



**The Impact of New US EPA Methods - A
Case Study of Contortions and
Permutations
US EPA Method 5035**

Ruth L. Forman, CEAC
Rock J. Vitale, CEAC, CPC
Environmental Standards, Inc.



A banner image showing a sunset over a field with silhouettes of trees and animals. The sky is filled with orange and yellow clouds, and the sun is low on the horizon.

Overview

- New/Updated SW-846 Methods – Stay or Go?
- Case Study – SW-846 Method 5035
- Possible Solutions



Method Creation

US EPA SW-846 Methods

Represented a venue by which new analytical methods were submitted, reviewed, proposed, and ultimately promulgated.

As we have all heard – “It’s Only Guidance”

With each published update, new methods were proposed, existing methods were promulgated and/or revised, and in some cases, obsolete methods were deleted.

It was never quite clear, however, as to when or if new methods or method revisions would be required (*i.e.*, effective date) or if a deleted method could no longer be used.

What Has Happened With Recent Releases?

- No clear use of status language with method release, leading to confusion within state regulatory agencies; resulted in piecemeal recognition for monitoring and remediation uses by these agencies that have caused interstate accreditation recognition difficulties.
- Update IV includes quality control requirements that conflict with previous method versions causing laboratory and regulatory community confusion when determining which criteria to apply.



Case Study – SW-846 Method 5035



Methods 5030 and 5030A

- Method 5030 - promulgated in SW-846 3rd Edition, September 1986.
- Used for both aqueous and soil samples.
- Purge and trap of sample directly (low level) or a portion of methanolic extract (medium level) - Soils with a heated purge.
- Method 5030 was used for sample preparation for 8000 series methods (8010, 8020, 8240) - replaced by 5030A. Note: Methods 8010, 8020, and 8240 were deleted from SW-846.
- Method 5030A - promulgated in SW-846 3rd Edition, Update I, July 1992 to clarify definitions and include capillary GC column techniques (8260).

Sampling And Preservation Techniques For Methods 5030 and 5030A

- Aqueous samples - 2 or 3 40-mL VOA vials with Teflon[®] septa, no headspace, pH < 2 with HCl
- Soil samples – 4-ounce widemouth glass jars (or similar sampling devices, such as brass sleeves), minimal headspace, non-homogenized, and not preserved
- Samples submitted on ice at 4°C
- Unpreserved solid samples subject to a 14-day collection-to-analysis holding time

Advantages, Limitations, And Concerns For “Old” Methods

- Advantages:
 - We have always collected and analyzed samples in the same manner.
 - Data are “assumed” to be consistent for all sampling.
- Limitations and Concerns:
 - Significant low bias of soil sample results (typically 0.1% to 5% of “true” value)
 - Volatilization of compounds during sampling, transport, and sample preparation
 - Biodegradation due to lack of preservative (documented to be significant)
 - Lack of comparison between low-level and medium-level soil results
 - Extended holding time of 14 days from collection
 - Comparison to earlier data at the same site on a long-term project

Motivation For Change To Obtain Good Data

- BAD SAMPLE + GREAT LABORATORY = BAD DATA
- Being Responsible
- Properly Assessing Risk (liabilities to client); risk assessments may be seriously underestimated for risks related to soil exposure.
- Obtain Accurate Data for Remedial Design
- Is a bigger number better data? It depends on your perspective.

The New “Improved’ Techniques

- Method 5030B: SW-846 3rd Edition Update III, Revision 2, December 1996
- Promulgated in June 1997
- Replaces 5030A in SW-846 (6 months from promulgation date implementation period)
- Can only be used for waters and high-concentration soil extracts prepared by Method 5035
- Low-level soil preparation has been deleted.



To Reiterate.....

- SW-846 Method 5030B (issued in June 1997) does NOT include criteria for the collection of solid samples for VOC analyses.
- Method 5030B specifically references Method 5035 for the collection of soil samples for low-level VOC analyses.
- For emphasis, Method 5030 was deleted from SW-846 methods.
- Method 5030 no longer exists as a viable method for the collection and preservation of solid samples for VOC analyses.
- SW-846 Method 5035 (and the more recent version, Method 5035A) provides collection and preservation criteria for solid samples collected for low-level VOC analyses.

