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EVOLVING WITH CHANGES IN ENVIRONMENTAL ANALYSIS

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INTRODUCTION

The environment is constantly changing as a result of increased human industrial and agricultural activity, and environmental regulations and analytical methods must keep apace. As this new e-book shows, adapting to change requires some effort but also presents opportunity. As regulations change, environmental laboratories can update outdated technology, adopt more-efficient sample preparation procedures, optimize common laboratory practices, and possibly also reduce operating costs.

In the first article, the authors describe how one environmental services laboratory implemented strategies to update its capabilities to adapt to changing needs and regulations in environmental analysis. The lab faced challenges stemming from updated state regulations that required 10-fold lower minimum reporting limits for semivolatile organic compounds (SVOCs) in groundwater. They responded by updating instrumentation to meet the new requirements. In the process, they also streamlined their complex sample preparation procedures and cut their use of methylene chloride in half.

Regulatory methods for volatile compounds (VOCs)—a particularly challenging class of analytes—are also evolving, as our second article in this e-book explains. U.S. Environmental Protection Agency (EPA) Method 524 for VOC content in drinking water was updated in 2009 and 2011. Changes to Method 624 for VOCs in wastewater were proposed in 2015 and updates to Method 8260 for VOCs in hazardous waste, soil, semisolids, and groundwater are pending final review. New technologies can help analysts not only adapt to these evolving regulations but also address challenges in VOC quantitation such as purge efficiency issues, carryover considerations, moisture management, trapping efficiency, and data interpretation.

We close the e-book with an interview with Shen-Yi Yang of the US EPA. Yang is the agency's organic methods program manager and methods team lead for the Resource Conservation and Recovery Act program. She outlines the agency's criteria for method flexibility and talks about current updates to the SW-846 methods for sampling and analyzing solid waste. In particular, she shares details about updates that are being incorporated into Methods 8260, 8270, and 1340.

We hope you enjoy this new e-book, and find it helpful as your laboratory adapts to changes in environmental analysis methods and regulations.

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EVOLVING WITH CHANGES IN ENVIRONMENTAL ANALYSIS, PART 1: ARE YOU USING THE RIGHT TOOLS FOR SEMIVOLATILE ANALYSIS?

Summary of a Recent Webcast

By Dwain Cardona and Patty Schultz-Benker

Introduction

The goal of this presentation was to discuss how labs can transform the challenges of regulatory updates into opportunities to improve lab capabilities for semivolatile (SVOC) analysis. Within the discussion, specific upcoming updates to EPA SVOC regulatory methods were discussed to demonstrate the challenges that labs experience. Strategies for overcoming these challenges were then reviewed that reveal the opportunity of updating lab capabilities. Finally a collection of SVOC workflow-based solutions were explored to illustrate how lab capabilities can be improved using new technology and tailored approaches to existing challenges.

Review of SVOC Regulations

In the United States, the Environmental Protection Agency (EPA) sets regulations for monitoring contaminants in environmental matrices like water, soil, and air. The various offices provide detailed analytical methods grouped first by sample type (or office that the regulation emanated from) and then contaminant type.

Common SVOC methods include the 500, 600, and 8000 series methods, as well as the toxic organic air methods. These methods set the current regulatory standards for environmental sample analysis. However, as we all know, our environment is constantly changing. Increased human industrial and agricultural activity impact the changing contaminant picture. In response, our regulators must update regulations periodically to manage the evolving risk of contaminant threat.

Factors that influence proposed updates to regulatory methods include:

- newly produced or discovered compounds;
- improved knowledge of the health effects of contaminants;
- changes in contaminant occurrence frequency;
- changes in contaminant observed concentration.

It is important to note that changes in observed concentration of contaminants could be due to changing environmental conditions or even advances in the sensitivity of our instruments. Additionally, changes in the feasibility or the cost of monitoring and/or of treatment technologies can influence regulations.

Thus, proposed regulatory method updates may include the use of alternative sample-extraction technologies, such as solid-phase extraction (SPE), or the inclusion of technology advancements that add additional flexibility to methods like triple quad or other instrumentation. Other areas of proposed updates include reduced detection limits, clarified requirements, and sometimes brand new methods altogether.

One of the recent proposed method updates aligned with these examples is the update to Method 525.2 for SVOC content in drinking water by GC–MS. The proposed update to Method 525.3 includes improvements to sample preparation practices as well as the allowance of enhanced instrument technology; it was finalized in 2015.

Two other examples include the 2015 method update rule that proposes revisions to 625 for SVOC and 608 for organic chlorine and PCBs analysis in wastewater as well as the proposed update to SW-846 version VI that includes updates to 8270E for SVOC by GC–MS. In the past two years, both updates have been proposed with finalized comment collection periods and are currently in the comment review period. The output of both updates has not been finalized, but will be prepared when the review of comments is completed. A recent status update from the EPA at the Department of Defense's Environmental Monitoring and Data Quality Workgroup workshop in April indicated that the agency plans to release the SW-846 update in three phases with phase one dedicated to organic methods (8270E).

In both of these examples, the EPA is taking steps to include new technology for extraction and analysis, which

may add flexibility to the methods while maintaining the current method requirements. We have also observed the EPA attempting alignment or harmonization of SVOC methods between the offices of drinking water (500s), waste water (600s) and resource conservation and recovery (8,000s).

The harsh reality of implementing regulatory updates is that method updates can create challenges for laboratories. For environmental labs, changes in routine lab operation can be disruptive. Regulatory method changes may require updates to existing standard operating procedures (SOPs), new analytical methods (which require validation prior to implementation), additional instrumentation/software, new report templates, and, finally, additional staff training requirements.

In any case, activity associated with method changes require work and time that might be better spent reviewing data. However, failure or delay to adopt updates presents other potential risks to our organizations. Samples could be misrepresented or incomplete due to outdated calculations or procedures. Lab errors could lead to accreditation issues or qualification concerns. Failure to adopt updates could also make contract labs less competitive and ultimately result in the loss of customers. Fortunately, by adopting the updates, labs cannot only avoid these consequences, but they can also use these updates as opportunities to improve laboratory capabilities.

Strategies for Overcoming the Challenges of Method Updates

To overcome the challenges that updates pose, labs can start by constructing strategies to manage the risk and impact that accompany update adoption. If we identify the requirements for updates and steps needed to achieve compliance, opportunities for lab improvement will be revealed. Examples of this can include the need to update outdated technology, adoption of more efficient sample prep procedures, optimization of common lab practices and operating cost evaluations.

Identifying the opportunity that regulatory updates create transforms obstacles into opportunities and can make change a good thing. In the next section, we've used PDC Labs to demonstrate some of the benefits that go along with updating lab capabilities. PDC Labs has implemented several strategies including updates to triple quad MS technology in replacement of their outdated single quad technology to overcome some of the obstacles that they have encountered.

PDC Labs Case Study

PDC Labs is a full-service environmental analytical lab established in 1981 that analyzes groundwater, drinking water, wastewater, solids in soils, solid waste, and storm water. In the main lab in Peoria, Illinois, the sample load is between 10,000 and 11,000 samples a month in all the different matrices for which they use the traditional 500, 600, and 8,000 series methods.

