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A DOE MANUAL: DOE METHODS FOR EVALUATING ENVIRONMENTAL AND WASTE MANAGEMENT SAMPLES

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## **A DOE Manual: DOE Methods for Evaluating Environmental and Waste Management Samples**

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### **Abstract**

Waste Management inherently requires knowledge of the waste's chemical composition. The waste can often be analyzed by established methods; however, if the samples are radioactive, or are plagued by other complications, established methods may not be feasible. The U.S. Department of Energy (DOE) has been faced with managing some waste types that are not amenable to standard or available methods, so new or modified sampling and analysis methods are required. These methods are incorporated into *DOE Methods for Evaluating Environmental and Waste Management Samples (DOE Methods)*, which is a guidance/methods document for sampling and analysis activities in support of DOE sites. It is a document generated by consensus of the DOE laboratory staff and is intended to fill the gap within existing guidance documents {e.g., the Environmental Protection Agency's (EPA's) *Test Methods for Evaluating Solid Waste*, SW-846}, which apply to low-level or non-radioactive samples. *DOE Methods* fills the gap by including methods that take into account the complexities of DOE site matrices. The most recent update, distributed in October 1993, contained quality assurance (QA), quality control (QC), safety, sampling, organic analysis, inorganic analysis, and radioanalytical guidance as well as 29 methods<sup>(1)</sup>. The next update, which will be distributed in April 1994, will contain 40 methods and will therefore have greater applicability. All new methods are either peer reviewed or labeled "draft" methods. Draft methods were added to speed the release of methods to field personnel.

*DOE Methods* is available at no cost to the user and is supported by the Laboratory Management Division of DOE. As a living document, *DOE Methods* provides a vehicle for technology transfer within the environmental restoration and/or waste management (EM) community. As *DOE Methods* evolves, its usefulness and applicability is anticipated to grow to meet the demands of the DOE/EM mission.

## Background

Since 1991, standard operating procedures have been accumulating from the DOE and DOE contractor analytical laboratories within the DOE complex. The first acquisition of procedures from all DOE sites was completed in 1993. These procedures were placed in the *Procedures Database*<sup>(2)</sup>, a repository of standard operating procedures that have been or are being used at various DOE sites for EM sampling and/or analysis. The database is managed by Los Alamos National Laboratory (LANL) and uses SEARCHmate™, allowing the user to search all procedures quickly through a modem or network connection. Procedures can be sent by FAX directly from the database to the user by selecting that option in the program.

In parallel to the development of this database, a compendium of new and consolidated methods was developed. This compendium, "*DOE Methods for Evaluating Environmental and Waste Management Samples*" (*DOE Methods*), is managed at PNL and contains methods that are intended to provide long term guidance to EM activities throughout the DOE and its sphere of influence. The first issue of *DOE Methods* was completed in October 1992 and contained QC, safety, sampling, organic analysis, inorganic analysis, and radioanalytical guidance as well as four methods<sup>(3)</sup> (see Figure 1). Shortly after its release, it was described in the literature<sup>(4, 5)</sup>. Revision 1, completed in March 1993, had an expanded guidance section and seven methods<sup>(6)</sup>.