Method 5035: Closed-System Purge-And-Trap And Extraction For Volatile Organics Revision 0, December 1996

- Promulgated in SW-846 3rd Edition, Update III, June 13, 1997
- Describes a closed-system purge-and-trap process for the analysis of VOCs in solid samples.
- Designed for use with samples containing low levels of VOCs.
- Provides optional procedures for the collection and preparation of solid samples containing high concentrations of VOCs and for the collection and preparation of oily wastes.

Which Sample Collection Vessel For Volatiles Is Correct?



Method 5035: Sampling Options

- Use of 4-ounce wide-mouth containers is NOT acceptable for the collection of solid samples for VOC analyses.
 - EnCore® or Terracore® Samplers
 - Sodium Bisulfate Preservation
 - Methanol Preservation
- Soil samples must either be analyzed within 48 hours from collection or preserved within 48 hours from collection (and analyzed within 14 days from collection).



Method 5035A

(But I haven't started using 5035 yet!)

- US EPA SW-846 3rd Edition, Update III, July 2002
- Non-promulgated method
- Provides additional options and details for sampling
 - Empty vials – frozen within 48 hours
 - Deionized water vials – frozen within 48 hours

Method 5035 Considerations And Mishaps

- Reporting Limit Considerations

**Table 1: State of Washington Method A
Soil VOC Cleanup Levels (Unrestricted
Land Use).**

Volatile Organic Compound (VOC)	Cleanup Level (ug/kg)
Benzene	30
Ethyl Benzene	6,000
MTBE	100
Naphthalene	10,000
Tetrachloroethylene	50
Toluene	7,000
Trichloroethane-1,1,1	50
Trichloroethylene	30
Xylene	9,000

Source: Table 740-1 (Chapter 173-340 WAC)

- Fizzy samples
- Crystalline sodium bisulfate
- Acetone production

Why Are We Still Sampling “Old-School”?

- The pre-approved, site-specific SAP/QAPP still allows the consultants to collect and submit soil samples in these vessels.
- The sampling consultants are unaware that Method 5035 exists and that 4-ounce containers are no longer acceptable collection vessels.
- Higher costs associated with EnCore samplers
- Regulators have not yet realized that the sampling consultants are still following Method 5030.
- Regulators have not mandated 5035/5035A.

Why Are Laboratories Still Honoring These Requests?

- The pre-approved, site-specific SAP/QAPP still references Method 5030.
- Laboratory personnel are not comfortable telling consultants that they are incorrect and that sampling in 4-ounce containers is no longer acceptable.
- Laboratory assumes that consultants know what they want, need, and require.
- Laboratory personnel assume that if incorrect materials are requested, the blame falls on the consultant.



Possible Solutions?

- Laboratories to educate data users.
- ORCR to provide a strong written confirmation that the latest version is the preferred version of a test method.
- A 6-month to 1-year time-period be stipulated for implementing new versions of methods.
- ORCR to include termination dates for all old methods and develop specific regulatory milestones for replacement.



Possible Solutions?

- ORCR to implement a policy that employs more strict criteria for revising methods.
- ORCR to provide a change summary in the beginning of each new method to clearly indicate what has been changed and its quality impact.
- ORCR to reconfigure the method status table on the SW-846 methods page to just indicate the most recent version.



Possible Solutions?

- ORCR to provide clear definitions and intended use for terms such as draft, obsolete, withdrawn, final, preferred use, method, etc.
- ORCR to caucus with states, the NELAP Board, and other interested stakeholders to develop accreditation strategies.

A banner at the top of the slide features a sunset over a body of water with silhouettes of trees and birds in the sky. The banner has a dark blue background on the right side where the text is located.

Thank You



*Setting the Standards for Innovative
Environmental Solutions*