The Organic Analysis Department uses traditional standalone GCs, 12 GC–MS systems for volatiles and semivolatiles, and a high-resolution mass spectrometry (MS) system for dioxins and furans. Five of the GC–MS systems are Thermo Scientific™ ISQ™ single quads, one system is a triple quad, and the remainders are outdated Agilent single quad models. The department uses the Thermo Scientific™ Xcalibur™ Software, the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) Software, and Target. PDC started acquiring and replacing the outdated Agilent GC–MS single quad instruments with Thermo Scientific™ single quad and triple quad GC–MS instruments in late 2012.

In their lab, they faced challenges stemming from updated state-based regulations that required ten-fold lower minimum reporting limits (MRLs) for SVOCs in groundwater. With their previous instrumentation, they were unable to meet the minimum detection limits for chlorinated pesticides and PCBs. In addition to this analysis, they were also using multiple methods including 8141 for NT pesticides, 8081 for chlorinated pesticides, 8082 for PCBs, 550 PNAs by HPLC, 531 for carbamates by HPLC as well as several other SVOC methods. Indeed, to cover the broad range of analytes and methods four separate extractions were required for each sample making complexity of the process considerable.

Recently, PDC constructed an action plan to increase the productivity, efficiency, and profitability of the lab by combining SVOC methods. Using updated MS technology with selected reaction monitoring (SRM), they were able to achieve the lower minimum detection limits (MDLs) for PCBs and chlorinated pesticides. The Thermo Scientific™ TSQ™ 8000 Evo triple quad was used for simultaneous full scan/ SRM data collection to achieve both traditional and new method objectives for SVOC analysis. In addition to the MS, an ECD detector was added as a second confirmatory detector for the multi-component compound patterns of PCB, toxaphene, and chlordane. The TSQ 8000 also features a variable emission current, which enabled a varying of the degree of ionization to optimize detector response and sensitivity.

Sample preparation practices were also modified using a combination of SPE technology and extensive liquid/ liquid extraction development to combine the extraction procedures for multiple compound types. Optimizing this process drastically reduced solvent volumes and sample preparation durations.

The lower MRL requirements expanded calibration ranges from 0.5 ppm to 100 ppm for full scan data collection and 0.05 ppm to 10 ppm for SRM data collection. The average response factor for Endrin was 0.08 ppm in full scan mode and five times higher at 0.44 ppm in SRM mode, illustrating the increased sensitivity using SRM.

The remainder of the compound types were combined using the Thermo

AQUEOUS MDL COMPARISON

	Agilent * (µg/L)	ISQ (µg/L)
bis(2-ethylhexyl)Phthalate	2.66	0.40
Benzo(a)pyrene	1.90	0.13
4-Nitroaniline	1.51	0.21
2,4-Dinitrophenol	9.47	0.55
Pentachlorophenol	5.83	0.53

* average of three instruments 5971, 5972 and 5973

Figure 1: PDC: MDL - Old vs New Technology.

Scientific™ ISQ Single Quadrupole for method 8270. A comparison of previously achieved MDLs obtained using the outdated Agilent equipment with those obtained using the new ISQ instrumentation show a factor of 10 increase in sensitivity (**Figure 1**). This enabled the lab to meet the new regulatory limits for Illinois ground water analysis. In fact, the data indicated that even lower limits could be confidently achieved.

It was necessary to maintain accreditation during the process of technology and methodology update. Time-consuming full data validation was required. The lab had to successfully complete two proficiency-testing studies. SOP updates and newly authored SOPs were also needed to document and control the changes to the methods.

Many features aided in the update process, but one worth mentioning was the vacuum interlock capability of the Thermo mass spec instruments. The vacuum interlock allows source maintenance without having to vent or cool the system. Also, the filaments are conveniently separate from the source, which reduces the frequency of filament replacement. Using the Agilent MS, filaments would have to be replaced during source maintenance due to the configuration of the source and the need to vent the MS an additional time should a filament fail outside of the scheduled maintenance timeframe.

Another feature that aided in the lab's efforts to update methods was the Auto SRM Software. This software is a built-in program that guides users through transitioning single quad methods to triple quad by automating the SRM transition identification step of method development. In addition to the method development automation, the results of the Auto SRM development studies are easily exported directly into the instrument and processing methods. This feature specifically made transferring methods from single quad technology to triple quad technology very easy.

In summary, the changes and update of MS instrumentation combined with the updated procedures have changed the daily operations of PDC Labs. Because of the updates and the ability to combine various methods into a single analysis, they can now offer much more competitive pricing. Also many more samples can now be processed with fewer people and their use of methylene chloride has been cut in half, resulting

in not only savings but also less risk of exposure to a known health detrimental solvent.

Tolls that Improve Lab Capabilities

Updating methods and instrumentation in our labs can be a real challenge. At Thermo Fisher Scientific, we want to make this process as seamless as possible. For environmental labs specifically, Thermo Fisher Scientific is one of the few solution providers that offers tools that support the entire workflow for environmental labs from start to finish.

Starting with sample extraction, our products include SPE cartridges for manual or automated sample extraction. Simply moving from liquid–liquid extraction to SPE cartridges provides a significant reduction in solvent usage and exposure. The Thermo Scientific™ Dionex™ ASE™ system or the Thermo Scientific™ Dionex™ AutoTrace™ instrument can provide automated sample extraction for significant time-savings as well. Also provided is a large catalogue of instrument consumables, including columns and inlet liners, that can be used on our instrumentation as well as instruments from other manufacturers.

Our instrumentation portfolio consists of a wide range of instruments from modular standalone GCs, including simultaneous dual-injection capabilities, to the high-resolution magnetic sector instrumentation like the DFS required for dioxin analysis. Recently, an increasing amount of attention has been given to the Thermo Scientific™ Q Exactive™ GC Orbitrap™ GC–MS/MS instrument that features Orbitrap technology and the many benefits of high-resolution accurate mass for gas-phase analysis for the first time ever. The built-in flexibility and industry-leading capabilities allow labs to get more done with a single instrument

Perhaps one of the most important and progressive tools in an environmental lab is the software that is used to manage instrumentation and process data. Our various software packages have been designed to meet specific lab requirements including the environmental version of the Thermo Scientific™ TraceFinder™ Software and an expanded Chromeleon CDS that can now be used for GC and GC–MS instrumentation. On top of all this, we provide Laboratory Information Management Systems (LIMS) allowing labs to track samples and sample data from receipt to data reporting, making it one of the only comprehensive solutions for environmental analysis.

We are always striving to provide unique tools to improve sample analysis in each area of the environmental lab workflow. Instrument features are developed and tailored to the specific needs of environmental customers, with dedicated attention to supporting high sample load labs routinely running EPA methods like PDC Labs. For example, the modularity of the Thermo Scientific™ TRACE™ 1300 series GC offers flexibility through its user configurable detector and inlet modules. In addition to this, the

Helium Saver inlet configuration provides a lower cost of ownership by conserving helium usage during instrument operation and periods of downtime. As a final example, MS instruments like the ISQ Single Quadrupole provide improved technology that enhance method sensitivity in fact, the ISQ currently provides the lowest instrument detection limit specification on the market.

A specific solution that has been tailored for our U.S. environmental lab customers that assist in MS technology upgrade is the Thermo Scientific™ EPA 8270D Analyzer Kit Analyzer Kit. The 8270D Analyzer Kit provides a seamless solution for labs that want to migrate from the outdated instruments to new Thermo single quad capabilities. The analyzer kit removes the need for new method development offering a validated methodology that includes instrument and processing methods, as well an EPA environmental specific report package. All components of the kit were developed to fulfill the EPA 8270 method requirements. The method setup provides a single system configuration that can be used for both high concentration and low concentration samples reducing the need for additional columns or changes to the system.