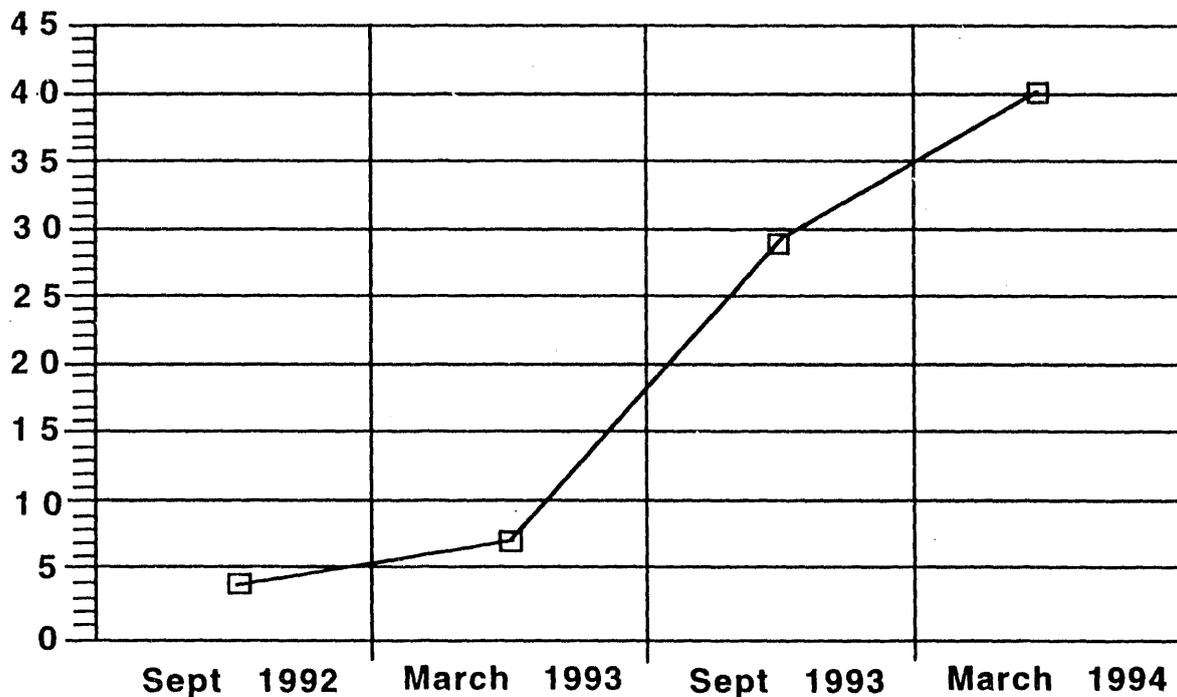


Figure 1. Methods in *DOE Methods*

*DOE Methods* is expected to contain 40 methods in April with more useful sampling and analytical guidance. It will be updated biannually (April and October) to quickly meet DOE/EM sampling and analytical needs<sup>(1)</sup>.

## Document Status

**Guidance Chapters.** Guidance chapters in *DOE Methods* have been modified periodically to address reviewer comments and to enhance the applicability of the document for EM needs. Method categories and guidance for their use outline the scope of this document. The titles of the major guidance chapters and the method categories are shown below.

Guidance Chapters	Method Categories
• Quality Assurance	• Sampling Methods
• Quality Control	• Organic Methods
• Safety	• Inorganic Methods
• Waste Handling	• Radiochemistry Methods
• Sampling and Analysis Plan	• Miscellaneous Methods
• Choosing the Correct Method	

**Method Numbers** Several analytical method documents exist in which numbers are clearly distinguishable. *DOE Methods* also uses a unique method numbering system that helps identify the category in which the method belongs. Table 1 gives the method numbering system found in *DOE Methods*, with an example to explain the system rationale.

**Methods.** The April 1994 update of *DOE Methods* will contain 40 methods<sup>(1)</sup>. These methods provide resources for EM sampling and analytical activities. While these methods do not cover all EM needs, it is anticipated that they will provide useful reference material for DOE/EM activities. Their titles and distribution are summarized in Table 2. The organic and inorganic analytical methods are either single laboratory adaptations of standard methods or new methods that were developed independently of standard methods, but fulfill DOE/EM needs. Radiochemistry and sampling methods generally reflect procedures that are used at one or more DOE sites, but which were consolidated to provide a standard reference for these activities. Some methods in *DOE Methods* were consolidated from procedures in the *DOE Procedures Database*<sup>(2)</sup>. Consolidated methods reference the standard operating procedures that were used to create the methods. In some cases, procedures may not have been available in the database, and procedures were obtained

from outside the database to fill a need. The DOE labs submit updates and new procedures to keep the DOE Methods program current with new developments.

