Wyndham City Center Hotel 1143 New Hampshire Ave., NW July 25 – 27, 2005

### NEMC 2005 Proceedings

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# **Plenary Session**









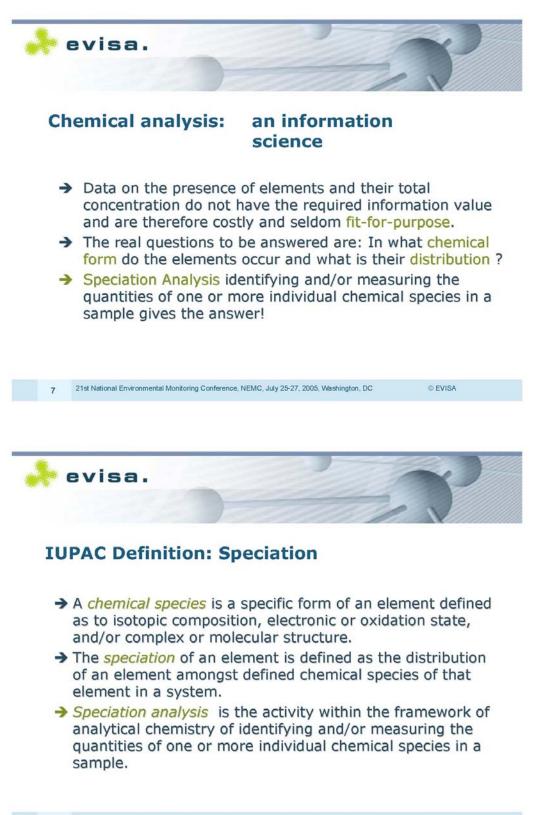
#### Toxicity and speciation

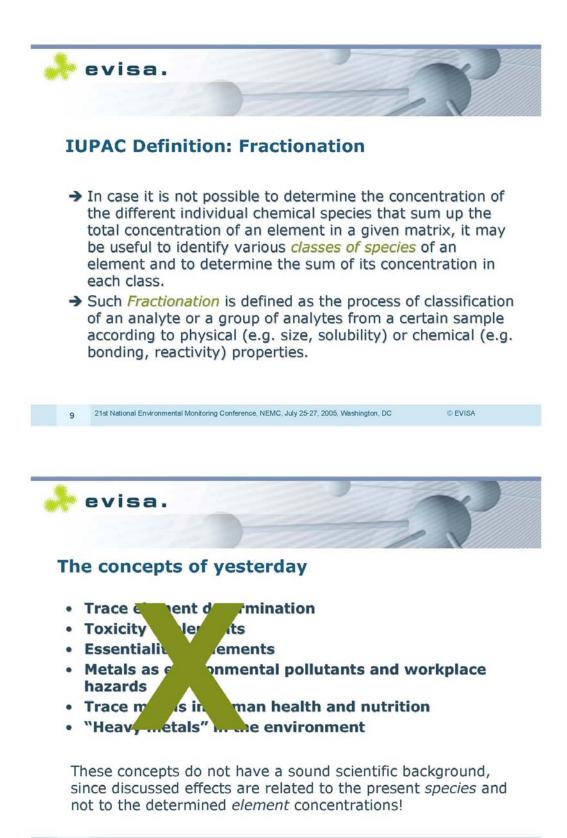
The toxicity of "toxic trace elements" depends on their speciation and concentration not only in a quantitative way but also in a qualitative way. Some examples:

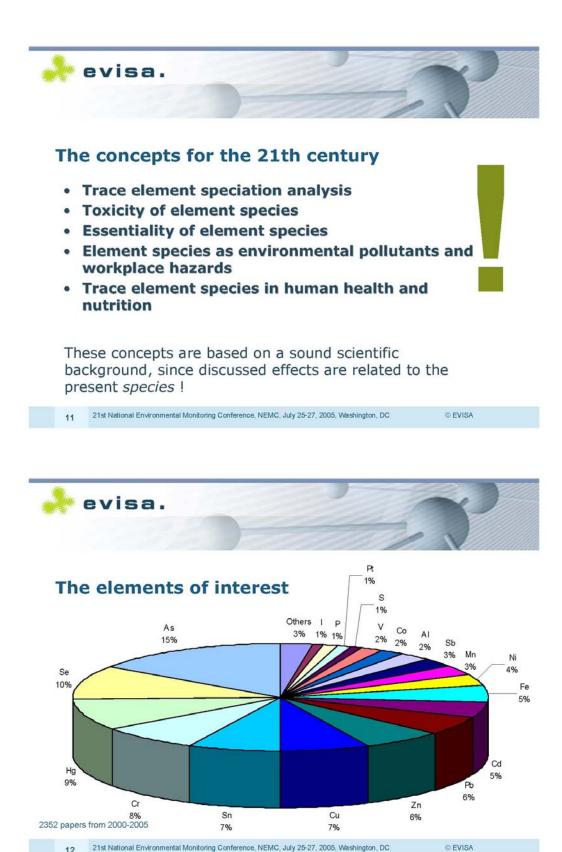
Chromium:	Cr(III) is considered to be essential while Cr(VI) is cancerogen
Arsenic:	Inorganic As(III) compounds are cancerogen while Arsenobetaine is essential non-toxic
Tin:	Inorganic tin compounds are nutrients for animals but tributyltin (TBT) is an endocrine discuptor
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- being present in the particular, colloidal, dissolved or gaseous phase
- being positively, negatively or non-charged
- · being present as a "free" ion, sorbed, complexed or bound









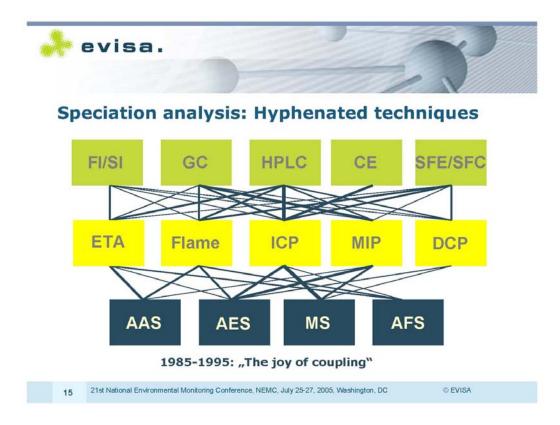
# Speciation analysis: Do we have the necessary tools ?

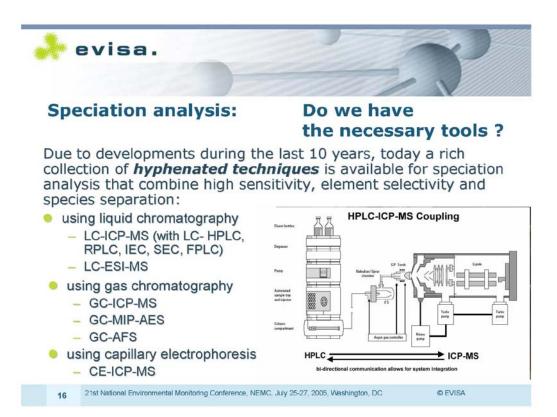
#### **Dissolved species determinations:**

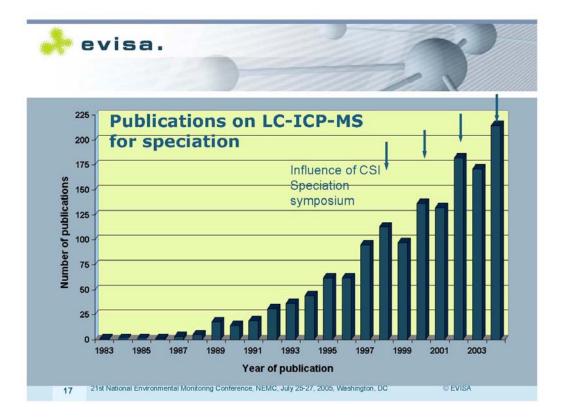
Few techniques are available that directly provide species (molecular) information, such as

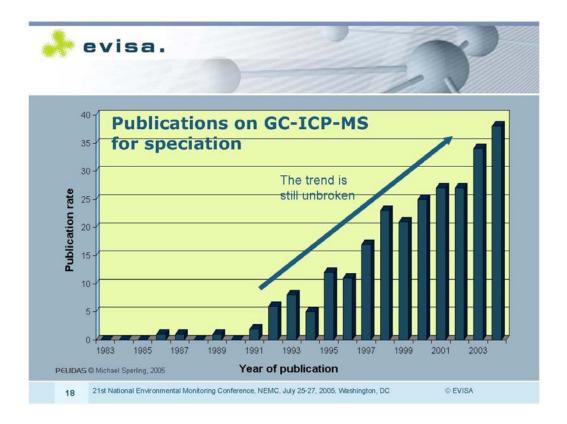
- Electroanalytical techniques
  - Potentiometry with Ion-specific Electrodes (ISE)
  - Voltammetry
- Magnetic resonance spectroscopy
  - Nuclear magnetic resonance spectrometry (NMR)
  - Electron spin resonance spectrometry (ESR)
- Nuclear spectroscopy
  - Mössbauer spectroscopy

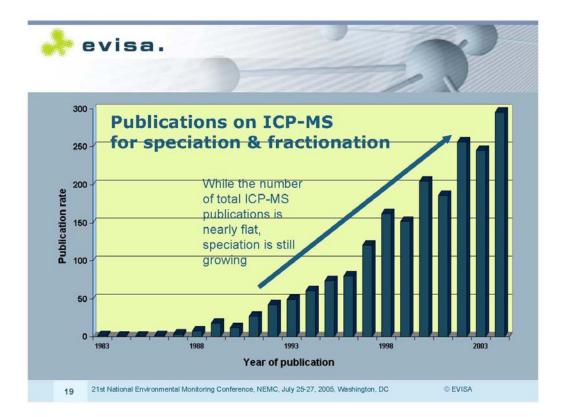
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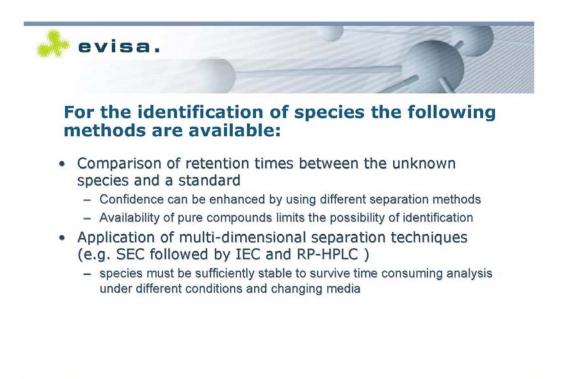


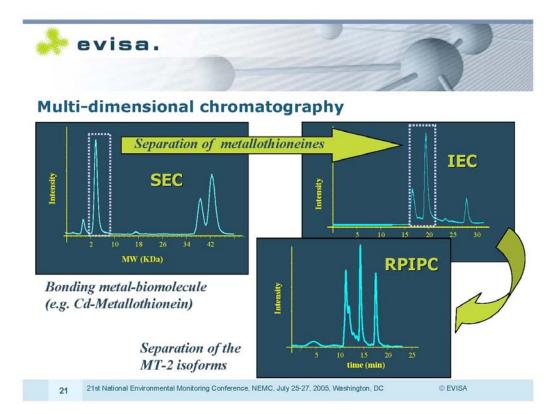


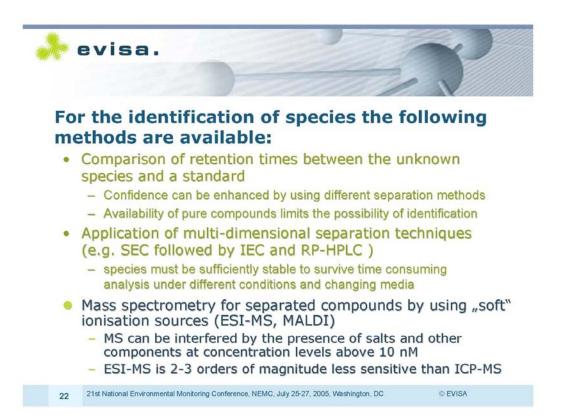


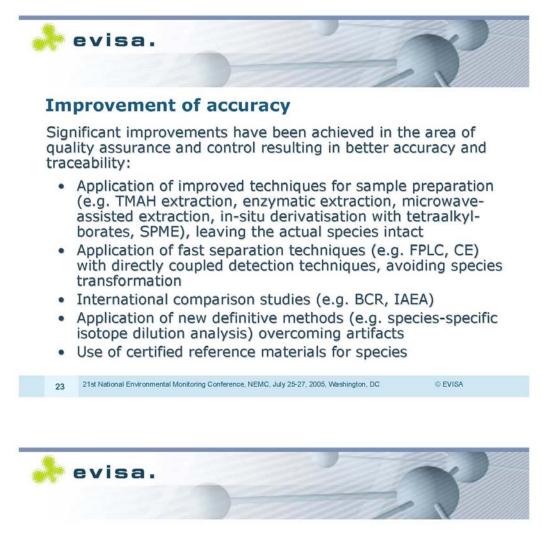












#### Situation: CRM's for the speciation of Cr

Supplier	Code	Certificate
NIST	SRM 2108	Cr(III) in solution
	SRM 2109	Cr(VI) in solution
BCR	CRM 544	Cr(III)/Cr(VI) in lyophilised solution
	CRM 545	Cr(VI) in welding fume (loaded on filter)
	CEMENT 1	Cr(VI) in cement, low concentration
	CEMENT 2	Cr(VI) in cement, high concentration



# Situation: CRM's for the speciation of Sn

Supplier	Code	Certificate
BCR	CRM 424	TBT in habour sediment
	CRM 462	Butyltin-compounds in coastal sediment
	CRM 477	Butyltin-compounds in mussel tissue
	CRM 646	Butyl/Phenyltin- compounds in fresh water sediment
	CRM 710	DBT and TBT in oyster tissue

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# Situation: CRM's for the speciation of Sn

Supplier	Code	Certificate
NRCC	PACS 1	Butyltin-compounds in Marine sediment
	PACS 2	Butyltin-compounds in Marine sediment
NMIJ	7301-a	Butyltin-compounds in Marine Sediment

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Situation: CRM's for the speciation of Hg			
Supplier	Code	Certificate	
NRCC	DORM 1	Total Hg and Methyl-Hg in fisch muscle (dogfish)	
	DORM 2	Total Hg and Methyl-Hg in fisch muscle (dogfish)	
	DOLT 1	Total Hg and Methyl-Hg in fisch liver (dogfish)	
	DOLT 2	Total Hg and Methyl-Hg in fisch liver (dogfish)	
	LUTS 1	Trace elements and Methyl-Hg in lobster tissue	
	TORT 1	Total Hg and Methyl-Hg in lobster tissue	
	TORT 2	Total Hg and Methyl-Hg in lobster tissue	
NIES	NIES 13	Total Hg and Methyl-Hg in human hair	



# Situation: CRM's for the speciation of Hg

Supplier	Code	Certificate
NIST	SRM 1974 a	Total Hg and Methyl-Hg in mussel tissue
	SRM 2974	Total Hg and Methyl-Hg in mussel tissue
	SRM 2976	Total Hg and Methyl-Hg in mussel tissue
	SRM 2977	Methyl-Hg in mussel tissue
	SRM 1566b	Methyl-Hg in oyster tissue
	SRM 1946	Methyl-Hg in fresh water fish

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# Situation: CRM's for the speciation of Hg

Supplier	Code	Certificate
IAEA	<b>IAEA</b> 142	Total Hg and Methyl-Hg in mussel homogenate
	IAEA 350	Total Hg and Methyl-Hg in fish homogenate (tuna)
	<b>IAEA 140</b>	Total Hg and Methyl-Hg in marine plant homogenate
	IAEA 085	Total Hg and Methyl-Hg in human hair, spiked
	IAEA 086	Total Hg in human hair
	IAEA 356	Methyl-Hg in contaminated marine sediment
	IAEA 405	Methyl-Hg in estuarine sediment



# Situation: CRM's for the speciation of Hg

Supplier	Code	Certificate			
BCR CRM 422		Methyl-Hg in fish muscle (cod)			
	CRM 463	Total Hg and Methyl-Hg in fish muscle (tuna)			
	CRM 464	Total Hg and Methyl-Hg in fish muscle (tuna)			
	CRM 580	Total Hg and Methyl-Hg in sediment			
	CRM 710	Methyl-Hg in oyster tissue			
Immuno	Seronorm Whole blood	Trace elements and Methyl-Hg in whole blood			



# Situation: CRM's for the speciation of As

Supplier	Code	Certificate		
BCR	CRM 626	Arsenobetaine in solution		
BCR	CRM 627	Organoarsenic-compounds (DMA, AsB) in tuna fish tissue		
BCR	CRM 710	Arsenobetaine in oyster tissue		
NIES	NIES 14	Innorganic arsenic compounds in brown algae		
NIES	S NIES 15 Arsenobetaine in clams			
NIES NIES 18		Arsenic species in urine		

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## Situation: CRM's for the speciation of Pb & Se

Supplier	Code	Certificate
NRCC	CASS 3	Total Se and Se(IV) in coastal sea-water
BCR	CRM 605	Trimethyllead in urban dust
BCR	CRM 605	Trimethyllead in urban dust

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Matrix	Hg	As	Sn	Cr	Se	Pb	others
Aqueous solution							
Natural water							
Sea water							
Sediment							
Soil		I					
Air particulate							
Cement							
Plant							
Fish							
Mussel, Oyster							
Urine							
Serum							
Whole blood							
Hair							



#### Speciation: Why is it not yet done routinely ?



**Historical reasons:** Inorganic analysis and especially trace metal analysis has been evolved historically through the development of *atomic* spectrometry.



Unfortunately, sources for atomic spectrometry (atomizers) are meant to produce *atoms* destroying most of the information of originally present chemical species.



Most of the techniques used (AAS, AES, MS) work best with liquid samples, calling for some *sample preparation* (sample preservation, digestion etc.) destroying molecular information even in front.



#### Speciation: Why is it not yet done routinely ?



#### Methodological difficulties:

In order to do speciation analysis, the original distribution of chemical species in the probed compartment must be either preserved within the sample or the speciation analysis must be performed *in situ* (on-site).



Both strategies do require more sophisticated instrumentation, higher knowledge about the chemistry and better control of the methodology than required by the total element analysis.

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#### Speciation: Why is it not yet done routinely ?



Lack of species-related legislation:

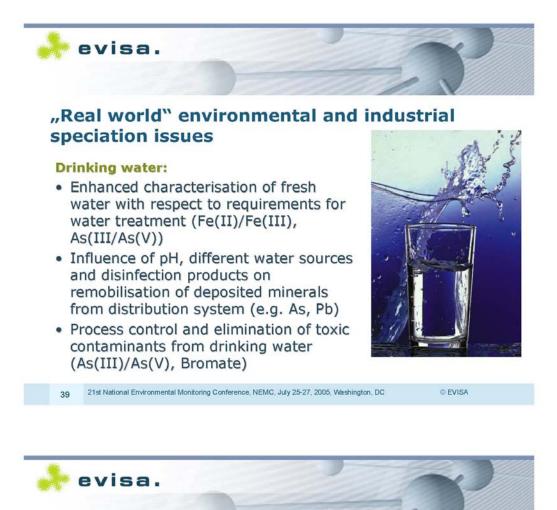
Most existing rules and legislation forces analytical laboratories to perform total element determinations.



