



Putting the Pieces of the Puzzle Together

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Topics to Be Covered

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Brief Review of Current Method Team Projects in the Queue

Item 2

What's New in SW-846?

Item 3

The Status of Update V

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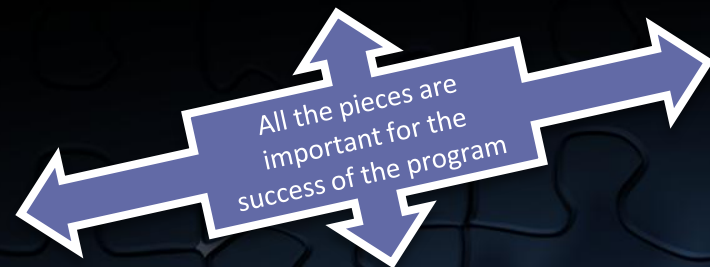
Resource



Methods Team Projects In the Queue

Top Priorities:

- Toxaphene
 - Method 8276 for the determination of toxaphene and 8 congeners in fish tissue
- Update V Methods Package
- Public Outreach
 - Forum on Environmental Measurements (FEM)
 - <http://www.epa.gov/fem/index.htm>
 - MICE Service
 - Methods Team Website



Toxaphene

Method 8276

- **Toxaphene** historically analyzed with organochloride pesticides by Method 8081
- Method 8276 developed for technical toxaphene and 8 toxaphene congeners using gas chromatography negative ion chemical ionization mass spectrometry (GC-NICI/MS)
- Method 8276 was posted online in March 2010:
<http://www.epa.gov/osw/hazard/testmethods/pdfs/8276.pdf>
- EPA hopes to include a revised version of 8276 in Update V

Phase I – Initial Demonstration of Proficiency – completed December 2008

Phase II – Validation for groundwater, soil, and sediment – completed November 2009

Phase III – Validation for Fish Tissue – **Completed in March 2012**

Phase III Summary: 8 laboratories participated in validation study for fish tissue

- % Recovery for QC samples ranged between 45-150%
- %RSD for fish tissues ranged from 3-33%, depending on the congener

For more info contact Shen-yi Yang at: Yang.shen-yi@epa.gov

Accuracy

Range of % Recovery for Laboratory Control Sample

Analyte	Minimum	Maximum
Toxaphene	84.1	150
Hx-Sed	77.5	113
Hp-Sed	74	116
P26	47.5	116
P40	64.5	115
P41	85.4	122
P44	57.5	120
P50	80.7	112
P62	45.8	125
PCB 209*	77.1	107
eHCH*	76.3	113
*Surrogate		

Precision

Relative Standard Deviation for Two Fish Tissue Samples

Analyte	TC1	TC2
Toxaphene	16.7	17.2
Hx-Sed	6.65	7.75
Hp-Sed	5.59	6.37
P26	4.62	2.71
P40	9.37	4.57
P41	3.6	2.53
P44	7.28	7.4
P50	7.25	4.87
P62	31.5	33.3

Update V 18 New and Revised Methods

Fall 2012

Method Number	Method Title
1313	Liquid-Solid Partitioning as a Function of Extract pH using a Parallel Batch Extraction Procedure
1316	Liquid-Solid Partitioning as a Function of Liquid-to-Solid Ratio in Solid Materials using a Parallel Batch Extraction Procedure
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
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) Determinations For Dioxin-Like Chemical Activity with the CALUX® Bioassay

Update V 18 New and Revised Methods

Fall 2012

Method Number	Method Title
5021	Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis
6010	Inductively Coupled Plasma-Atomic Emission Spectrometry
6020	Inductively Coupled Plasma-Mass Spectrometry
6800	Elemental and Speciated Isotope Dilution Mass Spectrometry
8000	Determinative Chromatographic Separations
8276	Toxaphene and Toxaphene Congeners by Gas Chromatography/Negative Ion Chemical Ionization Mass Spectrometry (GC-NICI/MS)
9013	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
9016	Free Cyanide in Water, Soils and Solid Wastes by Microdiffusion

Update V Key Features and Changes to SW-846

- **ORCR Policy Statement**

- Clarifies terms used in SW-846 (e.g., Final Method, Validated Method, Revised Methods)

- **Important Changes to Update V Methods:**

- **Revised** – Initial Demonstration of Proficiency (IDP)
- **Replaced** the method detection limit (MDL) with **the lower limit of quantitation (LLOQ)**
- **Improved** overall method formatting for consistency with new SW-846 methods style guidance
- **Addition** of RSE (Relative Standard Error) as an acceptable calibration model
- **Revised** Chapter One

- **Major Revisions Include:**

- **8000**

- Items regarding instrument maintenance were condensed and streamlined for ease of reference

- **6010/6020**

- Revised description of spectral interference check procedures
- Revisions to the section on matrix interference check samples: the post-digestion spike is recommended if a high concentration sample is not available for performing the dilution test

- **5021**

- Included response column and a classification system for analytes to provide an indication of which VOC responses were improved by the matrix modifier
- Added an alternative for calibration standard preparation to Sec. 7.4.1 that allowed for multiple calibration levels prepared by adding different volumes of one or more stock solutions



Update V Status

It's a Process....