**Table 1.** The Method Numbering System used in *DOE Methods*

S	Sampling	M	Inorganics
SAxxx	Air Sampling	MSxxx	Screening
SWxxx	Water Sampling	MPxxx	Sample Prep
SSxxx	Soil Sampling	MAxxx	AA Methods
SDxxx	Drum sampling	MBxxx	Immunological Assays
		MCxxx	Ion Chromatography
O	Organics	MIxxx	ICP Methods
OSxxx	Screening	MMxxx	ICP-MS Methods
OCxxx	Cleanup	MVxxx	Uncategorized Methods
OPxxx	Sample Prep		
OGxxx	GC Determinative/Various Detectors	R	Radionuclides
OHxxx	HPLC Determinative/Various Detectors	RSxxx	Field Screening
OMxxx	GC-MS or LC-MS Determinative	RPxxx	Sample Prep
OIxxx	IR or GC-IR Determinative	RIxxx	Instrumentation
OVxxx	UV-Vis Determinative	RAxxx	Miscellaneous Radiochemistry
OXxxx	TOC, TOX		
		X	Miscellaneous Methods

Example:       OG081R  
                  1  2 3

1. The first two letters designate classes of methods, as indicated above.
2. Then each method is given a distinct number (the next three digits).
3. Methods designed for use with radioactive samples have an "R" at the end of the number.

## Document Distribution

The distribution of *DOE Methods* has grown from 100 to 700 over FY 1993, reflecting increased interest in EM consolidated methods.

**Table 2.** Methods to be Contained in DOE Methods (April, 1994)

### Sampling

- ST010 General Method for Sampling Liquids and Solids in LLW Storage Tanks (Draft)

### Organic Chemistry

- OS010 Total Organic Cl in Oil
- OS020 Immunoassay for PCBs in Soil
- OS030 A Photoacoustic Infrared Method for the Determination of Selected Chlorinated Volatile Organic Chemicals (VOCs) in Water (Draft)
- OC010R A Preparation and Cleanup Method for Hydrocarbon Containing Samples for the Analysis of volatile Organic Compounds
- OP010R Remote Purge-and-Trap in a Hot Cell (Draft)
- OP020R Ultrasonic Solvent Extraction for Volatile Organic Compound Analysis of Solid Radioactive Mixed Wastes (Draft)
- OP030R Purge-and-Trap
- OP100R PCBs in Aqueous Radioactive Mixed Waste
- OP120R Reduced Scale Liquid-Liquid Extraction of Semivolatile Organic Compounds
- OP550R Ultrasonic Extraction
- OG015R Major Nonhalogenated Volatile Organics in Radioactive Aqueous Liquids Analyzed by Direct Aqueous Injection Gas Chromatography (DAI-GC) (Rev 1)
- OG081R Analysis of PCBs as Aroclors in Solid Radioactive Mixed Waste (Rev 1)
- OH100R Direct Analysis of TCLP by HPLC (Draft)

### Inorganic Chemistry

- MB100 Immunoassay for Mercury in Soils
- MP100R Solvent Extraction of Uranium and Thorium from Radioactive Liquid Wastes
- MP110R Cleanup of Transuranic Liquid Wastes using Extraction Chromatography
- MS110 An Indicator Strip-Based Colorimetric Test for Chromate Ions ( $\text{CrO}_4^{2-}$ ) in Aqueous Samples (Draft)
- MS210 An Indicator Strip-based Colorimetric Test for Lead in Water
- MS310 An Indicator Strip-based Colorimetric Test for Nitrate in Water and Soil
- MS410 An Indicator Strip-Based Colorimetric Test for Nickel ( $\text{Ni}^{2+}$ ) in Aqueous Samples (Draft)
- MU012R Total CN by Microdistillation/Argentometric Titration (Draft)