While the European Water Framework Directive (2000/60/EC) specifies that the species of Cd, Pb, Hg, Ni as well as tributyltin have to be controlled in water, there are very few national rules and standards implemented, that regulate species related measurements.



- RoHS
- WEEE



# "Real world" environmental and industrial speciation issues

Waste management:

- Risk assessment (mobility of pollutants, degradation and transformation, potential toxicity),
- Waste management (Cr in leather tannery waste, Se in waste water, mobility of toxic metals from solid wastes such as fly ashes,
- Optimization of remediation strategies (As, Cd, Cr, Hg and Pb in waste disposal sites, abandoned industrial production places etc.)



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#### **EVISA's Members**

Partnership has been conceived as to ensure full complementary coverage of the different aspects of speciation with respect to:

- Experience (organometallic species, valency species, metal-biomacromolecules...)
- Application area (industry, food, health, environment, research...)
- Location (35 partners from 10 countries)

EVISA is meant to be an open structure, ready to accept new partners in order to complete its area of competence



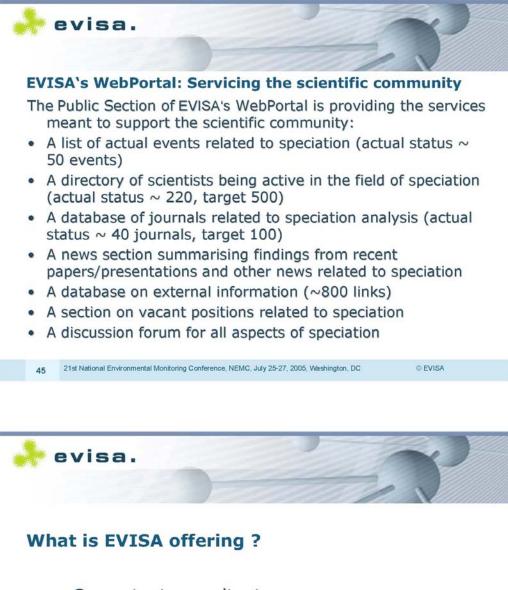


#### evisa's web portal: The Virtual Institute

The web portal of EVISA plays a main role for structuring the Institute itself but also for providing the tool for fulfilling its specific objectives at the operational level.

For this purpose the web site has been organized in the following way:

<b>Entrance Hall:</b>	Public Site (www.speciation.net)			
Front-desk:	Login Area			
First floor:	Customer area			
Second floor:	Partners area (EXTRANET)			



- Competent consultant
- Professional analytical services
- Quality assurance-related activities
- Fit-for-purpose analytical developments
- Workshops and training courses
- Speciation related information





#### EVISA is supporting ...

Politicians and rule makers...

- to define the state-of-the-art of speciation analysis
- to access species related information in an efficient way



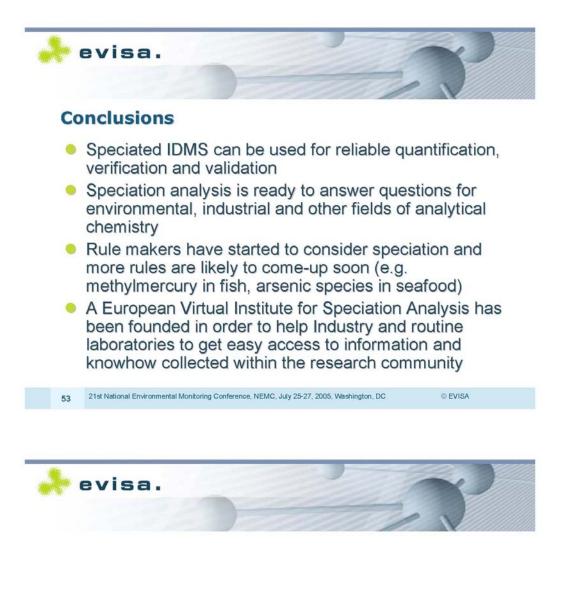
#### EVISA is supporting ...

Scientists...

- by giving easy access to valid information
- by enhancing their opportunities for interdisciplinary cooperation
- by improving their mobility and education
- by enhancing their visibility and recognition

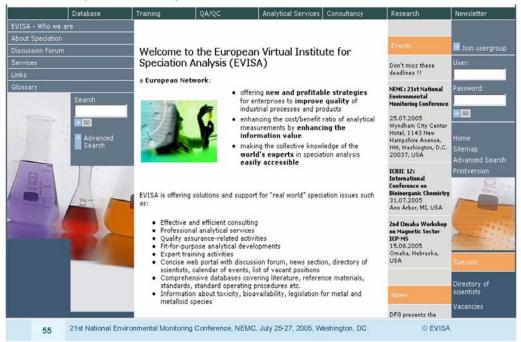






# Many thanks for your attention !

# For more information, please visit our web site at: <u>http://www.speciation.net</u>

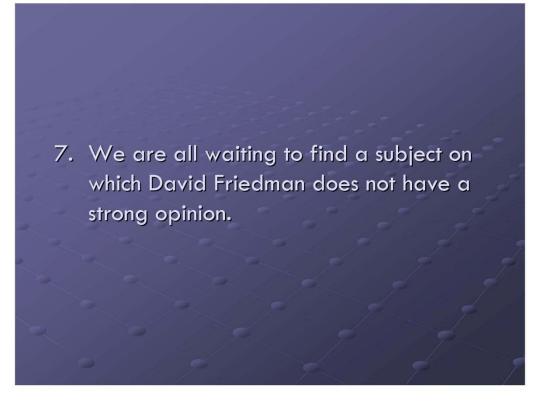


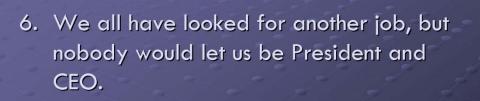
## TOP TEN REASONS

We Have All Stayed in the Environmental Laboratory Business

10.We are all waiting to see if a Republican Administration/Congress will ever prove to us how **business friendly** they are by putting more money into the environmental business. 9. We are all waiting to see if a Democratic Administration/Congress will ever actually spend money on the environment to prove how **environmentally friendly** they are.

8. We are all waiting to see how long it takes for the price of a volatiles sample to go so low that the lab will have to pay the client for analyzing their sample, and report the results **before** the sample is collected?











2. Our last career was in the oil business and it didn't have a future.



## The Number ONE Reason

We Have All Stayed in the Environmental Laboratory Business:



# INNOVATIONS AND IMPROVEMENTS IN ENVIRONMENTAL QUALITY IN NJ: PERFECT TOGETHER

Stuart J. Nagourney NJ Department of Env. Protection Office of Quality Assurance (609)-292-4945 stu.nagourney@dep.state.nj.us

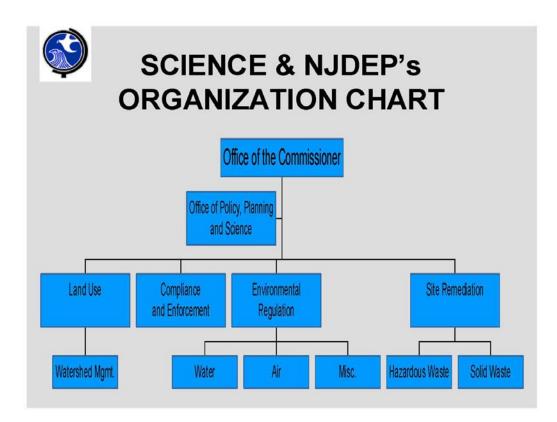


## ANAL. CHEM. & NJDEP MISSION STATEMENT

• Defining and publishing reasonable, clear and predictable scientifically based standards

• Achieving the Department's goals in a manner that encourages compliance and innovation

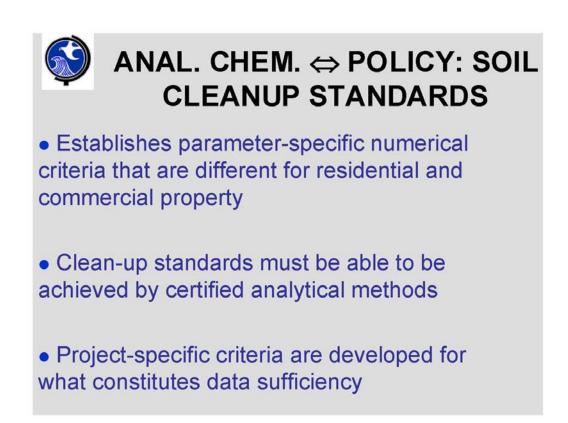
 Assuring that the best technology is planned and applied to achieve long-term goals



## SCIENCE & NJDEP: POLICIES & PRACTICES

 Specific procedures change with every Commissioner, but certain practices remain the same

- Must consider views of diverse interest groups
- Many managers have technical expertise
- Staff always has input to policy decisions
- Consensus sought across program lines





## SCIENCE $\Leftrightarrow$ POLICY: GHG's

• NJ was the 1st state to develop a GHG Action Plan with a quantifiable goal to reduce emissions

• The plan was carefully crafted to achieve reductions in industrial, transportation, housing and other market sectors

Goal: 3.5% reduction in 2000 baseline by 2005

How was this numerical goal developed??



## NEW JERSEY QUANTITATION LIMITS (NJQLs)

• <u>Definition</u>: Multi-lab. MDL by method & matrix (from median of data population) X 5 using data from NJ certified facilities

• The NJQL should be value that can be achieved by most labs. under normal conditions

• How to derive NQLS? What to use them for?



## NJQLs: WHERE ARE WE GOING?

• NJ has the most comprehensive lab. cert. program: >850 labs., > 12,000 parameters

• Developing a database & supporting regulations to manage NJQL data & implement uses

### Potential Uses

- Set permit limits
- Filter labs. eligible to do NJ analytical work

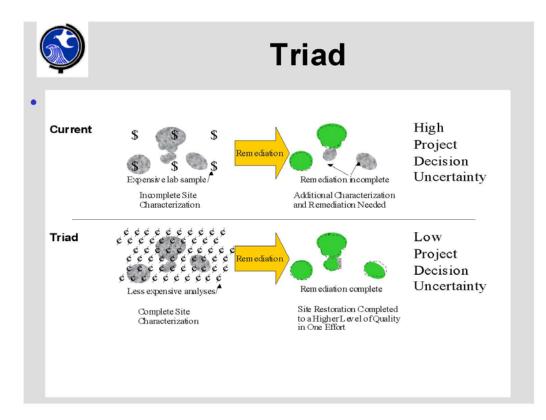


## Triad

• NJs Tech. Regs. (N.J.A.C. 7:26) have always allowed for the use of real-time analytical data for <u>at-risk</u> remedial investigations

### Triad

- Systematic Project Planning
- Flexible Work Planning
- Real Time Measurement Systems
- Triad provides less decision uncertainty







## Cr(VI)

• NJ has the nation's largest inventory of sites contaminated > 50 yrs. ago by Cr(VI)

• Clean-up strategies dependent in-part upon use of most effective available analytical technologies

• EPA withdrew non-aqueous Cr(VI) sample preparation method in early 90's: a historic event!

• NJDEP just completed a review of Cr(VI) issues



## Cr(VI) WORKGROUP CONCLUSIONS

 Only USEPA and NJDEP certified methods will be used for future remedial activities

• Options exist for use of determinative methods, but final site decisions must pass QA or use most definitive analytical technique (Method 6800)

• Development of speciated reference materials will provide additional objective insight into performance of labs. & analytical methods



# CONCLUSIONS

• Sound science (analytical chemistry) continues to drive the making of NJDEP policy

• As the science changes, policy changes as well

- Collaboration with peer organizations invaluable
  - USEPA
  - NIST
  - ITRC

## **Session 1**

## **Method Detection Limits**

#### Method Detection Limits: A Data User's Perspective

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#### ABSTRACT

Over the past several years, there has been considerable discussion in the environmental community regarding the merits of method detection limit (MDL) studies. In March 2003, US EPA withdrew its proposed rule that revised the detection and quantitation procedures for analytical methods under the Clean Water Act (CWA). This action was prompted by divergent comments about the proposed revisions and potential impact on the regulated community. In January 2005, the Agency conducted a public meeting and announced the establishment of the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in CWA Programs (FACDQ).

A recent e-mail inquiry to the US EPA Office of Solid Waste (OSW) Methods Information Communication Exchange (MICE) Service concerning MDL requirements received the following response:

"Actually, the EPA OSW is now in the process of removing requirements for MDL studies in both the individual methods and chapters. Hopefully, the Fourth Edition of the manual, which should be published sometime early next year, will include these revisions. In addition, the SW-846 Methods Team is discouraging the use and application (of) the MDL determination, regardless of the sample matrix type, as defined in 40 CFR Pt 136 Appendix B, for the simple reason that it is not a true indication of the method sensitivity. The MDL calculation has been used repeatedly for a number of EPA programs and it demonstrates the potential data variability for a given sample matrix at one point in time, however, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated."

Environmental Standards believes that the US EPA is finally openly addressing the fact that the procedure to determine MDLs as identified in 40 CFR Part 136 Appendix B is flawed. However, even with the US EPA reviewing the MDL procedures and SW-846 discouraging the use of MDLs, there are many state regulators and other offices within the US EPA that are mandating that data be reported to the MDL for compliance purposes. Environmental Standards will present issues that data users should be aware of when developing MDLs and using results that are reported to the laboratory MDLs.

#### **Detection and Quantitation Limits – Where Do We Go From Here?**

#### **Richard Burrows**, Ph.D.

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#### ABSTRACT

This paper will present a list of "consensus principles" that have received broad agreement as the desired outcomes of detection and quantitation limit procedures. We will discuss a variety of current proposals and how well they meet the goals set by the consensus principles.

- 1. The definition of quantitation must include both precision and bias
- 2. Detection limit procedures must take into account the variability and bias of method blank results
- 3. International definitions of  $L_{\text{Q}}$  ,  $L_{\text{D}}$  and  $L_{\text{C}}$  must be adopted
- 4. False positives and false negatives must be addressed by detection limit concepts
- 5. Precision, bias and qualitative identification must be addressed by the definition and concepts of quantitation
- 6. Detection limit procedures must include procedures for ongoing demonstration of sensitivity

#### **Detection Limits – Federal Advisory Committee**

#### **Richard Reding, Chief**

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#### ABSTRACT

We are working to establish a formal committee of about twenty individuals to provide advice to the U.S. Environmental Protection Agency on ways to improve detection and quantitation approaches in EPA's Clean Water Act programs. Our expectation is that the committee will provide advice on a common set of terms and concepts; one or more specific approaches and/or procedures for detection and quantitation; and recommendations for the interpretation and use of the numbers that result from measurements of pollutants in water. Committee members will be qualified, senior-level professionals with an emphasis on policy experience from diverse sectors, including state government; environmental professionals; regulated industry; environmental laboratories; publicly owned treatment works; and the environmental community. The establishment and makeup of this committee reflects EPA's emphasis on the need for open and inclusive approaches where stakeholders work together with EPA to develop solutions.

The committee will consider the technical and policy issues related to the calculation and use of detection and quantitation limits in Clean Water Act programs. Policy issues include consideration of how much uncertainty is acceptable to make a presence or absence decision, or a decision that a discharge limit has been exceeded. Technical issues may include topics, such as treatment of blanks and censored data, the number and types of samples, matrices, and laboratories required to develop a detection limit, and procedures for a laboratory to routinely demonstrate the capability to meet established limits. Neutral technical experts will be available to provide technical assistance to the committee. These experts would not be members of the committee nor participate in the deliberations.

We are planning to charter and operate this committee under the Federal Advisory Committee Act. We believe that this consultative process will be relatively short, e.g., five or six meetings over one year, and hope to convene this committee in June 2005. Updated information will be available at <a href="http://www.epa.gov/waterscience/methods/det">www.epa.gov/waterscience/methods/det</a>.

#### New Jersey Quantitation Limits: Putting MDLs to Practical Use

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#### ABSTRACT

The New Jersey Department of Environmental Protection (NJDEP), Office of Quality Assurance (OQA) is proposing to establish a program to develop New Jersey Quantitation Limits (NJQLs) for the State. The laboratory certification program administered by the OQA offers certification for a wide variety of analytical methods that measure chemicals in the following sample matrices: drinking water, wastewater, ground and surface waters, solid hazardous wastes and air.

In order for the NJDEP to develop and enforce environmental regulations, measurement of chemical contaminants in environmental samples must be of defined and defensible quality. This includes an assessment of the laboratories' capability to measure chemical contaminants at levels near the detection capability of the analytical method. For environmental compliance monitoring in the United States under United States Environmental Protection Agency (USEPA) regulations, the method detection limit (MDL) is defined at 40 CFR Part 136 Appendix B (July 1, 1993) as the minimum concentration of a substance that can be measured and reported with 99<sup>th</sup>-percent confidence that the analyte concentration is greater than zero. The OQA currently requires all New Jersey certified laboratories supply to the Department method detection limit (MDL) data they are currently required to generate MDLs at least annually for all analytes and methods for which they hold certification.

Since the MDL is a statistically-derived number, one cannot expect that any laboratory can quantify measurements for an environmental sample at that level. Other reasons that the MDL is <u>not</u> suitable as a regulatory level include the fact that the MDL varies from laboratory to laboratory, precision of measurements at the MDL are generally poor; and although the MDL provides adequate protection against false-positive results, protection against false-negatives is inadequate because samples containing contaminants at a concentration near the MDL will not always be measurable. For these and other reasons, quantitation and regulatory decision-making are not feasible at the MDL. This requires that a higher limit must be established to support and validate Department regulatory actions such as the writing of permits and the development of clean-up standards for hazardous waste sites. This higher limit has been designated by the Department as the NJQL, which is defined as the lowest concentration of a particular analyte that can be reliably determined, under routine operating conditions, within specified limits of precision and accuracy. The NJQL is an interlaboratory measure, taking into account performance variability within individual laboratories as well as within the laboratory community as a whole. The NJQL is also analyte, method and matrix-specific; for example,

cadmium and lead (the analytes) will have very different MDLs and NJQLs, and for cadmium in drinking water alone, the MDL and the NJQL may differ depending upon whether flame, graphite furnace atomic absorption spectrophotometry, inductively coupled plasma emission spectroscopy or inductively coupled plasma mass spectrometry is/are used for detection.

For any given analyte, method and matrix, the MDL is expected to vary from laboratory to laboratory because of differences in instrumentation, expertise of personnel, quality of chemical reagents and other factors. The Department plans to use the median of all results for each analyte, method and matrix, and derive a NJQL for each by taking that median of each data population and multiplying the median of that data population by a factor of 5. Since the NJQL is defined as being five times higher than the median MDL, the NJQL should be achievable by most certified laboratories in their daily operations.