Summary and Conclusion

To wrap up, in this discussion, we reviewed regulations, regulatory updates and how they can affect your labs by creating challenges. Patty at PDC Labs helped us construct strategies for dealing with the challenges created by regulatory updates and provided guidance for taking advantage of the opportunities that are created to improve lab capabilities. We also reviewed some tools for that can be used in those strategies like the 8270D Analyzer Kit and updated MS technology. Overall, by identifying the opportunity that obstacles create labs can use these strategies and tools to improve practices and enhance productivity.

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EVOLVING WITH CHANGES IN ENVIRONMENTAL ANALYSIS PART 2: MEETING EVOLVING REGULATORY REQUIREMENTS OF EPA METHODS FOR MONITORING VOLATILE COMPOUNDS

Summary of a Recent Webcast

By Dwain Cardona and Anne Jurek

Introduction

Environmental sample analysis is a constantly evolving field. Changes in the environment and the contaminants present are mirrored by updates to regulatory guidelines and methodologies. For environmental labs, the challenge of staying up to date with regulatory requirements and maintaining productivity is a balancing act. This manuscript discusses the evolution of regulatory requirements for volatile organic compounds (VOCs) focusing on recently proposed updates and some of the typical challenges experienced in VOC analysis. The discussion also provided guidance for navigating these challenges using solutions that can be incorporated into plans for updating instrumentation. Also featured in this article is a review of the available solutions that can help improve current lab practices and offer additional opportunities to lower operating costs and optimize capabilities for VOC analysis.

Volatile Organic Contaminants Analysis Background

Volatile organic contaminants are organic contaminants that have low molecular weights, low boiling points, and high vapor pressures. Because of these properties, VOCs tend to readily evaporate and equilibrate with earth's atmosphere at ambient temperature, hence, the label "volatile". Common sources of these compounds include industrial solvents, paints, adhesives, and petroleum products. Another major source of VOCs are emissions from automotive and industrial activity, which has been the recent focus of EPA activity. Because of their assortment of chemical properties and the volatile nature of these compounds, analysis of VOCs is accompanied by frequently occuring challenges. VOC analysis is particularly challenging because the properties of the target compounds constantly drive their migration out of sample extract or matrix prior-to or during analysis.

Among other considerations, volatility challenges are dealt with in two ways: airtight sample containers with limited headspace must be used to capture samples and analysis methods require capabilities that efficiently drive vaporization, capture and transfer of the analytes out of matrix and into instrumentation without diffusive loss. To sample the evolved vapors or gases from environmental samples, either headspace or purge and trap techniques are used. Since in the environment these compounds are constantly equilibrating into the atmosphere, samples tend to be at lower concentrations. Consequently, purge and trap is the preferred technique for quantitative analysis due to its ability to concentrate low concentration analytes, while headspace is primarily used as a screening methodology. With purge and trap (**Figure 1**), a gas is used to purge VOCs out of the sample and concentrate them onto an analytical trap column. Note: This is separate from the analytical GC column used to resolve the components. The trap column is then heated to elute the VOCs from the trap column. Carrier gas flow then sweeps the unbound VOCs into the GC or GC–MS system for analysis. In addition to providing a tool to concentrate low-concentration VOCs, the purge and trap concentrator (analytical trap) provides an avenue to limit the introduction of water to instrumentation. Typically, large amounts of water sample must be concentrated to meet VOC detection limits, which can produce challenges to the operation and functioning of the GC and GC–MS systems. Another key function of the purge and trap technique is that it filters out the volatile portions of the sample, leaving behind the heavier sample artifacts and potential matrix interferences.

Environmental VOC Regulations

In the United States, the EPA sets regulations for monitoring the presence of VOCs in environmental matrices

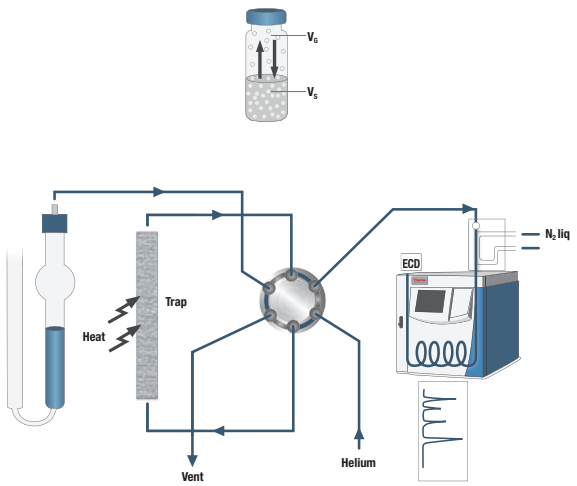

Figure 1: A general purge and trap schematic.

like water, soil, and air including the analytical methods that detail how to extract, identify, and quantitate these contaminants. There are many different considerations for method groupings. Generally, the method series for VOCs are broken up by sample type (or office that the regulation emanated from) and the type of contaminants to be analyzed. Common VOC methods include the 500, 600, and 8000 series methods, as well as the toxic organic air methods. These methods are the current regulatory standards for VOC analysis and typically include some type of GC or GC–MS analysis.

Gas chromatography methods for VOC analysis can include BTEX/MTBE, gasoline range organics, or total petroleum hydrocarbons. These methods are usually configured for a single compound analysis or small compound groups. In contrast, the GC–MS methods include an assortment of VOC types where spectra are used to differentiate between various compounds. Regulatory GC–MS-specific methods include EPA Methods 524, 624, 8260, and their associated versions.

Regulators update these methods periodically to manage new risks and/or revamp outdated methods with new requirements. For example, method 524 for VOC content in drinking water using GC–MS was updated in 2009 and again in 2011. In 2015, updates were also proposed, via the Method update rule, for U.S. EPA Method 624 for VOCs in wastewater. Additionally, the SW-846 version 6 update, still in its comment review and feedback period, included updates for EPA Method 8260 for the determination of VOCs in hazardous waste, soil, semi-solids, and groundwater.

Overall it seems the EPA is optimizing method performance by providing more flexibility in analytical procedures and taking steps to harmonize similar methods between offices. Specific updates include the addition of new technology, the use of hydrogen as a carrier gas, and additional analytes.