### Radiochemistry

- RS100 In-Situ Analysis of Gamma-Ray-Emitting Radionuclides by Borehole Logging (Draft)
- RP230 Iodine-129 Analysis in Aqueous Solutions
- RP300 Nickel-59 and 63 Determination in Aqueous Samples
- RP330 Separation of Nb for  $^{93\text{m}}\text{Nb}$  and  $^{94}\text{Nb}$  Determination (Draft)
- RP500 Strontium-89 in Water
- RP501 Determination of Total Radioactive Strontium in High Level Samples using Extraction Chromatography (Draft, revised)
- RP510 Determination of Strontium-90 in Environmental Samples (Draft)
- RP520 Determination of Strontium-90 in Soil, Water, and Filter Samples (Draft)

- RP530 Determination of Selenium-79 in aqueous Samples (Draft)
- RP550 Technetium-99 Analysis Using Extraction Chromatography
- RP580 Tritium Distillation from soil in Water Using Lachat Micro-Dist™ System (Draft)
- RP710 Gross Alpha and Beta Screening for Laboratory Analysis
- RP720 Rapid Determination of Gross Alpha/Gross Beta/Tritium in Water Samples Using LSC (Draft)
- RP730 Gross Gamma Screening for Environmental Matrices (Draft)
- RI010 Gamma Spectrometry (Draft)
- RI100 Liquid Scintillation Instrumentation Method (Draft)
- RA010 Procedures for Utilization of Electret Ionization Chambers for Characterization of Gross Alpha Emissions from Indoor Sources (Draft)
- RA020 Procedures for Utilization of Alpha Track Detectors for Characterization of Gross Alpha Emissions from Indoor Sources (Draft)

## **The Performance Approach**

*DOE Methods* encourages the use of performance-based methods. In July 1993, an approach was presented to allow new methods to be used following demonstration of their appropriateness to satisfy the data quality objective (DQO) needs of the project<sup>(7)</sup>. This approach allows use of new or modified consensus methods for characterization EM samples. When mixed waste samples are being analyzed, this approach is especially necessary to address the unusual and sometimes unique matrix effects and the ALARA concerns of these samples. In the April 1994 update, this document will include an appendix describing a process for demonstrating how an alternate method can be generated, yet still be appropriate for EM activities. By publishing the process in draft form, we will be encouraging acceptance from both regulators and the regulated community.

## **Needs List**

*DOE Methods* does not currently address all the EM sampling and analysis needs of DOE. We first need to identify gaps that need filling. A first draft to identify *DOE Methods* needs was produced<sup>(8)</sup>. To meet the identified needs, procedures from the database or from the field are being identified, selected, and/or consolidated. Consolidated methods are initially written as draft methods and then sent for peer review. Unique methods and methods using new technology are solicited from the field. These methods are formatted as "Draft" methods, then sent for peer review. The Needs List is a "living" document and will be updated as revisions are needed or new needs are identified.

**Method Verification.** The method verification process is ongoing. As Draft methods in *DOE Methods* are used by the field, information about how the method performs is sent to the editors of *DOE Methods*. The draft methods are also sent for peer review. Input from the method

users and the peer review comments are evaluated, enabling the "draft" status to be removed. This process will assist in verifying the usefulness of the methods in this document and will act as a cooperative pool of information for all DOE labs.

## Conclusion

*DOE Methods* contains guidance as well as sampling and analytical methods to address DOE's characterization needs for EM programs. It fills gaps currently not addressed by EPA or American Society for Testing and Material (ASTM) guidance, thus aiding DOE in being able to implement cost-effective strategies for monitoring, cleanup, and management of wastes and environmental contamination that are, in part, unique to the DOE complex. The DOE Methods Compendium Program works with the consensus of all DOE/EM lab staff and provides the vehicle for DOE and DOE contractors to submit procedures so they can be considered for inclusion in *DOE Methods*. *DOE Methods* is a standard reference document for guidance in conducting contaminant characterization at DOE sites. Now that *DOE Methods* contains a large variety of methods, we anticipate accelerated use by various EM programs in FY 1994.

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