Since the NJDEP certifies more than 800 laboratories for more than 10,000 test methods, the management of all of this MDL data in order to generate NJQLs is a daunting task. The NJDEP has therefore contracted with enfoTech to design a database system for this purpose. The management of MDL information from certified laboratories to the NJDEP will be accomplished by requiring each laboratory to complete a Department-supplied electronic deliverable listing all chemical test methods for which the laboratory holds certification. Additional ancillary information such as the low point on the laboratory's calibration curve will also be requested. The database will then sort this MDL information by analyte, method and matrix and allow calculations of NJQLs.

This paper will describe the database development project and the intended use of NJQLs by the NJDEP.

#### A Statistical Determination of Minimum Reporting Levels

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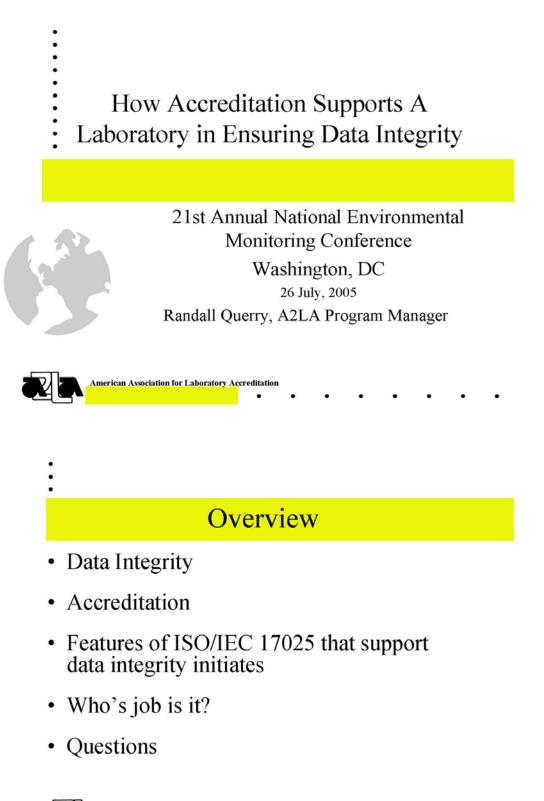
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- <sup>2</sup>U.S. Environmental Protection Agency, Office of Ground Water and Drinking Water, 26 West Martin Luther King Dr., Cincinnati, OH 45268; Email: <u>munch.dave@epa.gov</u>; Phone: (513) 569-7843
- <sup>3</sup>The Cadmus Group, Inc., 57 Water Street, Watertown, MA 02472

#### ABSTRACT

A new statistical procedure is being evaluated for minimum quantitation levels and for verifying minimum reporting levels (MRLs) by EPA's Office of Ground Water and Drinking Water. The lowest concentration MRL (LCMRL) is the lowest true concentration for which future recovery is predicted to fall, with high confidence (99%), between 50 and 150% recovery. The procedure takes into account precision and accuracy, simultaneously applied. Four data sets of 7 samples each are processed through the entire method procedure and the data is plotted as sample concentration (y-axis) versus true concentration (x-axis). An ordinary least squares regression line is calculated and prediction interval lines (99 % confidence) are drawn. At the points where the prediction intervals intersect with 50 and 150% lines of recovery, vertical lines are drawn to the x-axis, and the higher of the two values is the LCMRL. In the case of non-constant variance, a variance weighted regression is used. The LCMRL procedure is flexible because the data quality objectives (i.e., 50 to 150%) and the prediction interval confidence (99%) can be varied to suit program needs. The LCMRL determination is performed during method development only. Once an MRL is established, a simpler procedure is used for MRL lab verification. A validation of laboratory performance at or below an MRL is made using a single set of 7 samples run through the entire method procedure. If the calculated prediction interval is contained within data quality recovery limits (50 to 150%), the lab performance for that analyte is validated.

## **Session 2**

## **Laboratory Accreditation**





• Data integrity refers to the VALIDITY of data.



# Data Integrity-Compromised

- Human error when data is entered
- Errors that occur when data is transmitted
- Software bugs or viruses
- Hardware malfunctions
- Environmental conditions/Natural disasters



### Accreditation

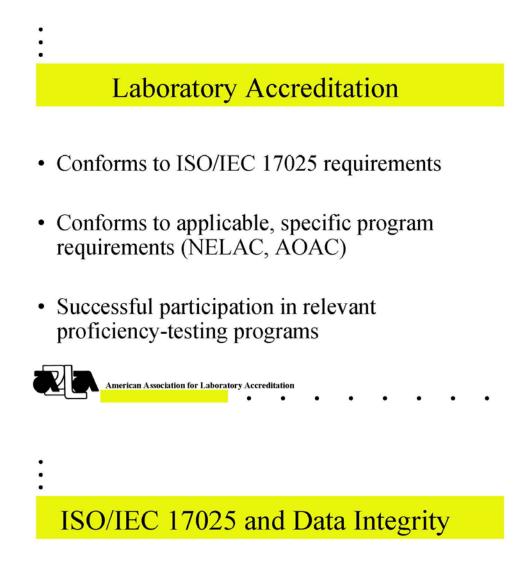
 third-party attestation related to a conformity assessment body conveying formal demonstration of its competence to carry out specific conformity assessment tasks



## **Accreditation Bodies**

- Operates to ISO/IEC 17011
- Conducts on-site assessments evaluating management systems *and* testing activities
- Employs the use of technical experts as assessors
- Issues Scope of Accreditation identifying applicants specific competencies





- ISO/IEC 17025 specifically promotes data integrity in three primary sections:
  - 4.1.5 Organization
  - 5.4.7 Control of data
  - 4.12 Control of records



### Organization requirements

- 4.1.5 states that the laboratory SHALL:
  - a) have managerial and technical personnel with the authority and resources...
  - b) have arrangements to ensure that its management and personnel are free from any undue INTERNAL and EXTERNAL commercial, financial and other pressures/influences



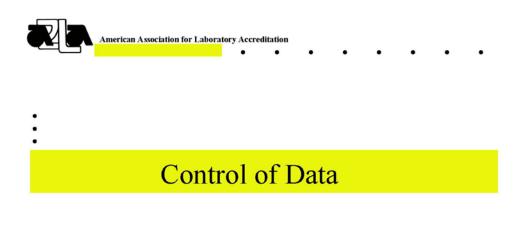
### Organization requirements

- 4.1.5 states that the laboratory SHALL:
  - c) have policies and procedures to ensure the protection of client's confidential data, including procedures for protecting the electronic storage and transmission of results
  - d) have policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgement or operational integrity



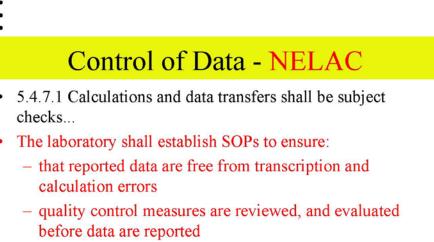
### Organization requirements

- 4.1.5 states that the laboratory SHALL:
  - g) provide adequate supervision of testing staff, including trainees, by persons familiar with the methods and procedures



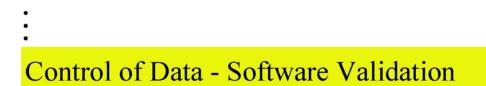
• 5.4.7.1 Calculations and data transfers shall be subject to appropriate checks in a systematic matter





addressing manual calculations including manual integrations





- 5.4.7.2 When computers or automated equipment are used for acquisition, processing, recording, reporting...the laboratory shall ensure that:
- a) computer software developed by the user is documented in sufficient detail and is suitably validated as being adequate for use.



### Control of Data - Protecting Data

 5.4.7.2 b) procedures are established and implemented for protecting the data, such procedures shall include, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing



## Control of Data - Maintenance

 5.4.7.2 c) computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of test and calibration data





- ISO/IEC 17025, Section 4.12.2.3 requires that all records shall be held secure and in confidence
- ISO/IEC 17025, Section 4.12.2.4 stipulates that the laboratory shall have procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records



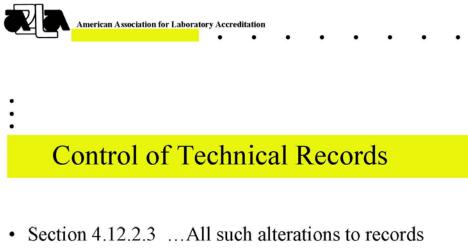
## **Control of Technical Records**

• ISO/IEC 17025 Section 4.12.2.2 states that observations, data and calculations shall be recorded at the time they are made and shall be identifiable to the specific task



### **Control of Technical Records**

• Section 4.12.2.3 requires that when mistakes occur in records, each mistake shall be crossed out, not erased, made illegible or deleted, and the correct value entered along side...



Section 4.12.2.3 ...All such alterations to records shall be signed or initialed by the person making the correction. In the case of records stored electronically, equivalent measures shall be taken to avoid loss or change of original data



### NELAC on Data Integrity

- NELAC Chapter 5 Section 5.5.2.7 requires the following to promote data integrity:
  - data integrity training for new hires and annually
  - topics shall be documented and provided to trainees
  - topics shall include: organization's mission, honesty, full disclosure in analytical reporting...



### NELAC on Data Integrity

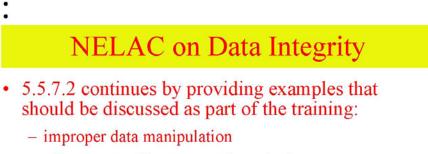
- Topic continued:
  - how and when to report data integrity issues
  - recordkeeping
- Training shall include a discussion of data integrity procedures, in-depth data monitoring, data integrity procedure documentation



### NELAC on Data Integrity

- 5.5.7.2 also requires that training include a discussion of the consequences of infractions of the procedures
  - detailed investigation that could lead to
    - immediate termination
    - debarment
    - · civil/criminal prosecution



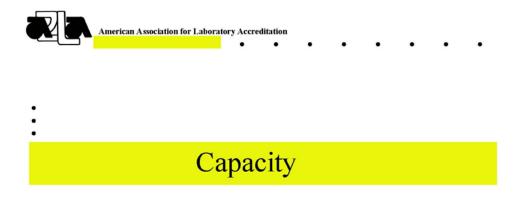


- adjustments of instrument time clocks
- inappropriate changes in concentration of standards



## **ISO/IEC 17025 Principles**

• Several important principles are imbedded in the requirements of the ISO standard that help assure data integrity:

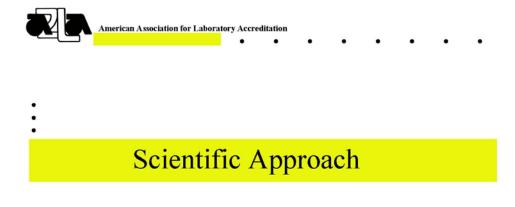


 A laboratory must have the resources, (people with the required skills and knowledge); an environment with the required facilities; equipment and instruments; procedures to ensure consistency of test processes, and quality control for the key steps in the testing processes, in order to carry out the test and produce reliable results.



### Responsibility

• A laboratory shall have personnel in its organization who have the authority to execute specific functions and can demonstrate accountability for their results.

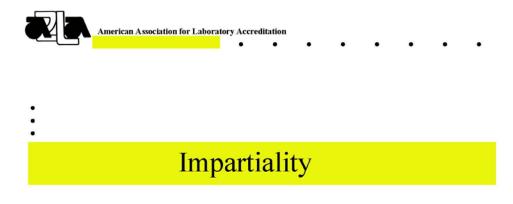


• A laboratory shall carry out its work based upon accepted scientific principles, preferably following consensus-based methods or standards.



# Objectivity

• The results generated should be based upon measurable quantities-and if results are subjective, they must be produced by people deemed qualified to make subjective judgements.

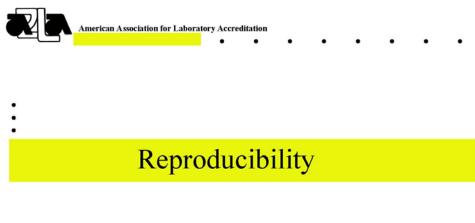


• The pursuit of reliable results through the use of accepted scientific principles is the primary and overriding influence on the persons carrying out the testing. All other influences are secondary and not permitted to take precedence.



# **Measurement Traceability**

• The results produced are based upon on a recognized system of measurement that derives from accepted known quantities (SI system if units of measurement) or other well-characterized references.



• The test method used to produce the results will produce results within an acceptable spread or range during future testing and within the constraints of using the same procedures, equipment and persons used for a prior analysis.



## Transparency

• The processes within a laboratory producing objective results must be open to external as well as internal scrutiny, so that factors which may adversely affect the laboratory's pursuit of objective results based upon scientific principles can be easily identified and resolved.



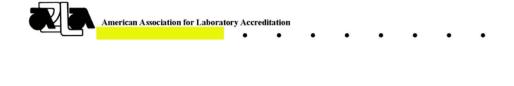
# Who's job is it?

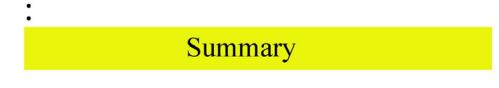
- Management
  - management's commitment to the quality system
  - providing adequate personnel, technical oversight
  - making available proper equipment and instruments
  - training programs



# Who's job is it?

- Analyst
  - Competent in quality and technical procedures





- ISO/IEC 17025 provides the framework from which to build a system that promotes the integrity of data.
- Accreditation is the attestation that a laboratory has been found to be competent in performing specific tests.
- Management and staff both share a role in data integrity.



# **Contact Information**

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# A Harmonized National Accreditation Standard: The Next Step for INELA Field Activities

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## ABSTRACT

The original charter of the National Environmental Laboratory Accreditation Conference (NELAC), when established in the early 1990's, was to "foster the generation of environmental laboratory data of known and documented quality through the development of national performance standards for environmental laboratories". However, it has been generally recognized within the environmental community, over the years, that the quality of environmental laboratory data can only be assured if minimum performance standards exist for field sampling and measurement activities – the "front-end" of the environmental data generation process. To assure the production of environmental data that are scientifically valid and can be used with a high degree of confidence by the end-user, control of environmental laboratory analytical processes **and** field sampling and measurement processes are of equal and significant importance. Accordingly, in July 1998, the Constitution of NELAC was amended to reflect the growing interest of many stakeholders to expand its scope to include both field sampling and measurement activities. Subsequent to this Constitutional amendment, the Field Activities Committee was officially established in 1999 as a NELAC standing committee responsible for the development of performance standards applicable to those organizations performing field sampling and measurement activities.

In July 2002, Chapter 7, *Field Activities Standard,* was added to the NELAC Standard to address minimum quality and technical requirements for field sampling and measurement activities. The initial draft of this chapter excerpted selected verbiage from Chapter 5, *Quality Systems,* of the NELAC laboratory standard and did not specifically address other accreditation components (e.g., proficiency testing (PT), on-site assessment, and accreditation process) or requirements for sampling specific environmental matrices. In 2003, NELAC divested itself of the environmental standards development process and the Institute for National Environmental Laboratory Accreditation (INELA), a consensus based standards development organization, was formed. Within this organization, the INELA Field Activities Committee (FAC) was established to continue the standards development work for an accreditation program designed specifically for field sampling and measurement organizations (FSMO).

## **Objective and Goals**

The primary objective of the INELA FAC is "to develop and maintain consensus accreditation standards and guidance materials for organizations engaged in environmentally related field sampling and measurement activities, consistent with regulatory and industry-specific requirements". Its long-range focus is to replace the 2002 NELAC *Field Activities Standard* (Chapter 7) with an INELA stand-alone, FSMO-specific accreditation standard(s) that meets the

following goals:

- Encompasses broad scope and wide ranging applicability;
- Based on internationally recognized standards for competency (ISO/IEC 17025) and conformity assessment (ISO/IEC 17011);
- NOT prescriptive in nature, allowing for the development of FSMO-specific policies and procedures; and
- Effectively supported by sound guidance.

## **Broad Scope and Applicability**

If the INELA FAC is to meet its objective of establishing performance standards for those collecting samples and conducting on-site measurements for improved environmental data quality, then the standard must be wide-ranging in scope and applicability to support existing and future state/federal environmental regulations governing field sampling and measurement activities. To this end, a primary goal of the INELA Field Activities Committee is to develop an accreditation standard (or series of standards) that will apply to organizations performing field activities for a wide variety of sampling and measurement media such as air, biological, water, soil, waste, and radiological. Due to the nuances, specific to each media, a "one size fits all" approach to standards development is not appropriate. Accordingly, the FAC has engaged field sampling and measurement "media experts" to collaborate on the development of customized, media-specific FSMO accreditation standards. The development of custom field standards for water and air are the current focus of the committee.

## **ISO Foundation**

It is the consensus viewpoint of the Field Activities Committee that the common denominator, or foundation, for the custom, media-specific INELA FSMO accreditation standard(s) must be ISO/IEC 17025, <u>General Requirements for the Competence of Testing and Calibration Laboratories</u> and ISO/IEC 17011 (soon to replace ISO/IEC Guide 58), <u>Conformity Assessment – General Requirements for Accreditation Bodies Accrediting Conformity Assessment Bodies</u>. Using this approach to standards development, the role of the INELA FAC will be to utilize its "media experts" to determine **how** to best apply these generic International Standards for a particular area of accreditation (e.g., field activities – water). The INELA FAC "application" of these International Standards, for each sampling and measurement media, will include, but will not be limited to, provisions for additional requirements, exclusion of specified ISO requirements. Using ISO as the foundation for custom-built FSMO accreditation standards facilitates harmonization of individual field standards specific to each sampling and measurement media.

## **Non-Prescriptive Standards Development**

Although sampling has, historically, been recognized as a major contributor to the overall measurement error, many organizations performing field sampling and measurement activities today are not currently subject to rigorous and prescriptive quality system requirements, accreditation, or routine oversight. Accordingly, the committee consensus was to take a practical and realistic first step towards improved environmental data quality by establishing an

accreditation standard, based on internationally recognized standards, which are minimally prescriptive to provide a high degree of flexibility for the FSMO when implementing the standard requirements. Simply stated, applying this "less is better" approach, the FSMO will be able to craft policies and procedures, which meet the intent of the INELA standard, but are practical, functional and, most importantly, implement-able. The INELA FAC believes that if the resulting field accreditation standards cannot be effectively implemented by all parties affected, large and small, public and private, due to overly prescriptive requirements, then we, as a committee, have not successfully completed our mission for improving data quality for better decisions.

## Sound Guidance

To support the "less is better" approach to standards development and to facilitate successful implementation by all FSMO impacted by the standard, the development of appropriate implementation guidance tools is a key component for realizing an improved outcome – sound and defensible data quality for better decisions. This is the long-term focus of the INELA Field Activities Committee - to "show the way" by providing the necessary guidance and support for standards implementation. Several of the many benefits associated with this INELA service to the environmental community include:

- Acceleration of the FSMO "learning curve" associated with "something new", keeping in mind that many FSMO have not been subject to quality system/accreditation program requirements, historically;
- Improved "buy-in" by minimizing the costs associated with implementation of a new and comprehensive accreditation standard; and
- Consistency of standards interpretation and implementation.