A specific benchmark for EPA updates to VOC methods where these efforts are demonstrated are the 2009 update for 524.3 and the 2011 update for 524.4. Changes to these methods included the addition of nitrogen as a purge gas, edits to instrument method parameters including data collection, shorter purge and desorb times for purge and trap

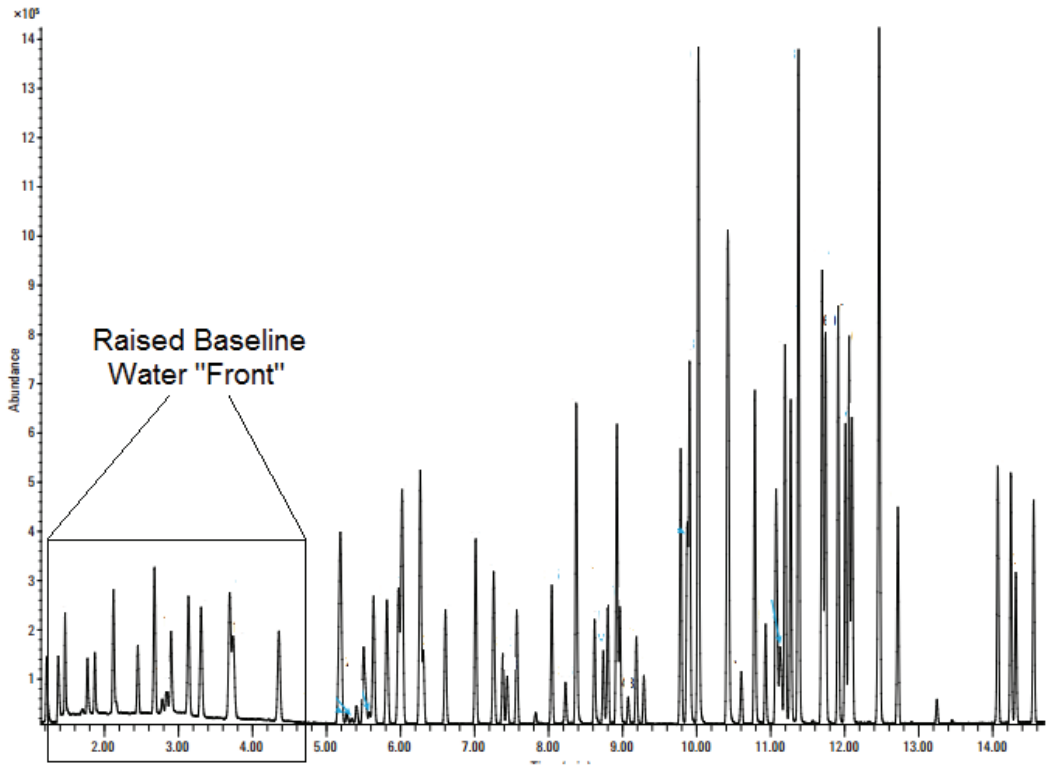

Figure 2: Moisture control - water front example.

sample introduction, edits to data processing and adjustments to Lowest Concentration Minimum Reporting Level (LCMRL) calculations.

Despite these updates, many challenges remain, including the addition of new compounds, purge efficiency issues, carryover considerations, moisture management, trapping efficiency, and data interpretation. Without the proper technology and tools, these challenges can lead to inaccurate quantitation, the presence of false positives, reanalysis requirements, difficulty with sample availability, and an overall decrease in lab productivity.

Answering Challenges with Tools That Evolve with Regulations

Moisture effects are a challenge that can cause a ripple effect in purge and trap GC–MS analyses resulting in a water "front" in the chromatography and internal standard inconsistencies, both of which can cause calibration problems. **Figure 2** illustrates the effects of a raised baseline due to a water "front" caused by moisture present in the GC–MS. If

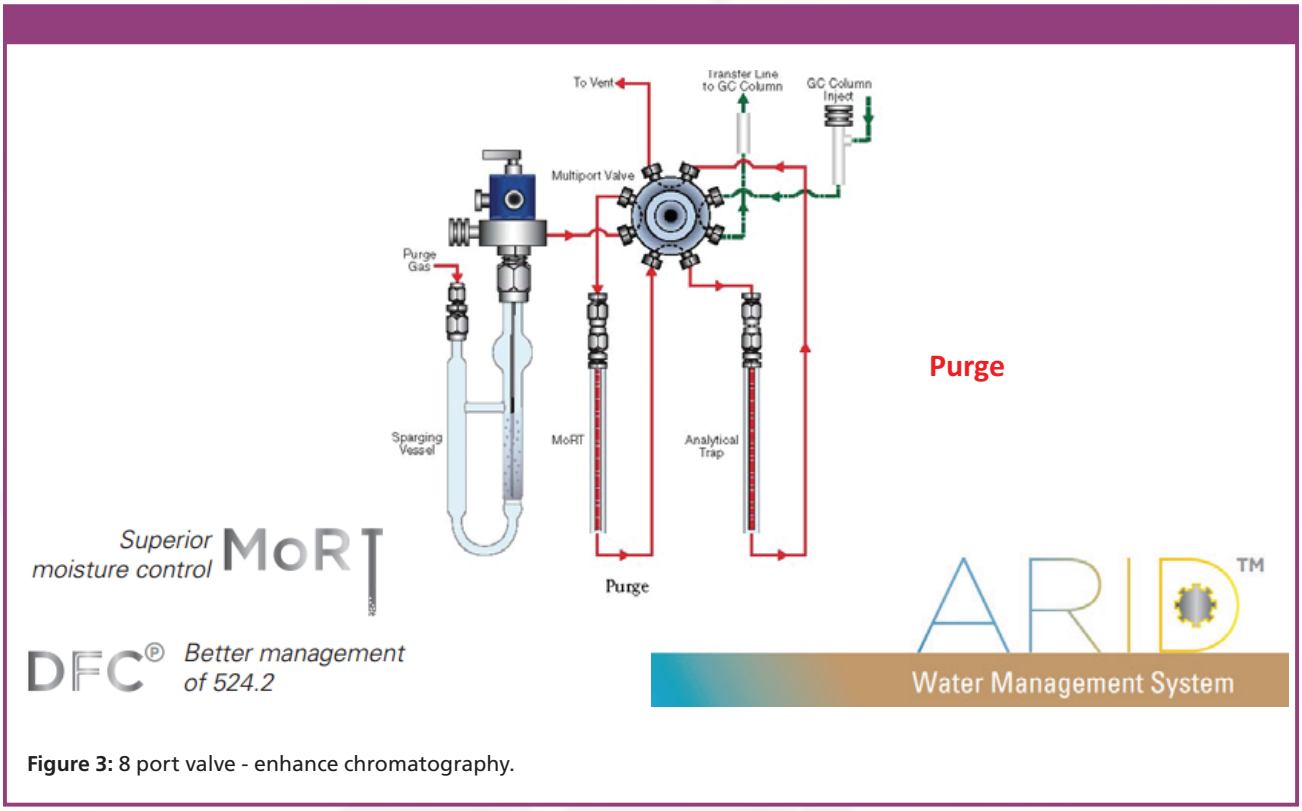

Figure 3: 8 port valve - enhance chromatography.

