Analytes
--	Chapter Four – Organic Analytes
--	Chapter Five – Miscellaneous Test Methods
--	Methods Status Table
1030	Ignitability of Solids
3200*	Mercury Species Fractionation and Quantification by Microwave-assisted Extraction, Selective Solvent Extraction and/or Solid Phase Extraction
3511*	Organic Compounds in Water by Microextraction
3572*	Extraction of Wipe Samples for Chemical Agents

Analytical Method No.	Method or Chapter Title
3620C	Florisil Cleanup
4025*	Screening for Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/Fs) by Immunoassay
4430*	Screening For Polychlorinated Dibenzo-p-Dioxins and Furans (PCDD/Fs) by Aryl Hydrocarbon Receptor PCR Assay
4435*	Method For Toxic Equivalent (TEQS) Determination For Dioxin-Like Chemical Activity with the CALUX®Bioassay
5021A	Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis
6010D	Inductively Coupled Plasma-Atomic Emission Spectrometry
6020B	Inductively Coupled Plasma-Mass Spectrometry
6800	Elemental and Speciated Isotope Dilution Mass Spectrometry
8000D	Determinative Chromatographic Separations
8021B	Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors
8111	Haloethers by Gas Chromatography
8270D	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry
8276*	Toxaphene and Toxaphene Congeners by Gas Chromatography/Negative Ion

Analytical Method No.	Method or Chapter Title
	Chemical Ionization Mass Spectrometry (GC-NICI/MS)
8410	Gas Chromatography/Fourier Transform Infrared Spectrometry for Semivolatile Organics: Capillary Column
8430	Analysis of Bis(2-Chloroethyl) Ester and Hydrolysis Products by Direct Aqueous Injection
9013A	Cyanide Extraction Procedure for Solids and Oils
9014	Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide
9015*	Metal Cyanide Complexes by Anion Exchange Chromatography and UV Detection
9320	Radium 228

* New Methods

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