## Accomplishments

These goals for standards development, as discussed in the previous sections, have evolved over a period of two (2) years as a result of the diligent work and "outside the box" thinking of the INELA FAC. The accomplishments, which follow in this section, have contributed greatly to the refocusing of the laboratory community (regulators and those regulated) on the importance of field sampling and measurement and its role, as the "front-end" portion of the environmental data generation process.

To facilitate the development of media-specific field standards, the committee has been very active in outreach activities to engage more stakeholders – the "media experts" - in the standards development process. The INELA FAC has grown from less then ten (10) members in 2003 to more than thirty (30) participating members today. The committee has also worked to achieve balance of membership, necessary for a consensus standards development organization, with representation from government and municipal agencies; engineering and environmental consulting firms, analytical laboratories and industry. Participation in national/regional conferences and collaboration with other organizations representing specific stakeholder groups will continue to be a focus for the INELA FAC. The committee's success in developing sound field accreditation standards depends on the continuation of these outreach activities.

Consistent with committee direction to develop "applications" of the ISO/IEC 17025 and 17011 standards, a generic (not specific to any one media) application of the ISO/IEC 17025 standard

has been completed and will be utilized by the "media experts" to guide the development of media-specific field accreditation standards. This generic application of ISO/IEC 17025 was affirmed by the INELA membership in late 2004. Additionally, the groundwork, in the form of a consensus-based conceptual model, for the application of the ISO/IEC 17011 standard was completed and presented at the INELA Accreditation Forum in Charleston, South Carolina last summer. Building on these endeavors, workgroups have been established and are tasked with producing the first Working Draft Standards for a generic application of 17011 and a mediaspecific (water) application of 17025 by the summer of 2005.

A great deal has been accomplished but there is more work to do.

## **Next Steps**

To achieve its on-going objective "to develop and maintain consensus accreditation standards and guidance materials for organizations engaged in environmentally related field sampling and measurement activities, consistent with regulatory and industry specific requirements", the INELA Field Activities Committee must effectively meet certain challenges. They are:

- To know, engage and understand the needs of all stakeholders who will be, ultimately, impacted by the standard(s).
- To know, engage and understand the needs of all potential clients, those who will adopt and implement such a standard(s).
- Finding a consensus viewpoint to the question of *what makes for good quality* to achieve consistent application of the ISO/IEC 17025 and 17011 standards for harmonized individual media-specific field accreditation standards.

With its new approach to standards development, the INELA FAC also has an opportunity to help chart the future path of INELA, as a standards development organization. At the 2004 INELA Summer Forum in Charleston, South Carolina, the INELA Board of Directors expressed their desire for INELA membership to seriously consider a restructuring of the NELAC laboratory standard to better meet the needs of stakeholders, existing and potential clients, and to achieve the desire growth into other areas of accreditation. There are a number of proposals for this restructuring initiative currently being considered by the INELA Board.

One of the proposals being considered has been developed by the INELA FAC, which details an approach to standard restructuring, consistent with the approach being taken for the development of media-specific field accreditation standards. This proposal has been designed to:

- Align with the INELA Strategic Plan.
- Provide a flexible framework for the development of harmonized accreditation standards in new areas such as Homeland Security.
- Positively impact a wide range of stakeholders.
- Appeal to accrediting authorities, regulators, private sector groups interested in adopting and implementing uniform standards of accreditation.
- Assure the production of scientifically valid data that can be used with a high degree of confidence by the end user.

The INELA Field Activities Committee is committed to the development of field accreditation standards using the approach detailed in this paper and strongly believes that this approach can

be effectively used for the development of new INELA standards in other areas of accreditation as well. To meet the current challenges and to adequately address the complexities of the field sampling and measurement "world", the committee must continue to focus its energies on thinking "outside the box", encouraging and listening to new ideas, and creating an environment where these new ideas can flourish. Your participation in the FAC activities is vital for the production of data suitable for its intended use **and** may have an influence on the future path of INELA as a consensus standards development organization. All are encouraged to join INELA and to get involved! More information on the efforts of the INELA FAC may be found on the INELA web site (www.inela.org).

## **Accreditation of Air Emission Testing Bodies**

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## ABSTRACT

This presentation is an update on the progress and current status of Air Emission Testing Body (AETB) accreditation. The presentation will briefly cover key requirements of the ASTM D7036 standard as well as of the draft ASTM accreditation standard currently awaiting balloting at ASTM. The presentation will also cover the current activities of the Source Testing Accreditation Council and look at proposed accreditation process models such as the AIHA model for lead laboratory accreditation.

### LIMS and Regulatory Compliance

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There are an increasing number of regulatory and productivity demands placed on the environmental laboratory, from NELAC, HIPPA, Sarbanes-Oxley, The Patriot Act, ELAP, CFR 21 Part 11 to name a few. This presentation will review the features in an automated Laboratory Information Management System (LIMS) from sample login through to reporting that greatly facilitate compliance.

Sample tracking functionality provides not only the capability to scan in a chain of custody form, but also provides for an internal chain of custody (the physical location of the sample within the laboratory). With the use of electronic notebooks, users can enter chain of custody information into the notebook and electronically upload that information into the LIMS thus avoiding transcription errors. Most LIMS provide CRF 21 part 11 (electronic signatures) compliance. The LIMS provides instant access to the sample status and location information. During sample login, users can also select from a pull-down list (which is limited to tests that can be performed on that matrix), project, site and GIS information can be recorded. QC samples can automatically be assigned as well as custom report requirements. Once samples are signed off, a date and time stamp is applied to the sample order. Users can enter results and an Electronic Data Entry Module can be configured automatically to import data from analytical instrumentation such as an ICP, GCMS or AA.

Any modifications to results include a complete audit trail (audit reports can also be easily printed). With integrated QA/QC, users can view trend analysis and create QC charts. In addition, users are alerted at result entry when results have exceeded pre-defined limits, which can immediately be checked. A major benefit of an automated LIMS is in reporting in both paper and electronic format to regulatory agencies often in a specified format that can be sent electronically. Permit limits can be configured in the system and triggers can be set to alert users if any limits have been exceeded for rapid response.

The Washington Aqueduct is a wholesale water utility that provides potable water to the District of Columbia Water and Sewer Authority (WASA), Arlington County, VA, and the City of Falls Church, VA. The Washington Aqueduct Laboratory serves as the contract laboratory for its wholesale customers. In January of 2004, the Washington Post reported on high lead results found in Washington, D.C. water. As the news spread throughout the region, newspapers, television stations, and radio stations delivered updates to the lead story on a daily basis causing area residents and elected officials to voice their concerns, questioning the safety of the area's drinking water. In an attempt to understand the extent of the problem, an extensive data collection effort began involving DCWASA, Arlington County, and Falls Church. All three increased their monitoring for lead by sampling area homes, schools, and daycare centers. In addition, the large numbers of samples continue to be collected by DCWASA as lead service lines are replaced and lead profiles are studied at different locations throughout the city.

The situation demands a massive collection of data to be processed and reported with the quickest turnaround time possible. The use of a LIMS has been essential to the timely processing of samples and reporting of results to customers and regulatory agencies. The ready availability of quality control data and electronic files has been critical in responding to Freedom of Information Requests. This presentation will highlight how the Washington Aqueduct utilizes its LIMS to effectively respond to increased analytical demands and data scrutiny. It will review the features in an automated Laboratory Information Management System (LIMS) from sample login through to reporting, that greatly facilitate compliance.

#### Introduction

A computerized LIMS is an important tool in the growing analytical laboratory. Most laboratories begin with a paper laboratory notebook and progress to Excel for data management and Word templates for reporting. However, most quickly outgrow these tools and require a more robust, relational, and secure database. A computerized LIMS brings numerous advantages to the analytical laboratory. These advantages include the automatic generation of bar-coded labels, the ability to pre-log in samples with pre-assigned tests, limits, QC, and the ability to rapidly log large batches of samples into the LIMS during major monitoring efforts. The ability to pre-configure tests with associated QC, and set up default methods and automatically generate bar-coded labels greatly expedites sample receipt and reduces errors associated with manual entry.

#### Sample Login and Tracking

Once samples are received into the laboratory, they are signed off and a computerized LIMS can record who accepted the samples and the date and time that they were received into the laboratory. In addition, it can record which analyses were assigned to those samples. Some systems also have features to record the hold times, which is the amount of time the sample, must be analyzed to obtain an accurate result. Since all of the data is in a central database, user can create a number of reports that can be automatically e-mailed to analysts. It can let them know which samples are approaching their hold times and for managers, backlog and production reports can be created that will provide information on how much work is waiting to be done, how much work was completed respectively.

#### Advantages of Automated Sample Tracking:

- 1. Pre-determined, user definable, sample number is automatically generated from a validated system, so that there is no chance of sample numbers being duplicated. Aliquots are also assigned a unique identifier.
- 2. Ability to scan in, link the chain of custody form, and link that to the sample order for easy retrieval by the laboratory users.
- 3. Selection of tests from pre-defined pull down lists that are limited by the matrix. Users are not permitted to add tests, methods or parameters on the fly to ensure that the database remains "clean". Otherwise, users could have lead, Pb, and Lead as tests that would all be recognized as different in the database.
- 4. Pre-defined QC can be configured for each test at login to ensure that certain QC is not forgotten to be run.

#### Sample Scheduling

Most LIMS offer the ability to automatically schedule sample collection in advance and even the ability to set up projects and studies. This ensures that sampling events are not missed and sample labels and worklists can be pre-printed and pre-logged into the LIMS. This allows the laboratory to prepare for the incoming workload and to prepare the sample bottles in advance.

#### Data Entry and Electronic Data Transfer

Users can manually enter data and if they choose to turn peer review on, the person that has entered the result cannot approve the result, a supervisor or another analyst must approve the results. The database administrator can configure the LIMS to assign LIMS access (by module and function) and LIMS permissions to specific users based on their laboratory functions. Many systems have unique features that utilize color-coding as results are entered, if they are within certain warning limits, the result is coded another color, and if they are outside warning limits the result is keyed yet another color. The effect of immediate feedback upon result entry allows analysts to double check their work and catch transcription errors prior to result validation and approval.

Another major automation enhancement includes the integration with instruments so that users do not have to re-enter instrument output files into the LIMS. The obvious advantages include; reduction of transcription errors, enhanced security, increased sample throughput and increased efficiency. However, a major advantage is the enhanced data quality. In addition, bi-directional instrument interfaces can be configured so that the LIMS sends the worklist and the order of the samples to be run to the instrument and once the samples have been analyzed, the instrument can export the data back to the LIMS in the correct format. This is especially useful with instruments, which are prolific in their output such as a tandem mass spec or an ICP.

#### QA/QC Functionality

A key feature of a LIMS is the ability to assign QC to samples, including: blanks, spikes, duplicates, and many other QC types. Users can also view control charts and view trends over times for various tests and at selected sites. Samples and QC standards are grouped into QC batches. It is then possible to view all the quality control related to a sample.

#### Resource Management

Many systems offer the functionality that will also keep track of employee training records, certification information, re-certification dates, and a description of the training. Users can also keep track of instrument calibration, maintenance, repairs, and calibration dates.

#### Chemical and Reagent Inventory

The ability to track reagents and chemicals in the laboratory, track lot numbers, expiration date, quantities on hand, vendor information and to create custom reports to reorder items with long lead times as in-house quantities are running low.

#### A few capabilities of a computerized LIMS that allow users to meet regulatory requirements:

- Document Management the ability to have on-line SOPs (Standard Operating Procedures) which are linked in the LIMS and available to analysts performing the various protocols.
- Chain of Custody within the Laboratory a detailed record of each location that the sample moved to and from during the analysis process, the date and time stamp, which analysts handled the samples and the storage location.
- Electronic Signatures each user must log in with a unique user name and password.
- Instrument Calibration instruments must be in calibration for results obtained on those instruments to be valid. A LIMS can compare the calibration date to the analysis date and if a particular instrument is past due for calibration, it can exclude the user from entering data for that instrument.
- *Employee Training Records* analysis must pass minimum qualifications for performing analytical analysis methods. The LIMS can block users that lack the training accreditations.
- Limit Checking users are alerted immediately and on-screen when results entered exceed the prespecified limits that were previously established.

**Table 1** reflects the regulatory compliance landscape and how it impacts various data management systems. There are numerous regulatory requirements and compliance documents that define how the data in a LIMS should be accessed, maintained and protected. CFR 21 part 11 deals primarily with electronic signatures, however there are several other aspects as well, several that deal with laboratory practices and procedures. As illustrated in the table below, there is considerable overlap between the various regulatory guidelines. LIMS administrators need to keep abreast of the latest regulations to ensure that the software solutions that they have implemented in their environments are compliant.

21 CFR	SOX-IAS	HIPPA	Patriot Act	Basel II
	x			x
	x			x
	x			x
x	x	× k		×
×	x	×	x	x
×	x	×	x	×
	x	x x x x x x x x x	x x x x x x x x x x x x x x x x x x x	x x x x x x

#### Advantages of Automated Reporting

Another major advantage of a computerized LIMS is automated reporting. Reports can be configured to automatically be printed to a specified server, auto-faxed or auto-e-mailed in a PDF format, which cannot be modified. The LIMS allows users to configure each customer as to how they will receive their report, either via fax, e-mail or hard copy or via the web portal, or any combination.

#### Washington Aqueduct LIMS

Since January 2004, the Washington Aqueduct has processed over 11,000 lead samples. The majority of these samples are for compliance with the EPA Lead and Copper Rule. The samples are for various customers with each customer having their own reporting requirements and turn-around times. The LIMS has been essential to meeting the demands of customers and the EPA.

#### Sample Receiving

Lead samples received by the laboratory usually belong to one of three programs: Lead and Copper samples, Lead Service Line Replacement Samples, Lead Profile Samples. Samples are received into the laboratory where they are logged into the LIMS and assigned a unique sample identification number. Turn around time requirements are then selected from a pull down menu. Each of the lead service line replacement samples has a unique homeowner id assigned to them by the contractor managing the collection of these samples. This ID is logged into the LIMS using the Customer Sample ID field in the LIMS (*Figure 1*). The lead service line replacement samples are delivered in batches to the laboratory. In addition to a chain-of-custody form for each sample, there is a bulk chain-of-custody form for the batch of samples.

Order ID	Sample ID	Test	Customer Sample ID	Site	Route id
0505101	0505101-006	Lead	LR558771-2	3034 RODM	Special
0505101	0505101-007	Lead	LR415431-1	1816 D ST 5	Special
0505101	0505101-008	Lead	LR415431-2	1816 D ST 5	Special
0505101	0505101-009	Lead	LR537823-1	1728 L ST N	Special
0505101	0505101-010	Lead	LR537823-2	1728 L ST N	Special
0505101	0505101-013	Lead	LR412833-1	1730 BAY S	Special
0505101	0505101-014	Lead	LR412833-2	1730 BAY S	Special
0505101	0505101-017	Lead	LR421307-1	5203 KANS/	Special
0505101	0505101-018	Lead	LR421307-2	5203 KANS/	Special
0505101	0505101-019	Lead	LR579713-1	4300 KANS/	Special
0505101	0505101-020	Lead	LR579713-2	4300 KANS/	Special
0505101	0505101-023	Lead	LR539177-1	1801 D ST 5	Special



#### Sample Workload Management

One primary analyst is responsible for analyzing all the lead samples. It is their responsibility to analyze all the samples within their required turnaround times. The Lead Service Line Replacement samples have a 4day turnaround time requirement. The Lead and Copper Samples have a 14-day turnaround requirement and the Lead Profile samples have a 21-day turnaround requirement. The analyst is able to use the Custom Report function of the LIMS to pull up a report listing samples to be analyzed and their associated due dates. This report allows the analyst to prioritize the analysis of his samples as required.

#### Data Entry, Validation and Approval

The results of all samples are entered into the LIMS. The EPA action levels for lead have been programmed into the LIMS so that when a result is greater than the EPA action limit, the result is flagged in red. This alerts the analyst immediately during data entry so that they can confirm that the high result is not a data entry error.

The Washington Aqueduct LIMS is configured that all entered results must be validated by the QC Officer and then approved by the Laboratory Chief before any data can be reported to a customer. The color coded results easily alert the QC Officer and Laboratory Chief to unusual results so that the customer can be immediately notified. The QC officer is able to view the QC results for samples at the same time as the individual sample results. This facilities the speed in which the QC Officer can validate data. In addition, the LIMS contains several user customizable fields. The Washington Aqueduct Laboratory has customized the field so that when the QC Officer does a raw data review by reviewing the lab bench sheets on certain samples, he can mark this field. During EPA inspections a query on this field easily relates to the inspectors the number of samples that have undergone a thorough raw data review in addition to a reasonableness check.

#### Data Reporting

Each customer and each program have unique reporting requirements. The LIMS allows the laboratory to assign reports to special projects and tests for each customer **(Figure 2)**. Each report can be designed to include as much or as little information as each customer desires.

Customers			
Customer:	WASA	■ District of Columbia Water and Sewer Ar	
) etails   Conta	acte   Projecte	Project Sampling   Project Pricing   Project Parameters   Project QC Types   RDLs	Reports
		The company in the co	nopone
Project ID:	Special		
	Test:	ReportName:	
Lead		WAD_Metals_autoreport	
	Copper	WAD_Lead_Copper_Autoreport	-
*			
Record: 1		2 • • • • • • • • • • • 2	
Record: IN			

Figure 2

The reports are generated in PDF format and e-mailed to the customer. All contact information for a customer is programmed into the LIMS for automatic delivery if desired. This includes a fax number and e-mail address. There can be more than one contact per customer so that a report may be mailed to multiple parties at once. Currently, all reports are initially e-mailed to the Laboratory Chief. The Laboratory Chief reviews the PDF file and attaches a digital signature using public key infrastructure (PKI) *(Figure 3)*. Each customer has previously been distributed a copy of the private key. The customer is then able to click on the digital signature to see a copy of the digitized signature *(Figure 4)*. The digital signature ensures that reports cannot be modified or changed.