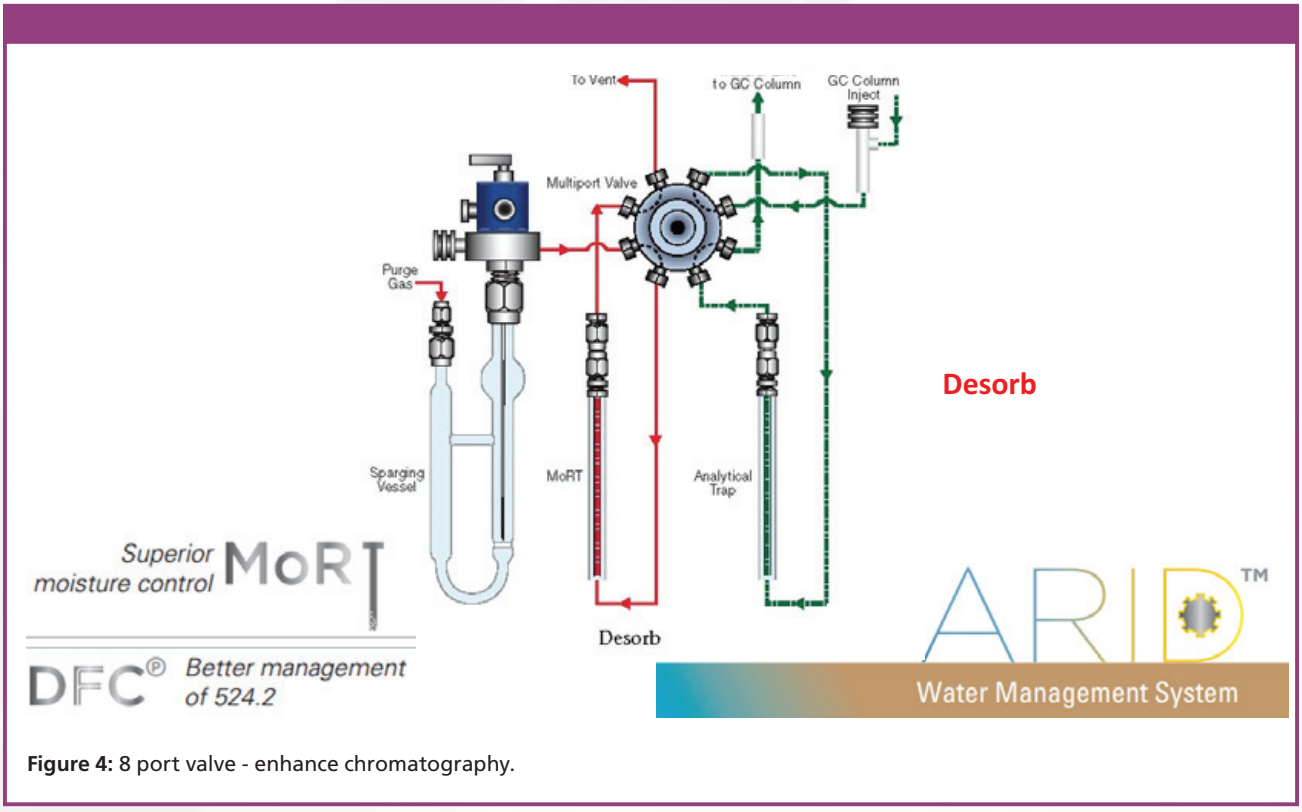

Figure 4: 8 port valve - enhance chromatography.

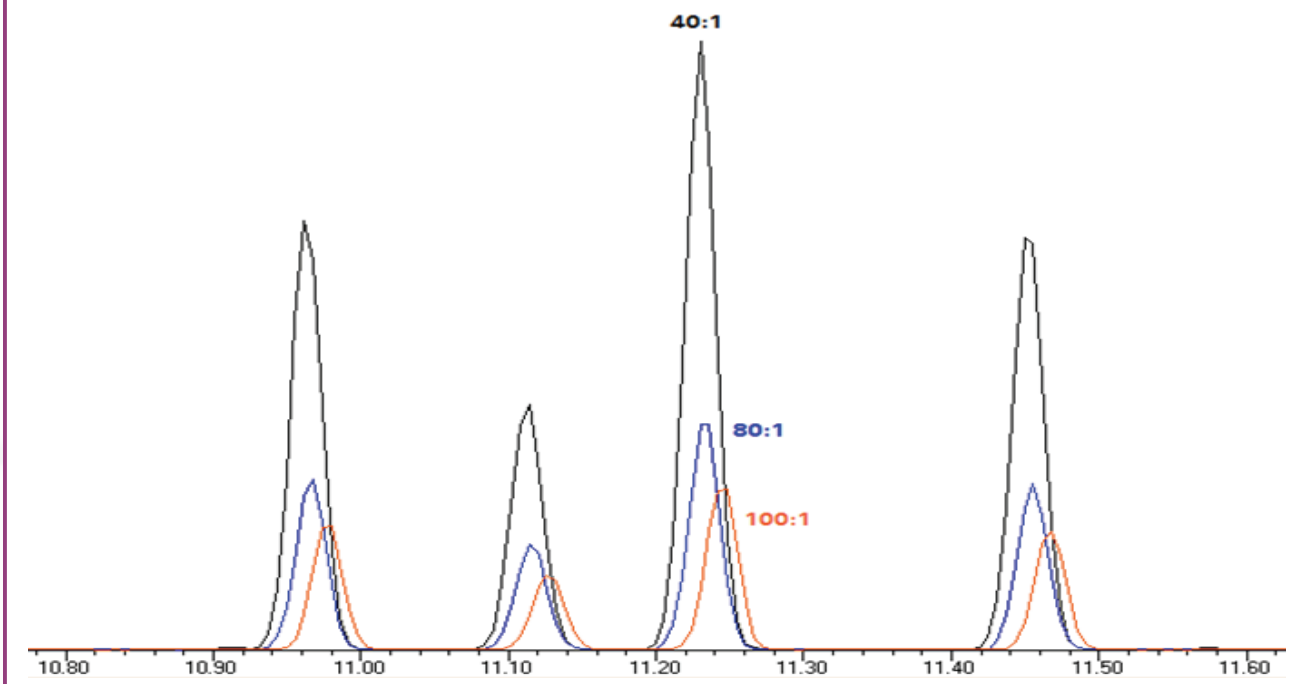

Figure 5: Split ratio chromatogram.

internal standard recovery is variable due to moisture contributions, compound recoveries may fail the acceptance criteria. There are four ways to avoid moisture effects: smaller sample volumes, shorter desorption times, a moisture trap in the concentrator, and higher GC inlet split ratios.

Modern GC–MS instruments, with improved sensitivity, can achieve the desired detection limits using a reduced sample volume (5 mL) as opposed to the 25 mL volume traditionally used, introducing less moisture. Shorter desorption times are also helpful in reducing moisture effects. While method 524.2 required a four-minute desorption, 524.3 requires only 0.5 to one minute. A half-minute desorption results in a substantial decrease in the amount of moisture introduced to the instrument. Another way to reduce moisture is by using a blank tube that does not contain any adsorbents, called a moisture retention trap (MORT). The MORT accumulates water, allowing the analytes of interest to pass through and adsorb to the analytical trap, as shown schematically in **Figures 3** and **4** for the purge and desorption steps respectively. By using an 8-port valve, the sample pathway avoids the moisture trap completely during the desorption step, resulting in better moisture control.

Moisture control can also be managed by using higher split ratios. Traditionally,

split ratios up to 100:1 have been recommended, which is great for controlling moisture. However, a side effect of a 100:1 split is higher helium consumption. Using moderate split ratios, like 40:1, is a good compromise between helium use and moisture control, and as presented in **Figure 5**, better sensitivity is obtained.

Methanol creates yet another challenge; excess methanol can cause linearity problems and compound response suppression, particularly in the more sensitive modern GC–MS instruments. In addition, other compounds commonly analyzed can be problematic for example; ethanol and 1,4-dioxane, both of which are becoming more common in VOC analyses. These compounds are miscible in water and are difficult to purge and remove from the sparging vessel after a sample is run. Furthermore, U.S. EPA Method 8260 requires System Performance Check Compounds (SPCCs). SPCCs are used to determine if there is a problem with the operation of the purge and trap concentrator. The minimum average response factors for SPCCs are determined for chloromethane (0.100- low response if purge is too fast), 1,1-dichloroethane (0.100-low response if transfer line is degraded or contaminated), bromoform (0.100-low response if there is poor purging), chlorobenzene (0.300) and 1,1,2,2-tetrachloroethane (0.300-low response if transfer line is degraded or contaminated) to ensure compound stability and to assess degradation caused by contaminated lines or active sites within the system.