Management Reviews

Who:

- ORCR Management
- EPA Offices Review
- AA Notification and Program Office Review
- ORCR Final Briefing and Review

What:

- Nine Communication Documents
- Chapters and Methods
- Notice

Package Finalized

- Docket Entries
- Change Website

ORCR Office Director Signature

- Federal Register Notice and Comment
- Notify Stakeholders



Where Do We Go From Here?

Our Priorities/Your Priorities

Update VI	<ul style="list-style-type: none">• Update more methods.• Evaluate the impact of and provide possible solution for the supply shortage of a calibrant needed for the analysis of PCBs, dioxin/furan using HRMS and the helium shortage for GC methods.• Resolve the preservation issue for vinyl chloride and styrene.• Collaborations with the ACIL work groups to improve timeliness regarding the issuance of methods.
Collaborations FEM ACIL Superfund DOD	<ul style="list-style-type: none">• Collaborate with USGS to develop a new extraction procedure for hexavalent chromium.• Participate on Four FEM Action Teams.• Review other EPA program methods.• Further develop method 9200 (lead bioavailability) for inclusion in SW-846.• Collaborate with EPA SPC, program offices, DoD, and external partners for possible method and standards development.
Method User Requests	<ul style="list-style-type: none">• 48 items and counting (some require a rulemaking)



Summary

2012 and Beyond.....

Toxaphene Phases I, 2 and 3 Complete.

Update V is Prepared and is Undergoing a Series of Reviews.

Methods Team Homepage Will Have a New Look and Feel.

Next Phase, New Update Package, New Method Development Activities and , Collaborative Efforts



Resources

Methods Information Communication Exchange (MICE)

MICE provides a valuable service to the public by answering questions and taking comments regarding "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)."

The MICE Service is maintained by the EPA Office of Resource Conservation and Recovery and operated by TechLaw, Inc., utilizing chemists, sampling experts, and environmental scientists knowledgeable in SW-846 procedures.

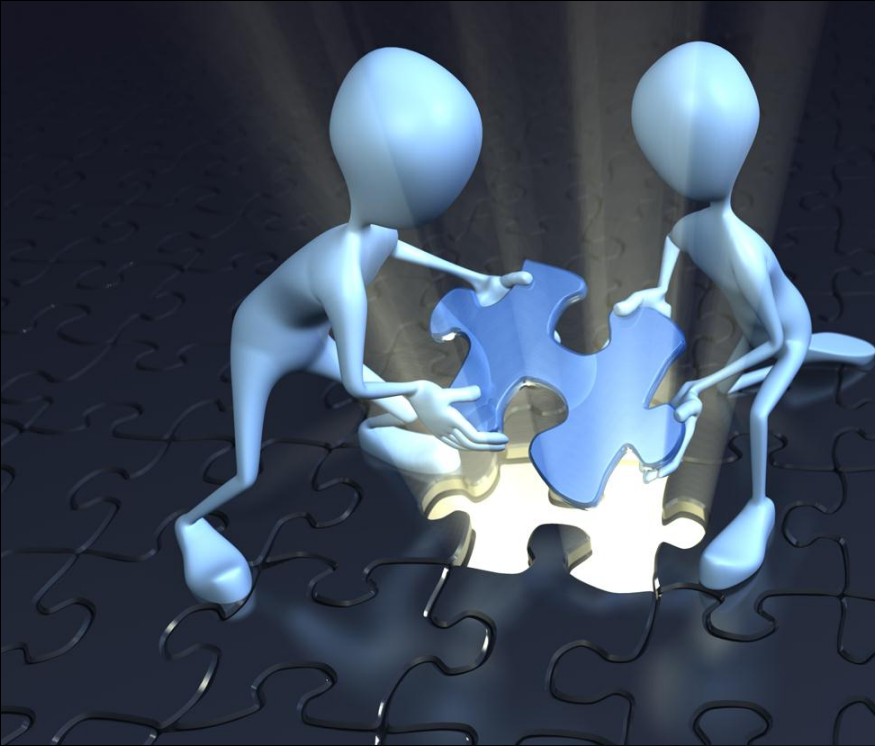
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A It's a TEAM Effort!!!

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