Figure 3

#### NEMC 2005 Proceedings

9 <u>6-76</u>	Name:	Elizabeth Turner	
S	erial number:	0AB5DB6A	
Certificate not	t valid before:	2004.04.23 10:16:43 -04	'00'
Certificate n	ot valid after:	2009.04.22 10:16:43 -04	'00'
User's disting.	uished name ((	N):	
cn=Elizabeth	Turner, o=Wa	ashington Aqueduct, c=US	
Certificate issi	uer's distinguis	hed name (DN):	
cn=Elizabeth	Turner, o=Wa	ashington Aqueduct, c=US	
Key usage:			Key algorithm:
Sign docume	nt, Sign transa	action, Encrypt document	RSA 1024-bit
- Fingerprir	nts		
MD5: 8	050 9324 CDC:	1 3E3F 4891 ED0D 6142 B8	34
SHA1: 6	4C3 84E2 164	3 EE57 70D8 A6F9 82B4 A4	EN E943 9628
Or man 1			



This feature of the Washington Aqueduct Laboratory reports was critical during an EPA data audit. There were discrepancies in some of the data reported to the EPA. The EPA was able to review all the Lead and Copper Reports that the Washington Aqueduct had created and transmitted to our customers. The digital signature and dated e-mails containing the reports provided an audit trail on the generation of reports and distribution to customers.

#### Data Export

In addition to the PDF files, the Washington Aqueduct customers require the data in Excel format so that they may be able to import the data into their own databases. Thus, the customer receives two files: an official PDF report containing a digital signature and an Excel file which the lab will not certify as official lab data. The MS Excel summary report is done as a courtesy to our customers.

Sample Information       Worklist ID:         QC Batch ID:         Prep Batch ID:         Order ID:         Sample ID:	Customer Information Customer ID: WASA Customer ID: UVASA Customer Name: District of Columbia W Route id Cust. Samp ID: Rec'd 1 emp [U]:
Matrix:     ▼       ✓     Test:     Lead & Copper     ▼       ✓     Method:     ▼       ✓     Parameter:     ▼       ✓     Department:     ▼       ✓     Department Status:     ▼       ✓     Date Collected:     4/1/2001        ✓     Date Collected:     4/1/2001        ✓     Date/Time Recv'd:         ✓     Sample Due Date:         ✓     Prep Due Date:	Site:  Betrieve Close
Prep Due Date:	

Figure 5

The "Master Query" Window was used to easily query data in the LIMS by test, customer and collection date range (*Figure 5*).

The exporting feature of the LIMS allowed the Washington Aqueduct to quickly respond to Freedom of Information Request. Once the data was retrieved it could quickly be exported to Excel. The excel file was then reviewed for formatting and password protected (*Figure 6*).

Disp © ©	# Results All Results (* Results to E Results to V Results to A	view only) ter Mi Mi Mi Mi Mi Mi Mi	ta Destination: tus (WK4) crosoft Excel versio crosoft Excel versio crosoft Excel versio crosoft Excel versio crosoft Excel 97 Include Field 1	n 3.0 n 4.0 C n 5.0 n 7.0 (9!	OK ancel de Cl mple				
Samp	ble Results	Sample Surrogate	s   Blank Results	Blank Surrogates	Spike Results   S	Spike Surrogati	es   !	Star	ndard Results
	Order ID	Sample ID	Test	Parameter	Result	Units	TC	+	Site
•	0504007	0504007-001	Lead & Copper	Iron	ND	ug/L			2139 Domir
	0504007	0504007-001	Lead & Copper	Copper	27.49	ug/L			2139 Domin
	0504007	0504007-001	Lead & Copper	Lead	0.5697	ug/L			2139 Domin
	0504007	0504007-002	Lead & Copper	Iron	ND	ug/L			2151 Domir
	0504007	0504007-002	Lead & Copper	Copper	26.62	ug/L			2151 Domin
3-9	0504007	0504007-002	Lead & Copper	Lead	0.8867	ug/L			2151 Domin
	0504007	0504007-004	Lead & Copper	Iron	2.666	ug/L			8302 Fores
3-2	0504007	0504007-004	Lead & Copper	Copper	9.173	ug/L			8302 Fores
	0504007	0504007-004	Lead & Copper	Lead	0.2130	ug/L			8302 Fores
	ord: <u>  </u>			26					•
<u>C</u>	lose	-		C: Result is Commer	nted				Audi

Figure 6

#### Conclusions

The numerous features of a user-friendly LIMS are critical in allowing laboratories to quickly respond to enhanced monitoring situations, from rapid sample login through to sample tracking and final reporting. There are several quality assurance and quality control features in a computerized LIMS that ensure a much higher data quality than in manual systems or non-relational databases. In the LIMS system used by the Washington Aqueduct, the LIMS administrator configured the various sites, sampling schedules, tests, parameters, and methods, with default methods and pre-assigned QC to ensure that the proper tests are performed on the proper samples. A key QC feature is peer review which ensures that the analyst that reviewed the sample cannot validate or approve the results for that sample, a QC officer or laboratory manager must review the results.

In addition to managing the thousands of samples analyzed annually by the laboratory, the LIMS also provides functionality that assists laboratories to meet regulatory requirements as those outlined in the beginning of this paper. A LIMS is a necessity in today's modern laboratory and critical to ensuring that high quality data is the output from all the sampling and analysis efforts that ensure a safe potable water supply.

## Qualification Testing by Japan Environmental Measurement & Chemical Analysis Association (JEMCA) – Preparation of samples, Data Analysis & Evaluation, and Feedback of Information

## Hideo Tabata, Mitsuo Hamaji, and Toru Matsumura

## ABSTRACT

Japan Environmental Measurement & Chemical Analysis Association (JEMCA) was founded in 1973. Currently, 560 Japanese private entities engaged in environmental measurement and analysis hold membership of the association. JEMCA is a board member of UILI from 2001, and is also active as an affiliate member of ACIL from 2001.

JEMCA has been conducting qualification testing since 1999 to ensure technology improvement and to secure confidence in quality management and measurement data in laboratories. The qualification test is conducted by having the membership entities to analyze common samples distributed by JEMCA, and evaluating the measurement results statistically. So far, 26 qualification tests have been implemented using environmental media such as environmental water, effluents, soil, and environmental air, with analyzed items such as pH, COD, Nitrogen, Phosphorus, Heavy Metals, VOC and Pesticides, etc.

Application for the qualification test can be done on JEMCA website. Historical test information and statistical evaluation results can also be obtained on the website.

In this presentation, methodology used in JEMCA for sample preparation, statistical data evaluation, and feedback of technical information to membership entities is introduced.

# Significance of Changes in 2005 NELAC/USEPA Proficiency Testing Requirements

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## ABSTRACT

Over the past year, there have been many changes in proficiency testing requirements that laboratories must comply with to become accredited. In 1998, USEPA published a Criteria Document that established PT requirements for the potable and non-potable water analytes that the Agency included in their historical PT programs. These included organic and inorganic chemistry, microbiology, radiochemistry and whole effluent toxicity. NELAC established requirements for additional potable and non-potable water analytes in water and requirements for RCRA-Solids.

In 2004, the NELAC program was split into laboratory accreditation Standards development (INELA) and regulatory Standards adoption (NELAC). A Proficiency Testing Board, with state and EPA participation, was established to set PT policy. Working under the PT Board, a broadly-based Fields of Proficiency Testing subcommittee worked to resolve issues with the historical PT requirements and establish expanded FoPTs to better respond to laboratory accreditation needs. In November 2004, NELAC published a major revision of the PT requirements, which must be implemented June 1, 2005. For the potable and non-potable water programs, both "Accreditation" and "Experimental" tables of analytes and PT requirements were established. The significance of the two sets of tables will be discussed and the list of PT analytes will be compared to those regulated or required by major state and federal programs. As it is likely that USEPA will soon defer to these new and revised PT requirements, they will apply to laboratories in non-NELAC as well as NELAC states.

In this paper, we will explain the significant changes in sample design requirements, reporting and acceptance limits. The expected impact on laboratory PT data quality expectations and pass/fail rates will be presented. The consistency of acceptance limits with other measures of laboratory performance such as LCS limits will also be presented.

## Newly Developed Biota- and Biological-related Standard Reference Materials for the Determination of Organic Contaminants

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## ABSTRACT

Since 1990, the National Institute of Standards and Technology (NIST) has issued a number of cryogenically homogenized tissue SRMs with certified and reference values assigned for organic contaminants. The cryogenically homogenized materials are powder-like with the endogenous water retained. We recently reviewed the development and availability of musseltissue SRMs<sup>1</sup> and marine-related tissue SRMs<sup>2</sup>. The series of natural mussel-tissue SRMs (Organics in Mussel Tissue, Mytilus edulis) has been developed from mussels collected in Boston Harbor, MA. SRM 1974b is the third and current material in this series and has certified and reference values for a range of polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners (1 non-ortho), total PCBs, chlorinated pesticides, methyl-Hg, Hg, selected trace elements, and total extractable organics. An additional mussel tissue SRM, SRM 2977, is also available. This is a freeze-dried tissue homogenate prepared from mussels collected in Guanabara Bay, Brazil<sup>1</sup>. Two cryogenically homogenized fish tissue SRMs<sup>2</sup> have been developed from filleted adult lake trout (Salvelinus namaycush namaycush). SRMs 1946 (Lake Superior Fish Tissue) and 1947 (Lake Michigan Fish Tissue) are characterized for a range of PCB congeners, chlorinated pesticides, methyl-Hg, Hg, selected trace elements, fatty acids, calories, and proximates. SRM 1946 was also examined for total toxaphene and toxaphene congeners<sup>3</sup> and SRM 1947 has been examined for selected polybrominated diphenyl ether (PBDE) congeners.

Two biologically-related SRMs are a cod liver oil SRM, SRM 1588a, and a human serum SRM, SRM 1589a. SRM 1588a is a reissue of the original cod liver oil SRM 1588 with an expanded list of PCB congeners and chlorinated pesticides having certified concentrations. The material has been examined for selected PBDE and toxaphene congeners, and total toxaphene<sup>3</sup>. Concentration values for additional PCBs, chlorinated pesticides, PBDEs, and selected fatty acids will be added to the material's Certificate of Analysis. The human serum SRM, SRM 1589a, was certified in conjunction with the Centers for Disease Control (CDC) with certified concentrations for natural levels of selected PCB congeners and chlorinated pesticides along with reference values for selected polychlorinated dibenzo-p-dioxins/dibenzofurans (PCDDs/PCDFs) congeners. Two new SRMs for serum analyses currently in development are SRMs 1957 and 1958. SRM 1957 will be characterized for natural levels of selected PCDDs, PCDFs, PCBs, PBDEs, chlorinated pesticides, toxaphene congeners, polychlorinated naphthalenes, and other halogenated compounds. SRM 1958 will be characterized for the same suite of analytes though these compounds will be added to the material. Measurements of organic contaminants in the biota- and biologically-related SRMs will be presented with an emphasis on the approach and methods used for the chemical characterization of these naturalmatrix SRMs.

- 1. D. L. Poster, J. R. Kucklick, M. J. Lopez de Alda, B. J. Porter, R. S. Pugh, M. M. Schantz, and S. A. Wise, *Analytical and Bioanalytical Chemistry* 378 (2004):1213-1231.
- 2. D. L. Poster, J. R. Kucklick, M. M. Schantz, B. J. Porter, S. D. Leigh, and S. A. Wise, *Analytical and Bioanalytical Chemistry* 375 (2003):223-241.
- 3. J. R. Kucklick, K. J. S. Tuerk, S. S. Vander Pol, M. M. Schantz, and S. A. Wise, *Analytical and Bioanalytical Chemistry* 378 (2004):1147-1151.

# **Session 3**

# Inorganic Methods: Elemental Analysis by ICP Techniques

## **Current Status of the RCRA Inorganic Methods Program**

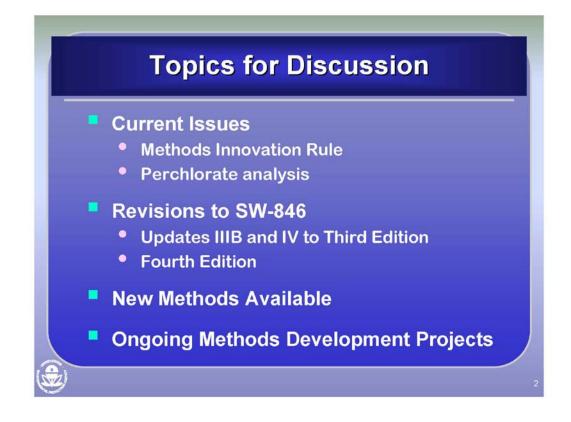
### Shen-Yi Yang

US EPA Office of Solid Waste Economics, Methods, and Risk Analysis Division, 1200 Pennsylvania Ave., NW (5307W), Washington, DC 20460 E-mail: <u>Yang.Shen-Yi@epamail.epa.qov</u>

## ABSTRACT

This keynote presentation will give an overview of the RCRA methods development activities for Update IV SW 846 methods (for inorganics and metals). This will include details on developments on the Methods Innovation Rule, Perchlorate monitoring, updates to SW-846 methods, new methods and method development.



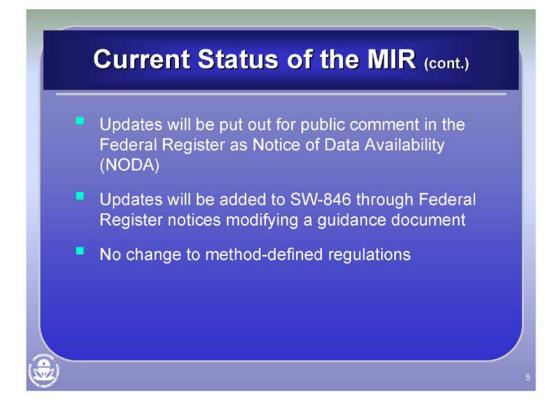


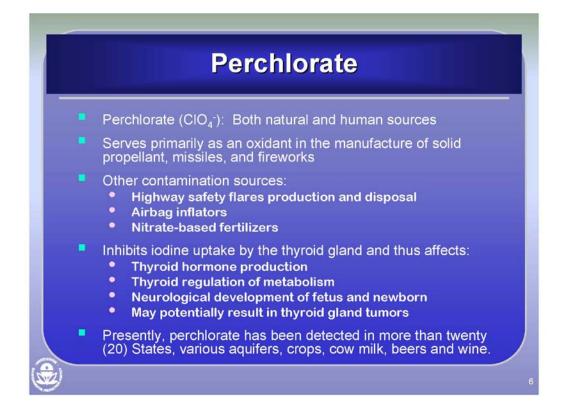


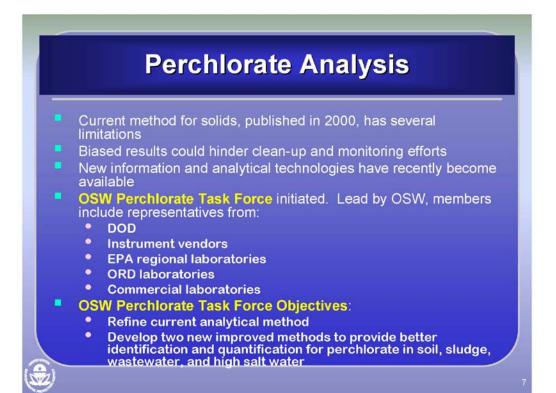
- Regulation development initiated to remove mandatory requirements to use SW-846 methods for analyses that are not method-defined parameters in the RCRA regulations
- MIR proposed on October 30, 2002 (67 FR 66251)
- Comment period closed on February 28, 2003
- OSW Methods Team completed responses to public comments

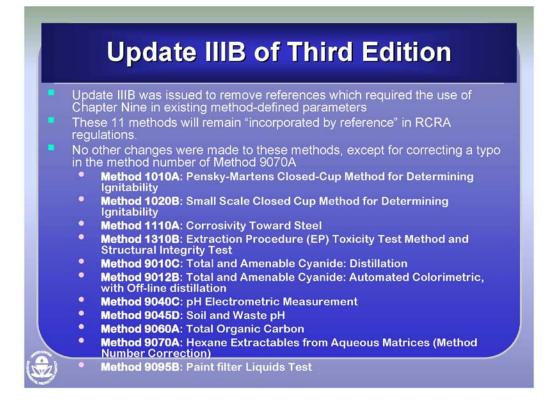


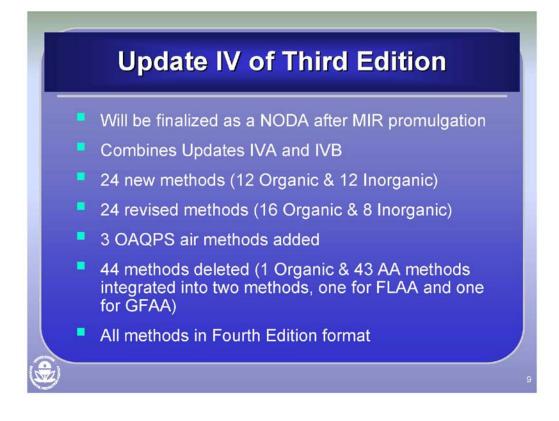
- Methods Team prepared the Final Rule and addressed all procedural issues
- Final Rule received side agreement concurrence
- Final Rule was signed by the EPA Administrator and published on June 14, 2005 (70 FR 34537)
- Promulgation of MIR eliminates the need to publish SW-846 Updates as regulation
- SW-846 functions as originally intended: as a "guidance document"











## New Inorganic Methods in Update IV

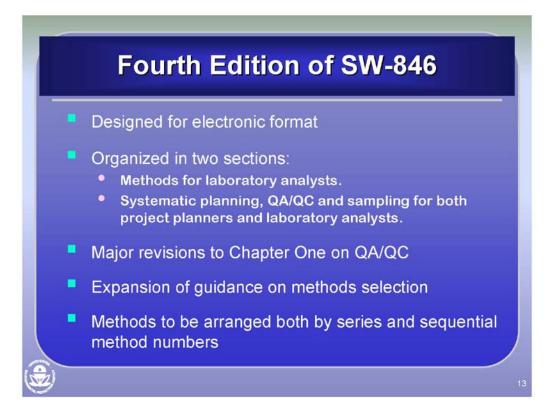
- Method 1040: Test Method for Oxidizing Solids
- Method 1050: Test Methods to Determine Substances Likely to Spontaneously Combust
- Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment
- Method 6500: Dissolved Inorganic Anions in Aqueous Matrices by Capillary Ion Electrophoresis
- Method 6800: Elemental and Speciated Isotope Dilution Mass Spectrometry
- Method 7010: Graphite Furnace Atomic Absorption Spectrophotometry
- Method 7473: Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry
- Method 7474: Mercury in Sediment and Tissue Samples by Atomic Fluorescence Spectrometry
- Method 9000: Determination of Water in Waste Materials by Karl Fisher Titration
- Method 9001: Determination of Water in Waste Materials by Quantitative Calcium Hydride Reaction
- Method 9058: Determination of Perchlorate Using Ion Chromatography with Chemical Suppression Conductivity Detection
- Method 9216: Potentiometric Determination of Nitrate in Aqueous Samples with Ion-Selective Electrode