Finally, compound carryover can also create difficulty for VOC analysis. Carryover after high-concentration sample runs can result in sample re-runs and a corresponding loss of productivity. There are multiple solutions for preventing carryover including higher bake temperatures, higher bake flows, hot water and/or methanol sparge vessel rinses. However, the most effective technique of controlling carryover is to heat the sparge vessel during bake to baking-off any remaining analytes in the glassware.

Workflow Solutions for VOC Analysis

From VOC sample receipt to data analysis, the Thermo Scientific environmental analysis portfolio integrates a broad selection of instrumentation and solutions to support this workflow. For screening samples, headspace capabilities include both syringe and loop-type configurations featured in the Thermo Scientific™ TriPlus RSH™ and Thermo Scientific™ TriPlus™ 300 Headspace Autosamplers. In purge and trap applications, Thermo Scientific's GC and GC–MS products integrate with most manufacturers' concentrator instrumentation. In addition to a complete selection of GC and GC–MS capabilities, Thermo Fisher also provides chromatography consumables

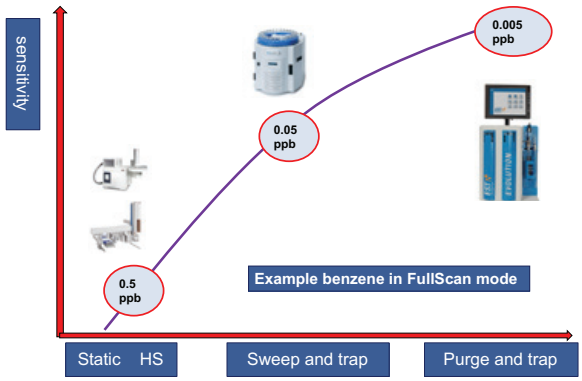

Figure 6: The sensitivity for benzene is greatly increased with purge and trap instrumentation.

Figure 7: Helium saver module for the lifetime of the instrument.

like inlet liners, septa and ferrules and last but not least they also supply specific environmental data analysis tools in Chromeleon Chromatography Data System (CDS) Software and TraceFinder Software options.

When updating or choosing new instruments, it is important to keep analysis goals in mind. For VOC analysis, the required sensitivity often dictates the choice of technology. As illustrated in **Figure 6**, sensitivity for benzene is greatly increased by moving from headspace to purge and trap instrumentation. Consequently, the detection limits and required reporting limits of the analysis need to be considered while also taking instrument capabilities into account. The TriPlus RSH system includes a dedicated gas syringe tool and heating block for headspace vials as well as auto-dilution functionality with the added available option of solid-phase micro extraction (SPME) configuration. By adding the SPME feature, a drinking water lab could analyze Geosmin and 2-Methylisoborneol compounds along with headspace samples, providing a dual-purpose instrument that could replace two older systems. Along with the autosampler capability, the Thermo Scientific™ TRACE™ 1300 Gas Chromatograph series includes dual detectors and dual injector modularity that can be configured to run simultaneous methods further improving productivity.

In addition to its configurable instrument features, the additional options of the Trace1300 series GC provide the opportunity to reduce laboratory operating costs. Because environmental labs have experienced increases in helium costs and decreases in availability helium conservation has become a key focus for reducing analysis costs. When using purge and trap for the extraction of VOCs, a large amount of helium is consumed. By using nitrogen as a carrier gas for purge and trap extraction labs can significantly reduce

the amount of helium consumed in a single run. In addition, the modularity of the TRACE 1300 GC system delivers a solution for reducing Helium usage during GC sample analysis. Using the Helium Saver Inlet Module, shown installed on a Thermo Scientific™ TRACE™ 1310 Gas Chromatograph in **Figure 7**, nitrogen blankets the inlet during injection and periods of downtime. Helium use is reserved for use only as a carrier gas to transfer VOC analytes through the column. When used in combination with nitrogen on purge and trap instrumentation, as allowed in EPA Method 524.4, overall helium volumes can be drastically reduced to as little as a single cylinder of helium over 15 years.

Another effort to help conserve helium is proposed in edits to EPA Method 8260, which includes the option for hydrogen as a carrier gas. In our evaluations using hydrogen as a carrier gas caused catalytic compound conversions and reduced overall sensitivity of the system over time. Along with the need for revalidation, which can be expensive and time-consuming, hydrogen carrier gas also comes with significant safety concerns. By using nitrogen as a purge and trap gas only, and helium as a carrier gas in conjunction with the Helium Saver Module for GC analysis, concerns for hydrogen carrier gas are eliminated allowing maintained productivity and reducing operating costs.

Conclusion

The regulatory landscape of VOC analysis is constantly changing, and while there are many challenges for laboratories to overcome, there are many tools and workflow solutions that can help reduce errors, improve productivity, and reduce operating costs.

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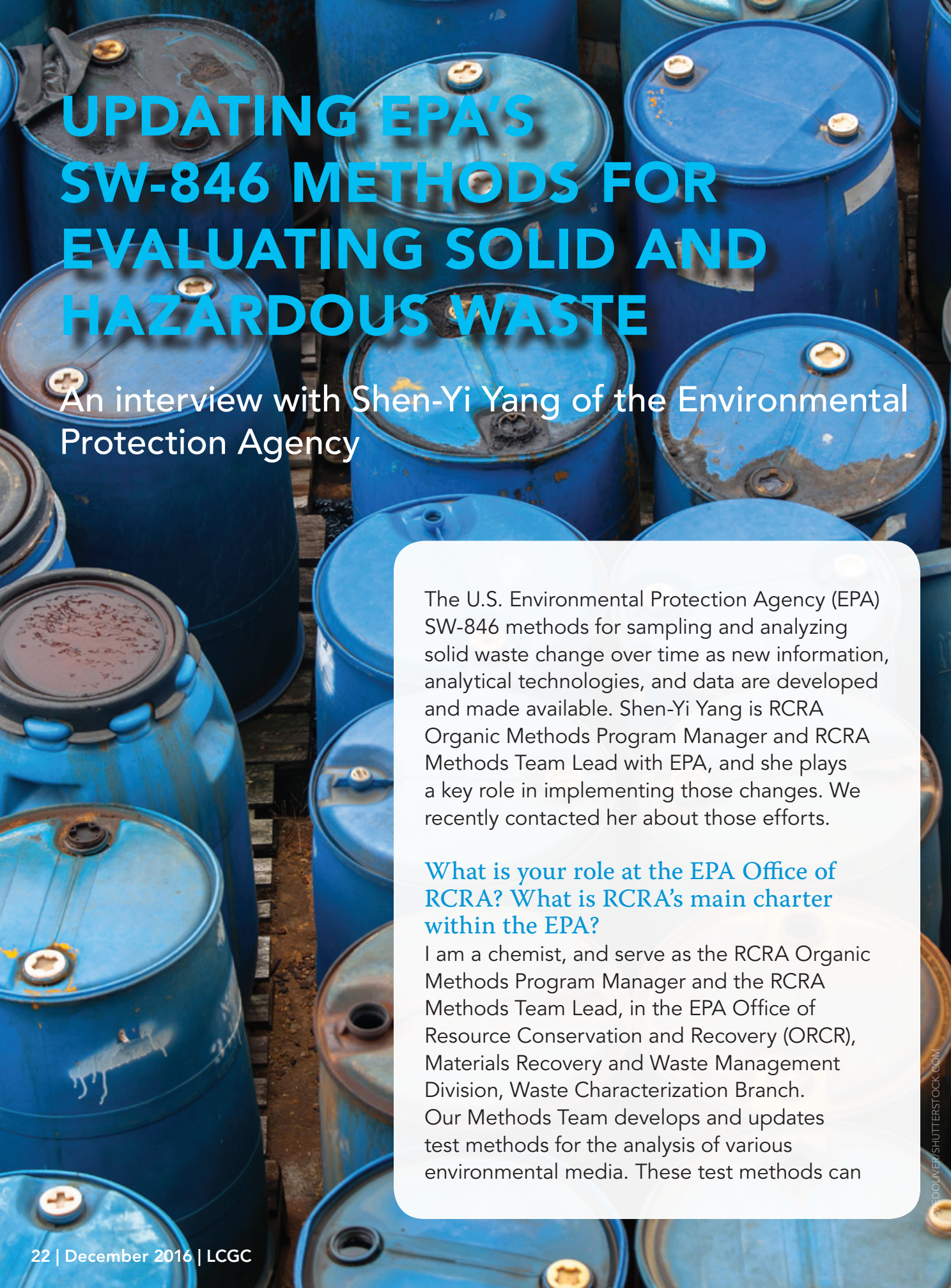
UPDATING EPA'S SW-846 METHODS FOR EVALUATING SOLID AND HAZARDOUS WASTE