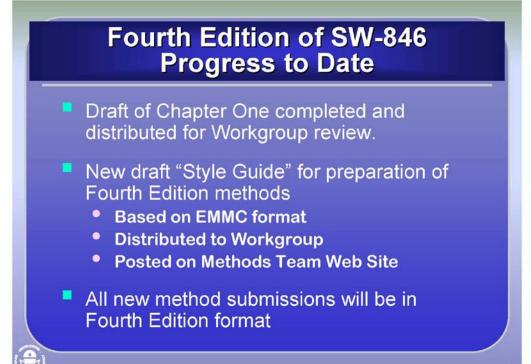


- Samples and Extracts
- Method 3051A: Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
- Method 6010C: Inductively Coupled Plasma Atomic Emission Spectrometry
- Method 6020A: Inductively Coupled Plasma Mass Spectrometry
- Method 7000B: Flame Atomic Absorption Spectrophotometry
- Method 7471B: Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)
- Method 9056A: Determination of Inorganic Anions by Ion Chromatography
  - Method 9210A: Potentiometric Determination of Nitrate in Aqueous Samples with Ion-Selective Electrode



43 Individual Flame AA and Graphite Furnace AA methods integrated into two methods, **Method 7000B -** Flame AA and **Method 7010** -GFAA



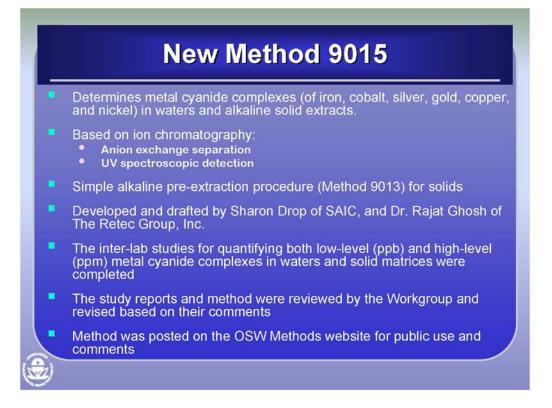


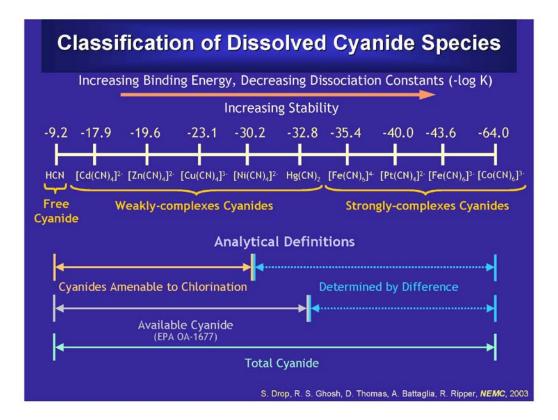
## Fourth Edition of SW-846 Progress to Date

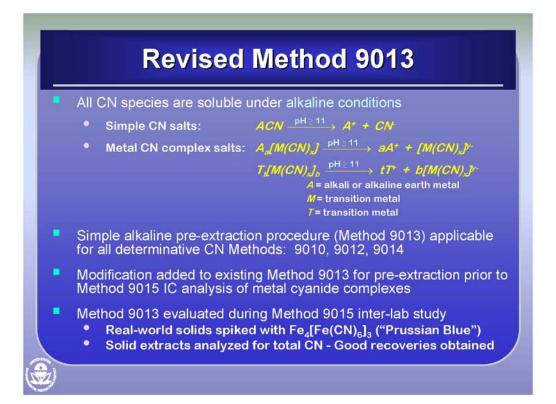
- All Third Edition methods currently being revised have been converted to Fourth Edition format including all Update IV methods and "New Methods"
- Original expected timeframe of having the Fourth Edition completed and ready for public comments concurrent with the promulgation of the MIR has now changed in light of the current budget situation.

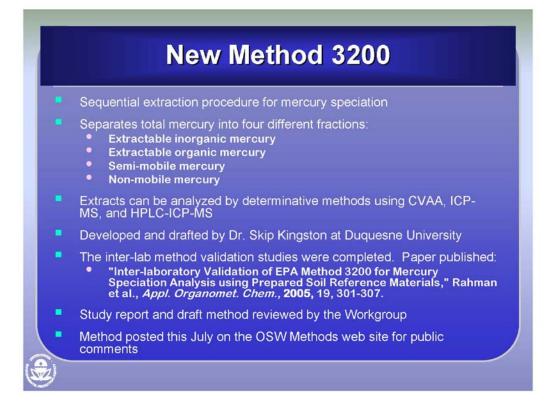
## Completed New and Revised Inorganic Methods for Fourth Edition

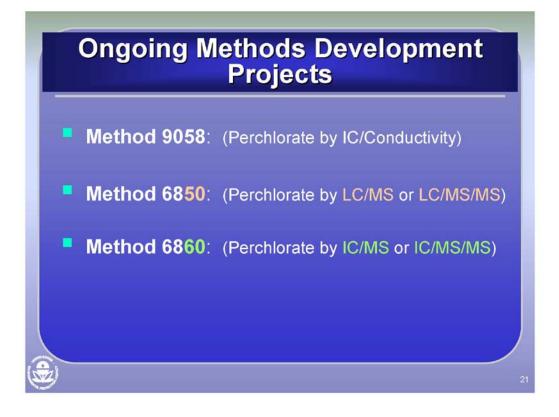
- Method 9015: Metal Cyanide Complexes by Anion Exchange Chromatography and UV Detection
- Method 9013A: Cyanide Extraction Procedure for Solids and Oils
- Method 3200: Mercury Species Fractionation and Quantification by Mircowave Assisted Extraction, Selective Solvent Extraction and/or Solid Phase Extraction

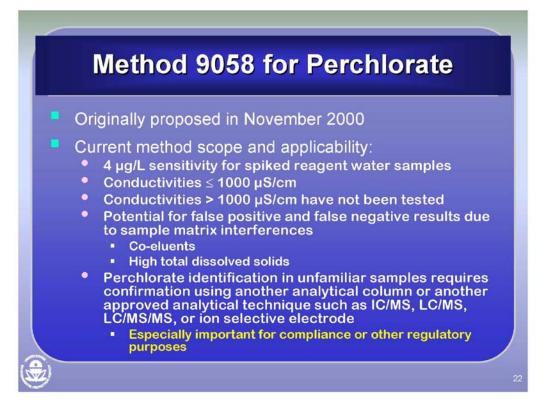




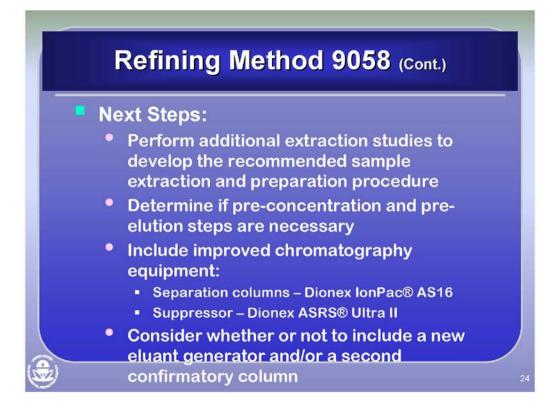






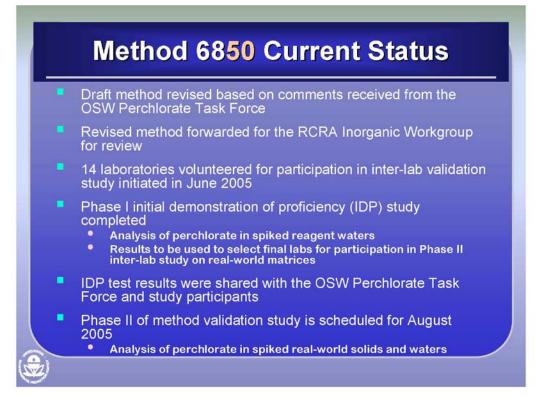


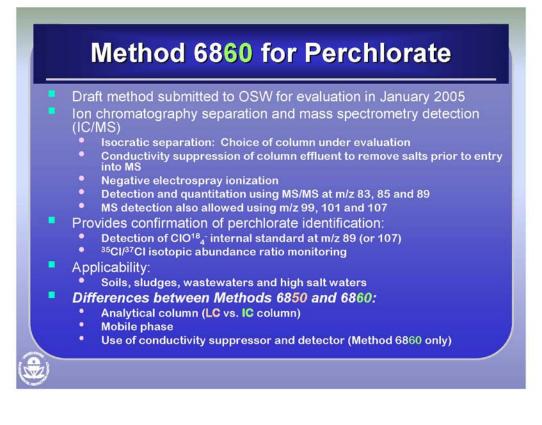


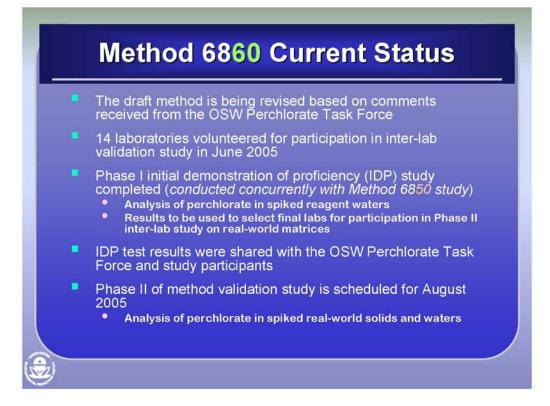




- Draft method submitted to OSW for evaluation in July 2004
- Based on high performance liquid chromatography separation and mass spectrometry detection (LC/MS)
  - Isocratic separation: K' (Prime) Technologies, Inc. KP-RPPX250 column
  - Negative electrospray ionization
  - Detection and quantitation using m/z 83, 85 and 89
- Provides confirmation of perchlorate identification:
  - Detection of CIO<sup>18</sup><sub>4</sub> internal standard at m/z 89
  - <sup>35</sup>CI/<sup>37</sup>CI isotopic abundance ratio monitoring
- Applicability:
  - Soils, sludges, wastewaters and high salt waters
  - Also possibly applicable to other matrices (e.g., biota), but these are not being evaluated by OSW
- Final method may allow flexibility in detection pending outcome of validation study:
  - MS detection m/z 83, 85, 89
  - MS detection m/z 99, 101 and 107
  - MS/MS detection m/z 83, 85, 89









# Technological Advances and Optimisation in ICP-OES to Meet the Demands of Modern, Routine Elemental Laboratories

#### Paul Neal

Thermo Electron Corporation, Mercers Row, Cambridge, UK. E-mail: <u>Paul.Neal@Thermo.com</u>

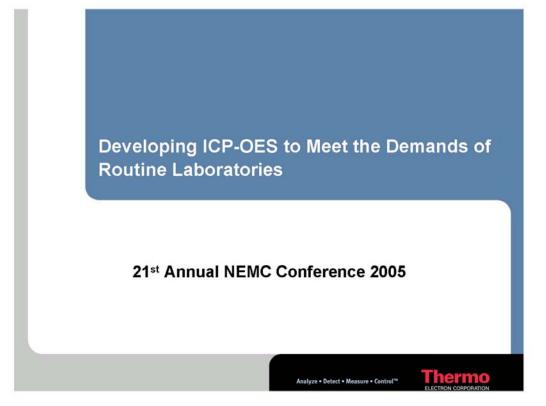
#### ABSTRACT

ICP-OES has become the technique of choice for a large range of applications. The technique is relatively interference-free and provides analysis in the low ppb levels for most elements.

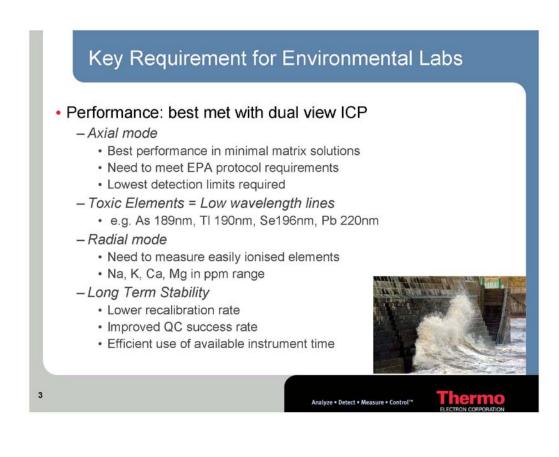
Modern laboratories require rugged, cost-effective and flexible ICP-OES solutions, which provide high throughput with low detection limits. In addition, laboratories are becoming increasingly aware of the need to provide easier set-up procedures for sample introduction, plasma optimisation and method development to maximise throughput and minimise the amount of operator time required.

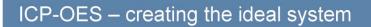
To meet the expectations of the analytical community, instruments need to increase sensitivity and stability thereby driving down detection limits to even lower levels all the while increasing productivity. Recent improvements in solid-state detector arrays, optical configurations and general instrument components will allow modern ICP-OES instruments to evolve and bring the technique closer to its full potential.

Technical solutions will be presented with supporting data to show how the technological advances have and will benefit the real analysis of environmental samples.









In order to overcome the following:

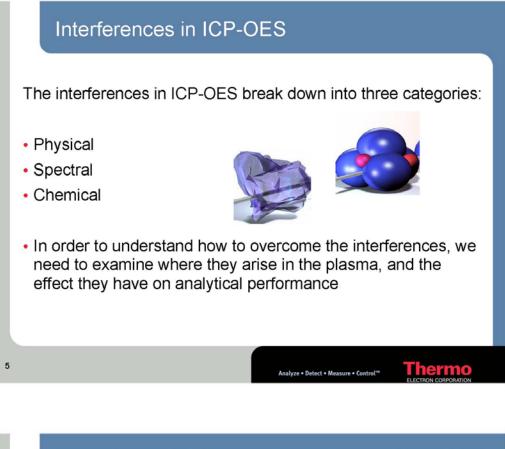
 Matrix tolerance, ionization effects, operating parameters effect on analytical performance

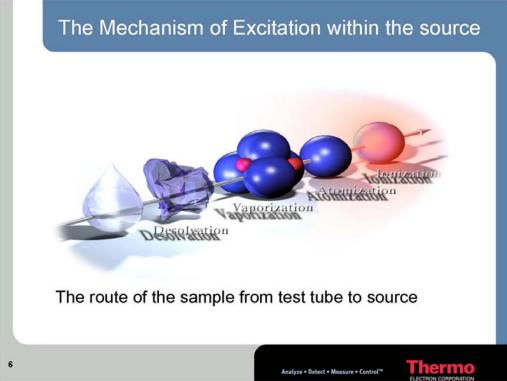
The ideal system should incorporate:

- Excellent matrix handling capabilities
- Robust source
- Easy user optimization

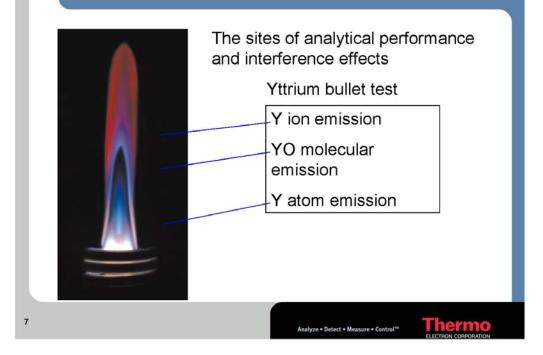


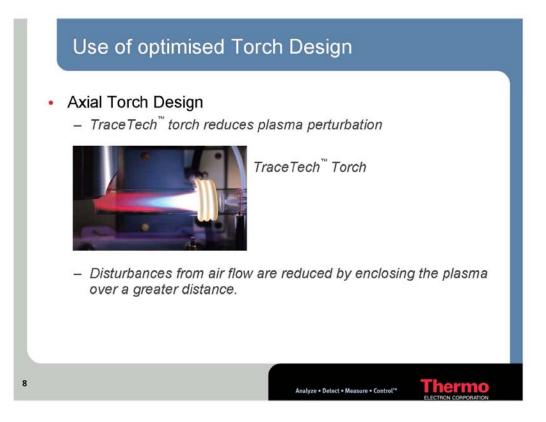
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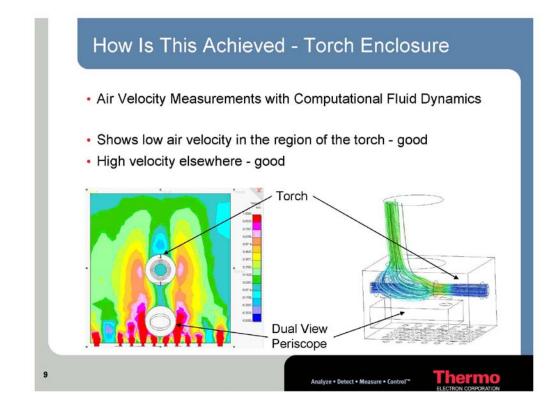






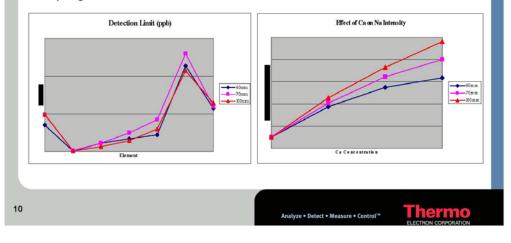




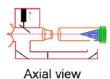


# Optimisation of torch length - 60mm,70mm,100mm lengths

- Torch length vs detection limit the longer torches provide better D.L.s than the shorter
- Effect of Ca on Na intensity shorter torches perform better coping with ionisation effects.