An interview with Shen-Yi Yang of the Environmental Protection Agency



The U.S. Environmental Protection Agency (EPA) SW-846 methods for sampling and analyzing solid waste change over time as new information, analytical technologies, and data are developed and made available. Shen-Yi Yang is RCRA Organic Methods Program Manager and RCRA Methods Team Lead with EPA, and she plays a key role in implementing those changes. We recently contacted her about those efforts.

What is your role at the EPA Office of RCRA? What is RCRA's main charter within the EPA?

I am a chemist, and serve as the RCRA Organic Methods Program Manager and the RCRA Methods Team Lead, in the EPA Office of Resource Conservation and Recovery (ORCR), Materials Recovery and Waste Management Division, Waste Characterization Branch. Our Methods Team develops and updates test methods for the analysis of various environmental media. These test methods can

be found in the EPA publication *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods,* also known as SW-846.

The Resource Conservation and Recovery Act (RCRA), enacted in 1976, is the principal federal law in the United States governing the disposal of solid waste and hazardous waste, and it creates the framework for the proper management of hazardous and nonhazardous solid waste. The law describes the waste management program mandated by Congress that gave EPA the authority to develop the RCRA program that protects communities and promotes resource conservation. To achieve this goal, EPA develops regulations, guidance, and policies that ensure the safe management and cleanup of solid and hazardous waste, and programs that encourage source reduction and beneficial reuse. On the EPA website, you can find an overview of the RCRA Act and information about RCRA's critical mission and the path forward.

In April 2014, EPA Administrator Gina McCarthy presented the U.S. Environmental Protection Agency FY 2014–2018 Strategic Plan, which charts U.S. EPA's course for protecting public health and the environment in every community in America during the next four years. The four objectives (that is, to promote sustainable and livable communities, preserve land, restore land, and strengthen human health and environmental protection in tribal lands) of Goal 3 (Cleaning Up Communities and Advancing Sustainable Development), in particular, are the main charter of RCRA. See https://www.epa.gov/sites/production/files/2014-09/documents/epa_strategic_plan_fy14-18.pdf for more information; see the Goals and Objectives section of the EPA 2014–2018 Strategic Plan.

What current method updates are you involved with that pertain to the RCRA?

The RCRA Methods Team is currently working on Update VI, which contains five new and four revised methods at various developmental stages. Five new methods (that is, Methods 1313, 1314, 1315, 1316, and 1340), are presented on EPA's SW-846 Methods website as "validated" methods. When they are ready, ORCR will post the four revised methods (that is, Methods 3050C, 6200A, 8260D, and 8270E) on the SW-846 Methods website for public comment at www.epa.gov/hw-sw846.

Can you tell us more about the EPA's criteria for method flexibility?

On October 6, 1997, the agency published in the *Federal Register* a notice of its intent to adopt the Performance Based Measurement System (PBMS), with the following goals:

- Reduce the cost of monitoring;
- Stimulate the development and use of innovative technologies;
- Speed up the introduction of new methods; and
- Improve the quality of science in the monitoring community.

In its efforts to implement PBMS, the agency determined that the "one-size-fits-all" approach for analytical methods did not work well across the four diverse environmental statutory programs (that is, the Clean Air Act, Clean Water Act, Safe Drinking Water Act, and RCRA). Therefore, in February 2008, EPA's Forum on Environmental Measurements (FEM) received approval from the Agency Science Policy Council (now named the Science Technology Policy Council) to move forward in a new direction: "Flexible Approaches to Environmental Measurement—The Evolution of the Performance Approach" (also called the Flexible Approach). This change allows the agency to make progress toward greater method flexibility without compromising its regulatory authority. The new Flexible Approach seeks to leverage those differences by allowing each program office to meet key goals within the constraints of their regulations.

Additional information regarding PBMS and the Flexible Approach can be found at the following links:

- Performance Based Measurement System (PBMS) Federal Register Notice of Intent
- Flexible Approaches to Environmental Measurement—The Evolution of the Performance Approach
- Federal Register Notice (FRN)
- Flexible Approaches to Environmental Measurements—Webinar Presentation with Notes
- Flexible Approaches to Environmental Measurement Webinar Series—Question and Answer Summary

The Flexible Approach provides many benefits to both regulators and the regulated communities, including greater flexibility in method selection, expedited approval of new and emerging technologies to meet mandated monitoring requirements, and development and use of cost-effective methods that meet program requirements and their associated performance criteria.

The FEM is committed to annual follow-up to ensure that significant progress is made, and it has documented in annual summary reports since 2009 the activities that each of the EPA program offices has undertaken to implement the key goals of the Flexible Approaches to Environmental Measurement.

EPA program offices have adopted the Flexible Approach for their programs, when appropriate. Various program documents and web pages explain their specific criteria on method flexibility for the Office of Ground Water and Drinking Water (1–3), the Office of Science and Technology (4–8), the Office of Air and Radiation (9,10), and the Office of Resource Conservation and Recovery.

The document *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846)* was initially created as a guidance for generally appropriate RCRA-related test methods. However, over time EPA published regulations that required the use of certain SW-846

methods. The Methods Innovation Rule (MIR) then revised RCRA regulations and limited the mandatory use of SW-846 methods to only those situations where the method (that is, "method defined parameter" or MDP) is the only method capable of measuring the property.