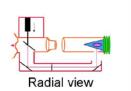
## **Dual View Optics**



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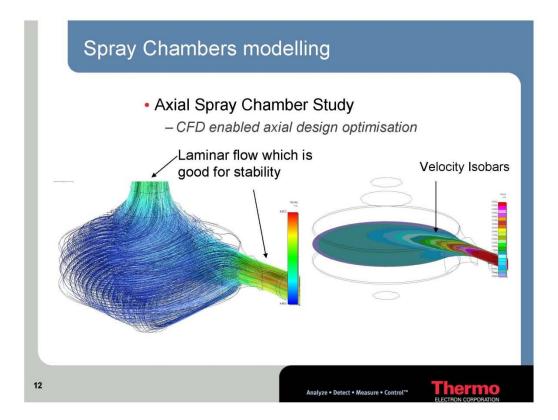
Viewed axially more light is obtained from the emission zone, providing increased sensitivity. However, it is not possible to focus only on the interference-free zones within the plasma. This configuration is more susceptible to plasma loading effects and easily ionized element interference

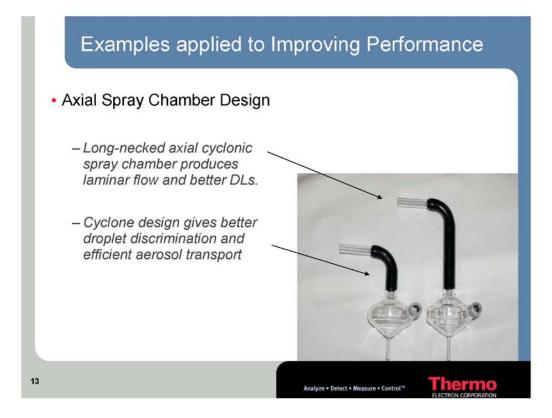
Viewed radially, the optics collect light only in the areas of ion and atom emission. The cooler zones of the ICP where interferences are present, are not viewed and are therefore not a problem

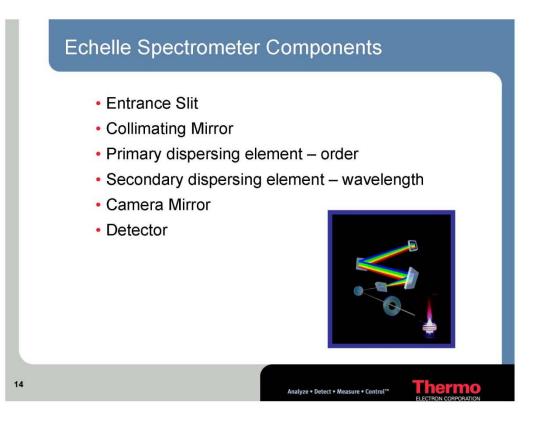


Analyze • Detect • Measure • Control<sup>\*#</sup>

Thermo







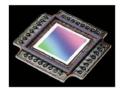
### 121



- · Simultaneous measurement of analytical signals
- Wide spectral response
- High quantum efficiency
- Low noise characteristics
- High Dynamic range

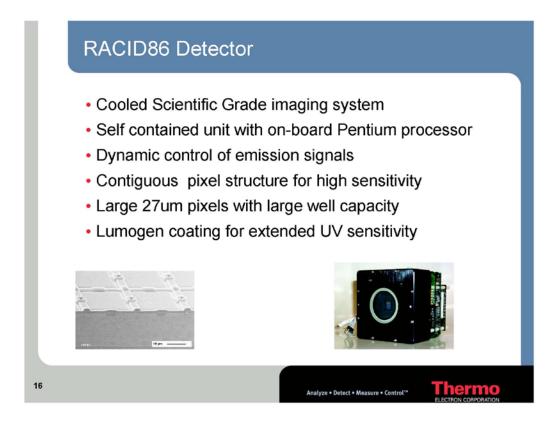
15

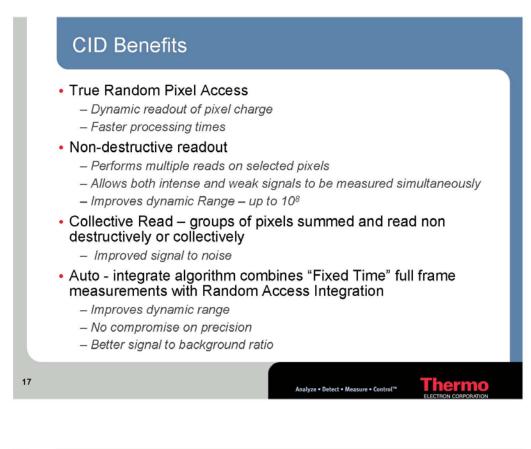
Fast measurement times

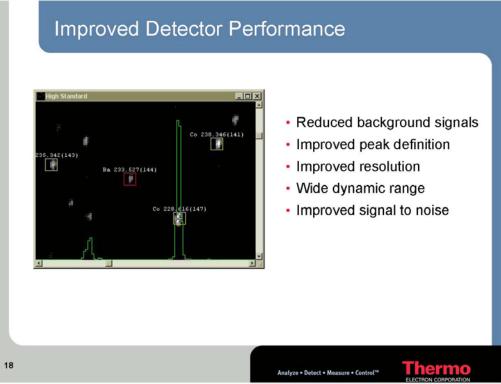


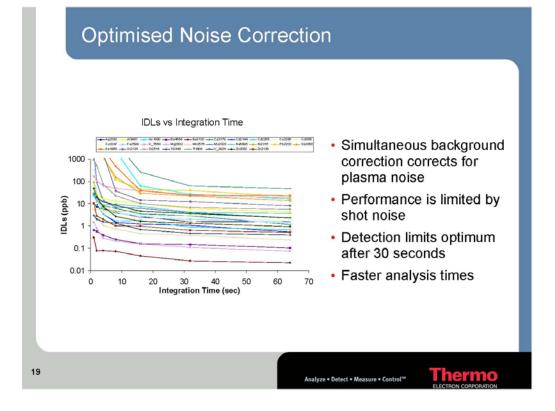
Analyze • Detect • Measure • Contr

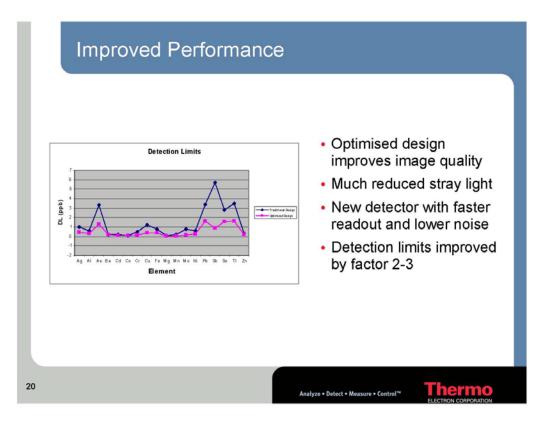
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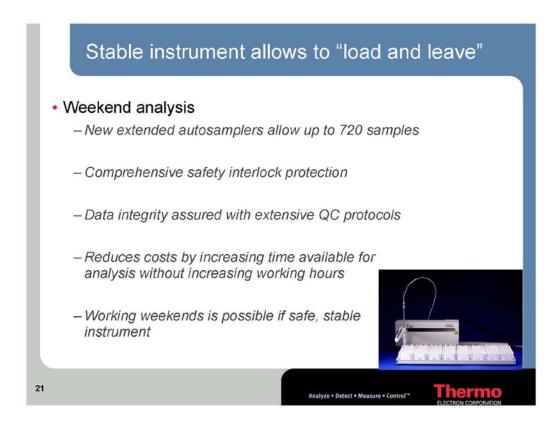




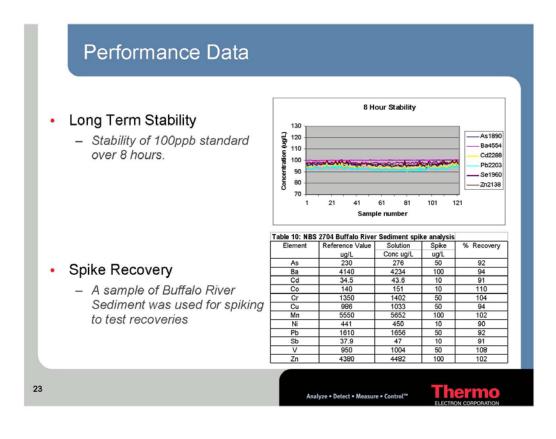








Element Ag Al As B Ba Ba Be	d Detection Limits Wavelength (rm) 338.289 308.215 189.042 208.959 455.403	Plasma View Axial Radial Axial	MDL (ppb) 0.5 11.4 2.1
Al As B Ba Be	308.215 189.042 208.959	Radial Axial	11.4
As B Ba Be	189.042 208.959	Axial	
B Ba Be	208.959		
Ba Be		Axial	2.2
Be		Radial	0.7
	313.107	Radial	0.1
Ca	317.933	Radial	2.6
Cd	214.438	Axial	0.1
Co	228.616	Axial	0.2
			0.3
			52
K	766.491	Radial	30
Li	670.784	Radial	1
Mg	279.079	Radial	40
		Axial	0.1
	2.0.2.100	1.00.00	0.5
			10
P	178.287	Axial	3.2
Pb	220.353	Axial	2.1
Sb	206.833	Axial	2.4
			2.8
			7.2
			1.8
TI			1.6
V	292.402	Axial	0.6
Zn	206.2	Axial	0.2
	Li Mg Mn Na Na Ni Pb Sb Sb Sb Sb Sb Si Sn Ti Ti V	Cu         324 754           Fo         271 441           K         766 491           Li         670 784           Mg         279 079           Mn         257 61           Mo         202 03           Na         599 592           Ni         231 604           P         178 287           Pb         220 353           Sb         206 833           Sce         196 9           Si         251 612           Si         251 612           Si         1384 941           Ti         190 884           V         292 402	Cu         324 754         Axial           Fe         271 441         Radial           Ki         786 491         Radial           Li         670 704         Radial           Mg         279 079         Radial           Mn         257 61         Axial           Mo         202 03         Axial           Na         599 592         Radial           P         178 287         Axial           Pb         1220 353         Axial           So         196 9         Axial           Si         251 612         Radial           Si         251 612         Radial           Ti         334 941         Axial           V         292 402         Axial



### Concluding remarks

- There are many areas that can be improved in routine lab analysis
  - Throughput: sample handling and measurement
  - Performance to meet the needs of regulations today and tomorrow
  - Stability to improve the reliability and repeatability of measurement
- There are many tools that can be applied to understand and improve ICP-OES instrumentation
  - Computer aided design (CAD)
  - Computational fluid dynamics (CFD)
  - Finite Element Analysis (FEA)

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- Design for manufacture/assembly (DFM/DFA)
- Expect higher performance, more stable instrumentation with higher throughput in the future.

#### Analyze • Detect • Measure • Control<sup>114</sup>

Thermo

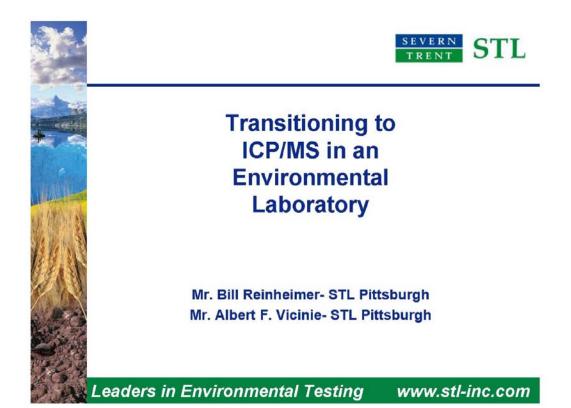
# Valuation and Comparison of ICP and ICP-MS for Environmental Applications

#### Mr. Albert F. Vicinie, Mr. William Reinheimer

Severn Trent Laboratories, 301 Alpha Drive, RIDC Park, Pittsburgh, PA 15238 Rvicinie@stl-inc.com

#### ABSTRACT

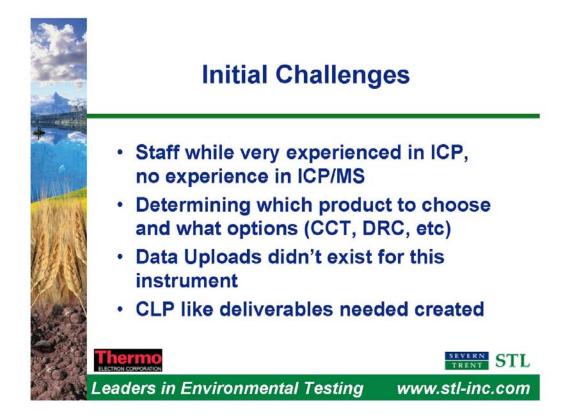
STL Pittsburgh is a full service environmental laboratory that provides analytical services to both routine and specialty market segments in the determination of metals in a variety of matrices. This presentation is to review the process and challenges of evaluating and implementing new techniques, different technologies and design approaches of various manufacturers. It will also provide a review of the factors motivating laboratories to transition from ICP-AES to ICP/MS, the benefits realized resulting from the tradition and an evaluation of its effectiveness in a production environment and comparison to previous generation ICP/MS currently in use.

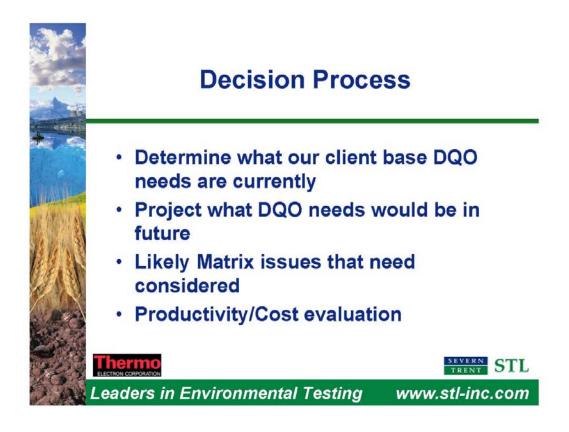


## "Pre-existing Condition"

1 TJA 61E ICP AES (radial) - Primarily for TCLP determinations - Used for Ca, Mg, Na, K 2 TJA Trace-ICP AES (axial) slightly different element configurations - Primarily for GW, WW, soils and tissues Worksharing GFAA ~ 2,600+ billable samples/month hermo TRENT Leaders in Environmental Testing www.stl-inc.com



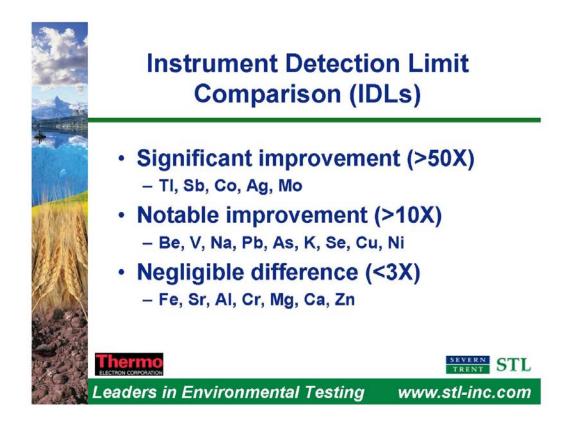










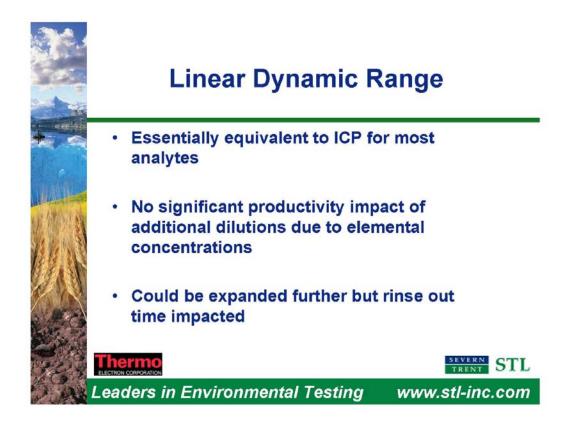


	Instrument Detection Limit Comparison (IDLs) ug/L									
Eller Do	Element	ICP/MS	ICP	Element	ICP/MS	ICP				
e e	TL	0.0035	3.3	B	0,106	1				
LUNNN .	SB	0.0086	3.1	MN	0.0228	0.19				
	co	0.0051	0.46	П	0.089	0.63				
U AMANIN SA	AG	0.0093	0.66	SI	0.978	6.1				
<b>新 相關的 \$</b>	MO	0.03	2.1	CD	0.046	0.23				
4 1 1 2	BE	0.014	0.45	SN	0.563	2.7				
4 1/4 / NV X	V	0.0347	1.1	BA	0.178	0.58				
12 2 2 2 2 2	NA	4.115	130	FE	5.016	12.9				
1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	PB	0.0468	1.4	SR	0.0873	0.22				
12 11 3	AS	0.061	1.5	AL	10.12	25.3				
	ĸ	3.67	67.3	CR	0.3	0.62				
	SE	0.18	2.5	MG	5.01	9.6				
LAND WA	CU	0.113	1.3	CA	6.44	8.2				
State of the second	NI	0.105	1.2	ZN	2.36	0.46				
The second	Thermo ELECTRON CORPORATION					RENT STL				
2 AL	Leaders in	Environn	nental	Testing	www.stl	-inc.com				

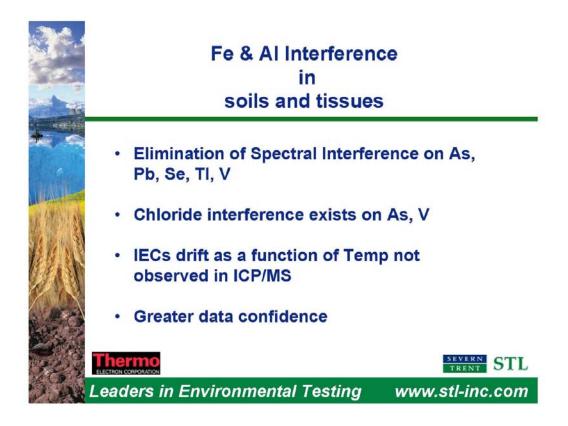
	Method Detection Limit Comparison Method 6020 Vs Method 6010B ug/L							
		ICP/MS MDL			ICP/MS MDL			
a flat and the	TL	0.014	2.800	ZN	0.560	3.000		
	co	0.014	0.630	B	0.570	0.610		
A LEAST AND A LEAST	MN	0.044	0.110	CR	0.610	0.760		
	PB	0.044	1.980	TI	0.670	0.690		
and Aller and a star	AG	0.050	0.490	MG	1.970	6.280		
	SR	0.054	0.170	к	3.210	33.300		
<b>新兴</b> 中国方言 美洲	SB	0.073	4.000	SI	3.650	7.000		
A Star Value	BE	0.096	0.710	NA	3.850	228.000		
13.18 1 20 2	v	0.100	1.300	CA	4.780	40.400		
	CD	0.120	0.140	AL	5.160	24.100		
	MO	0.130	1.200	-				
	CU	0.140	0.760					
	NI	0.180	2.660					
	BA	0.190	0.480					
	SE	0.300	3.100					
Carlos and a	AS	0.340	1.330					
	FE	0.380	22.800					
	PORATION	0.463	3.430			SEV	INT STL	
ALC DIST	ers in Er	vironm	ental	Testin	g w	ww.stl-	inc.com	