The MIR allows flexibility in method selection and use for meeting the analytical needs of the RCRA Program. 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. Thus, a method user can use another appropriate method or modify an SW-846 method—provided it is not one specifically required by regulation (that is, an MDP)— to best meet a waste matrix-specific analytical need, as long as the modifications meet the project-specific DQOs and performance acceptance criteria. The criteria may be published in regulations, technical guidance documents, permits, work plans, or enforcement orders.

You 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.

Why are these updates being implemented?

The *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods Compendium (SW-846)* document is a "living document" that changes over time as new information, analytical technologies, and data are developed and made 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 revising outdated methods. These updates address critical measurement needs, improved analytical method performance, and cost effectiveness. While we always have many methods to revise or create, we prioritize methods that address a national emergency or priority, are essential for continuing the EPA mission, are needed by the EPA Regional laboratories or other EPA program offices (such as the Office of Water), address an emerging contaminant of concern, make available new or updated technology, are collaborative efforts with other federal agencies, and provide greener chemistry alternatives or increased method safety.

Can you give us more details for method updates being incorporated into Methods 8260 and 8270 or other methods?

ORCR is preparing to release Update VI in three phases.

Phase I: Methods 8260D, 8270E, and 1340

Methods 8260D and 8270E: The two major organic methods are being revised to include analytes frequently found in Superfund sites; sample preparation

procedures (Method 3545, PFE, for 8270; Methods 5030 and 5035, for 8260) based on available supporting data; optional use of hydrogen as carrier gas to address the helium supply shortage issue; advanced measurement technologies (selected ion monitoring [SIM], chemical ionization [CI], and tandem mass spectrometry [MS/MS]); clarified language for lower level of quantitation (LLOQ) and method blanks (MB), based on comments received from the public for published Update V; and updated tuning requirements. Efforts were made to ensure consistency and possible harmonization among EPA methods.

Method 1340 (In Vitro Bioaccessibility Assay [IVBA]) for Lead in Soil: The analysis of lead bioavailability is important because the amount of lead that actually enters the blood and body tissues from an ingested medium depends on the physicochemical properties of the lead and of the medium. For example, lead in soil may exist as poorly water-soluble minerals as well as inside particles of inert matrices such as rock. These chemical and physical properties tend to influence (usually decrease) the absorption by the body (bioavailability) of lead when ingested. The extraction fluid in the method is intended to mimic gastric juices.

Phase II: Methods 3050C and 6200A

Method 3050C (Acid Digestion of Sediments, Sludges, and Soils): This revised method uses strong acid to digest almost all elements that could become "environmentally available." Elements bound in silicate structures are not dissolved by this procedure because they are not environmentally available. Poor recovery elements include certain noble or refractory elements such as palladium, silicon, tungsten, and zirconium. If total digestion is the objective, Method 3052 is recommended. New Appendix B is added to address incremental sampling.

Method 6200A (Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment): This method is revised for several technical and editorial changes. The requirement to perform a confirmation analysis by inductively couple plasma (ICP) was removed, and an optional comparability study may be performed in its place. The updated method has two modes, *in situ* for screening and *ex situ* for quantitative analysis. We also replaced old performance data with data using more-current instruments.

Phase III: LEAF Methods

Phase III is for four inorganic Leaching Environmental Assessment Framework (LEAF) methods (11) (Methods 1313, 1314, 1315, and 1316), and the User Guide. The LEAF methods were created to evaluate various conditions under which the leaching of constituents of potential concern may occur. The four

LEAF test methods each provide different information on how leaching can occur under different conditions. The LEAF Framework is then the utilization of the combined information from the different LEAF tests to evaluate possible leaching scenarios.

Method 1313 (Liquid–Solid Partitioning as a Function of Extract pH Using a Parallel Batch Extraction Procedure): This method is an equilibrium test that may allow for the evaluation of constituent leaching over a broad range of pH values. Results from Method 1313 may help determine the effect on leaching of chemical phenomena such as: aqueous solubility, mineral precipitation, adsorption reactions and redox reactions.

Method 1314 (Liquid–Solid Partitioning as a Function of Liquid–Solid Ratio for Constituents in Solid Materials Using an Up-Flow Percolation Column Procedure): This method may be used to evaluate the effect on leaching of the ability of constituents to percolate out of granular material. This test occurs in a column, and as the test is run, an increased amount of extracting solution is used versus the amount of solid granular material. Method 1314 may also be used to evaluate how constituents leach when additional chemical species are present as co-constituents.

Method 1315 (Mass Transfer Rates of Constituents in Monolithic or Compacted Granular Materials Using a Semi-Dynamic Tank Leaching Procedure): This method is a test that may evaluate the effect of the ability of constituents to diffuse out of a material on overall leaching. The material evaluated with Method 1315 can be monolithic or compacted granular material. This test is an equilibrium test that determines the rate that a constituent is released from a material over time. This rate of constituent release may be a limiting factor in the overall rate of environmental leaching for some environmental scenarios.

Method 1316 (Liquid–Solid Partitioning as a Function of Liquid-to-Solid Ratio in Solid Materials Using a Parallel Batch Procedure): This method is a test that is a series of equilibrium experiments. In these experiments, the total amount of liquid extraction solution versus the amount of solid material is varied. This method can determine equilibrium conditions such as constituent concentration, for the different ratios of liquid extractant to solid material.

References

(1) Expedited Approval of Alternative Test Procedures for the Analysis of Contaminants Under the Safe Drinking Water Act; Analysis and Sampling Procedures, *Federal Register* 81(138) 46839–46848 (2016). https://www.gpo.gov/fdsys/pkg/FR-2016-07-19/pdf/2016-16516.pdf

(2) Methods Approved to Analyze Drinking Water Samples to Ensure Compliance with Regulations https://www.epa.gov/dwanalytical-methods

(3) The Safe Drinking Water Hotline: (800) 426–4791

(4) Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures, *Federal Register* 77(97) 29758–29846 (2012) https://www.gpo.gov/fdsys/pkg/FR-2012-05-18/pdf/2012-10210.pdf#page=54

(5) *Code of Federal Regulations (CFR),* Title 40, Part 136, Section 136.6 - Method modifications and analytical requirements https://www.law.cornell.edu/cfr/text/40/136.6

(6) EPA Approval Letter on Flexibility to Modify CWA Methods,

November 7, 2007 http://www.health.state.mn.us/divs/phl/accreditation/docs/refepacwamethodflexibility.pdf
(7) Guide to Method Flexibility and Approval of EPA Water Methods Prepared by Analytical Methods Staff Engineering and Analysis Division (4303), Office of Science and Technology, Office of Water, U.S. Environmental Protection Agency, Washington, DC, December 1996 https://www.epa.gov/sites/production/files/2015-09/documents/guide-method-flexibility-approval-epa-water-methods_draft-1996.pdf
(8) Contact form regarding EPA research https://www.epa.gov/research/forms/contact-us-about-epa-research
(9) Building Flexibility with Accountability into Clean Air Programs https://www.epa.gov/clean-air-act-overview/building-flexibility-accountability-clean-air-programs
(10) Contact form for the Clean Air Act https://www.epa.gov/clean-air-act-overview/forms/contact-us-about-clean-air-act
(11) Website to download free LeachXSLite software http://www.vanderbilt.edu/leaching/leach-xs-lite/

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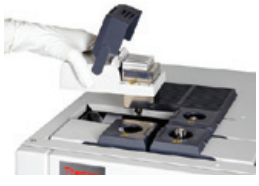

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