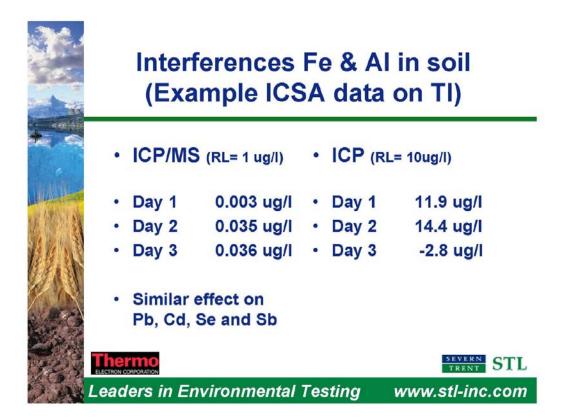
# **Tissue DQO Comparison**

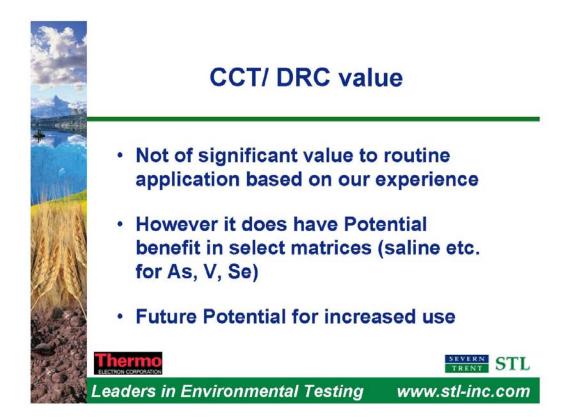
* ~									
and the second se		6010	6010	6020	6020				
the first state of the	ANALYTE	RLs	MDLs	RLs	MDLs	TDL <sup>1</sup>			
C. C.	Silver	0.500	0.388	0.100	0.0310	0.100			
ALC A CAN'S	Aluminum	20.000	6.515	3.000	0.4500	1.000			
	Arsenic	1.000	0.870	0.100	0.0120	0.100			
The state of the second s	Beryllium	0.400	0.144	0.100	0.0070	0.100			
· · · · · · · · · · · · · · · · · · ·	Cadmium	0.500	0.143	0.100	0.0070	0.100			
A CONTRACTOR	Cobalt	5.000	0.120	0.050	0.0030	0.100			
S	Chromium	0.500	0.110	0.200	0.0270	0.100			
4 11 N. 2	Copper	2.500	0.598	0.200	0.0110	0.100			
S 62 1 5 5	Iron	10.000	1.313	5.000	0.6180	10.000			
1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1	Manganese	1.500	0.010	0.050	0.0088	0.500			
1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	Nickel	4.000	0.477	0.100	0.0198	0.100			
	Lead	0.300	0.231	0.100	0.0002	0.100			
	Antimony	1.000	0.181	0.200	0.0083	0.100			
	Selenium	0.500	0.338	0.500	0.0190	0.200			
	Tin	10.000	1.414	0.500	0.2330	0.100			
ALC: NOT COME	Thallium	1.000	0.547	0.100	0.0005	0.100			
	Zinc	2.000	0.272	0.500	0.0749	2.000			
Thermo							SEVERN TRENT	STL	
ELECTRON CORPORATION							TRENT		
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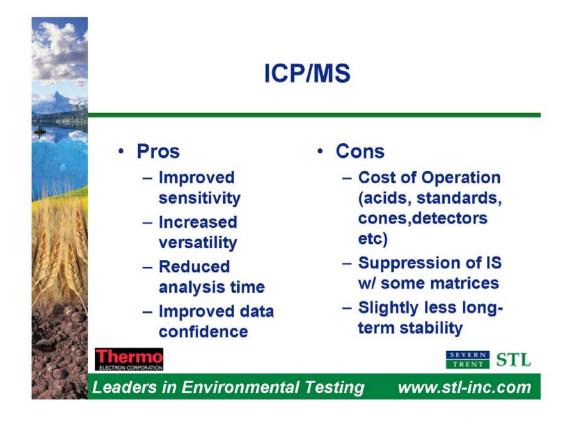


	Linear Dynamic Range Comparison (mg/L)								
		ICPMS 6020	ICP 6010B		ICPMS 6020	ICP 6010B			
China .	CA	1000	600	BA	12.5	10			
Contraction of the second	MG	1000	600	CU	12.5	10			
A CONTRACTOR OFFICE	ĸ	400	400	SB	12.5	10			
A State of the state	AL	250	600	SR	12.5	10			
	FE	225	500	TL	12.5	10			
S 1 1 1 1 3	NA	225	400	ZN	12.5	10			
12134845	SI	150	50	PB	12.5	5			
19.18 1 3 3 10	NI	12.5	100	co	10	100			
	v	12.5	50	CD	10	5			
	SN	12.5	30	в	7.5	30			
1 Bran	TI	12.5	30	BE	7.5	10			
Last and	CR	12.5	20	AS	4.5	10			
Sec. Sec.	MN	12.5	20	SE	4.5	10			
		12.5	20	AG	2.5	TRENT STL			
DAN L	eaders i	n Environ	mental T	estin	g ww	w.stl-inc.com			











### Do Current EPA Methods Compromise the Productivity of Modern Analytical Instrumentation? – Focus on ICP-MS

#### Phil Shaw, Bill Spence

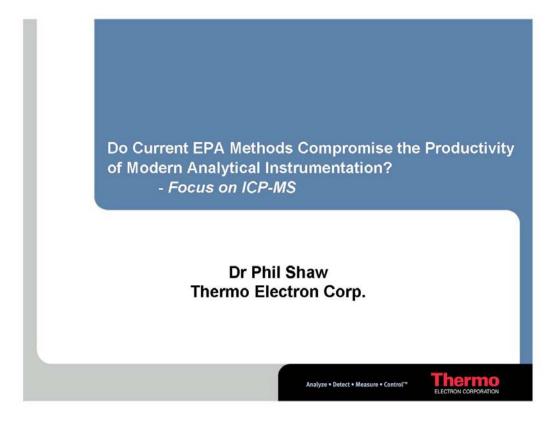
Thermo Electron Corporation, Ion Path, Road Three, Winsford, Cheshire, CW7 3BX, UK. Telephone: +44 1606 548100. Fax: +44 1606 552588. E-mail: <u>phil.shaw@thermo.com</u>

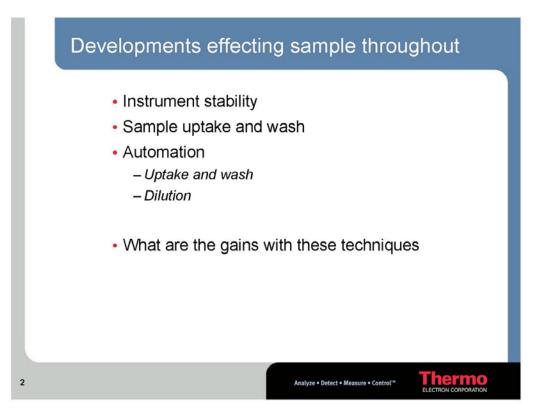
#### ABSTRACT

The EPA methods 200.8, 6020/6020a and ILM05 were either devised several years ago, or evolved from methods that were devised during the infancy of ICP-MS. As such, though many of the analytical parameters to do with data accuracy have changed to product robust measurement, many of the advances made by manufacturers in the productivity of the instrumentation have been ignored. Modern ICP-MS instrumentation is now capable of significantly better long term stability and drift free tolerance to higher matrix levels. They also have much faster sample uptake and washout times with tools for judging automatically if the wash is long enough or not. This can also be linked with automated sample dilutions based on data quality criteria leading to a re-analysis of the data without having to change from the current sample tube. These advances have dramatically reduced the cost of analysis for many laboratories around the world, but for laboratories following prescribed EPA methodologies many of these cost savings have been ignored to date as they have entailed contravening sample sequencing rules established in the Statements of Work.

This presentation will show the typical stabilities possible with modern ICP-MS which reduce the need for high frequency QC checking, even in high matrix samples. It will also describe the automation tools within modern software packages (available from several vendors) which can monitor the sample uptake and washout to improve productivity without compromising the analytical method. Data will be presented to show how these tools are proven as part of the method validation process to ensure analytical viability.

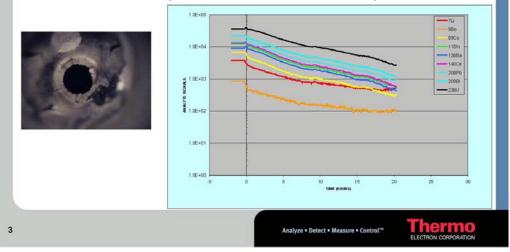
Finally, we will present data showing how modern instrumentation is capable of making intelligent decisions on measured data and then automatically diluting the sample and re-acquiring data without having to re-introduce it to the instrument, using only a fraction of the time it would take to make an off-line dilution of the sample and re-introduce it for re-analysis. Such automated dilution systems are available from several vendors now and offer dramatic improvements in productivity, cost saving and sample turn around times compared to older instrumentation. We will present data on how the dilution systems and the analytical data are validated and the cost savings that can be realised when utilising such devices.

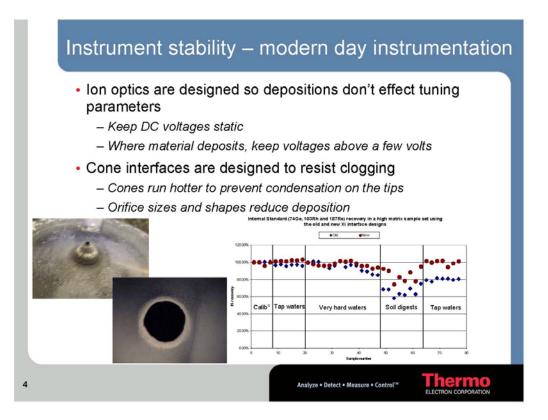




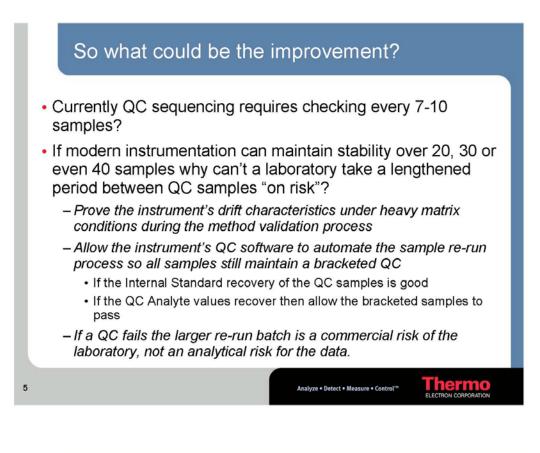
## Instrument Stability – As things were

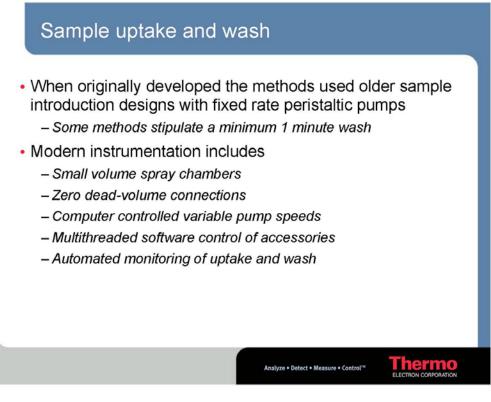
- When first developed the methods would commonly experience drift in even simple matrices.
- When the electronic drift was corrected instruments could still exhibit drift due to matrix deposition on the cones and ion optics.

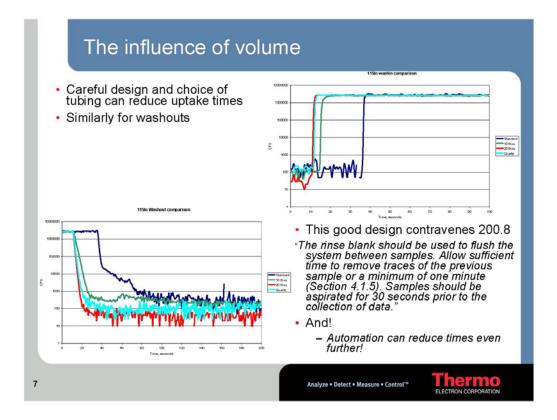


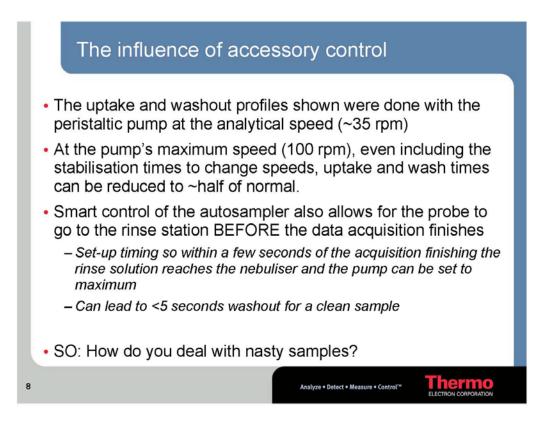


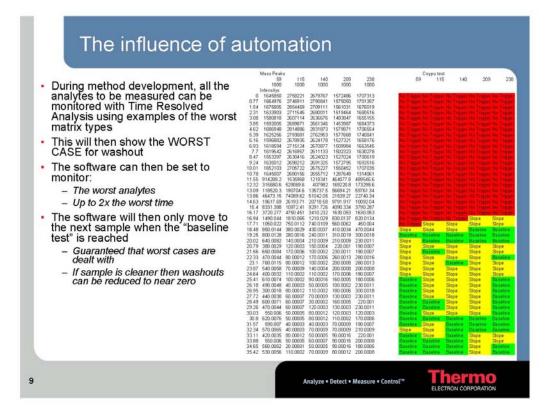
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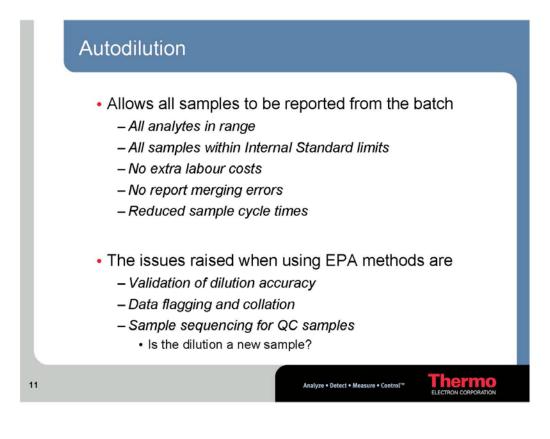


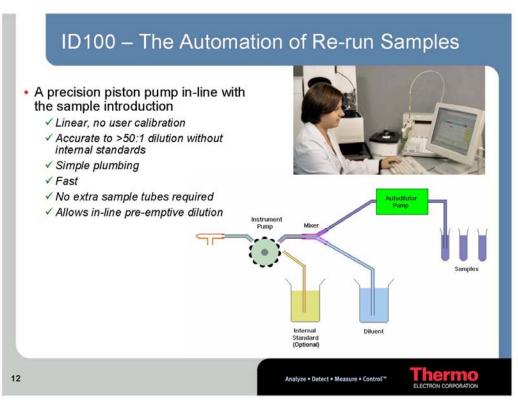


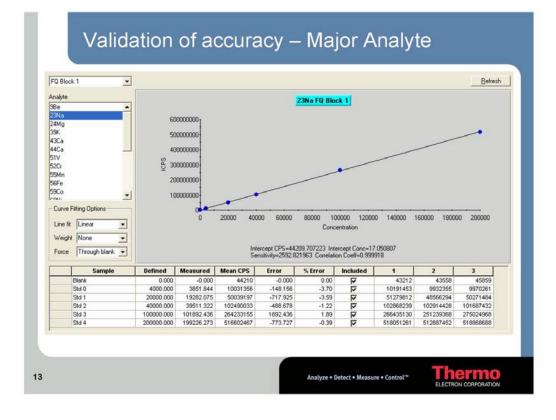


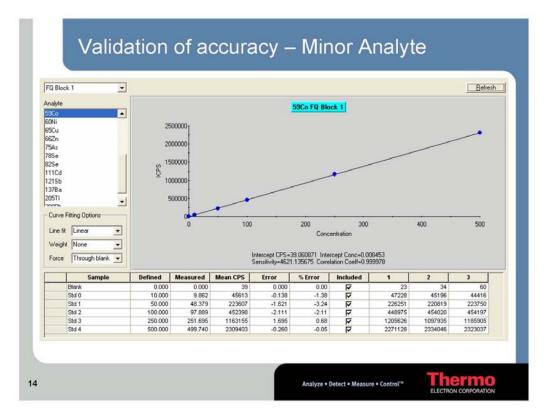


Dilution Dilutions are required in all EPA methods whenever - An analyte measurement is above the analytical range of the instrument Dilute appropriately to bring the analyte within the range and re-measure and report only analytes that were out of range - The internal standards are suppressed (or enhanced) beyond the prescribed limits · Dilute by a fixed ratio and re-measure and report all analytes These operations entail - QC checking data for failure - usually automated by LIMS or reporting software - Re-preparation of samples, rescheduling analysis and some form of report merging · Labour cost is probably ~10 minutes per failed sample · Additionally failures usually add an additional day to the total time to report reducing laboratory cycle times **l hermo** 10 Analyze • Detect • Measure • Control™







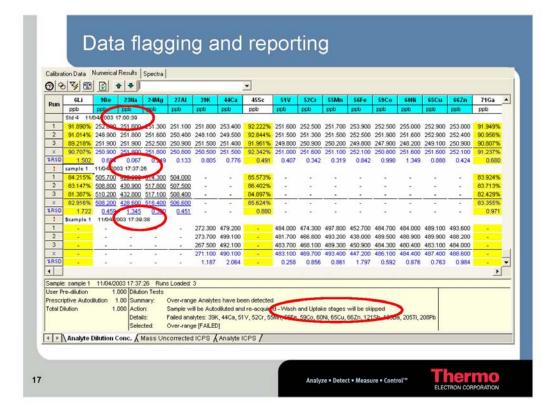


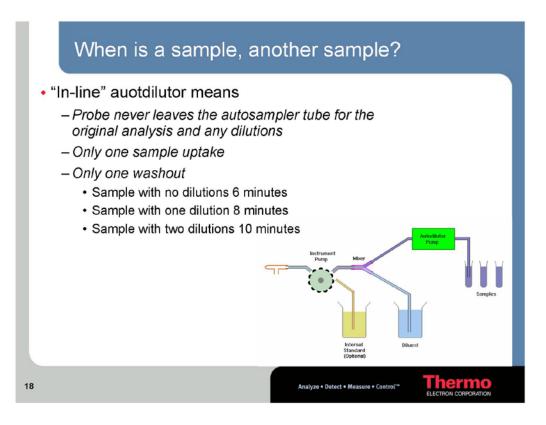
#### NEMC 2005 Proceedings

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IRSD	1.722	0.459	1.345	0.350	0.451			0.880	*S							80 - E	0.971	
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IRSD						1.187	2.064		0.258	0.856	0.861	1.797	0.592	0.876	0.763	0.984		-
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1	91.890%	252.000	251.600	251.300	251.100	251.800	253.400	92.222%	251.600	252.500	251.700	253.900	252.500	255.000	252.900	253.000	91.949%	
2	91.014%	248.900	251.800	251.600	250.400	248.100	249.500	92.844%	251.500	251.300	251.500	252.500	251.900	251.600	252.900	252.400	90.956%	
3	89.218%				250.900	251.500	251.400	91.961%		250.900		249.800	-	248.200	-	250.900	90.807%	
×	90.707%				250,800	250,500	251.500	92.342%	251.000	251,600	251.100	252.100	250.800	251.600	251.600	252.100	91.237%	
TRSD	1.502	0.694	0.067	0.249	0.133	0.805	0.776	0.491	0.407	0.342	0.319	0.842	0.998	1.349	0.888	0.424	0.680	
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			Selec	ted	Over-rang	e [FAILED	1									/		

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#### What are the gains?

#### Longer runs between QC samples

- Every ~20 samples saves 10% instrument time per batch
- Optimising the sample introduction,
  - Fast peri-pump speeds
  - Autosampler probe to wash during data acquisition
  - Using monitored uptake and wash
  - Ignoring the fixed EPA uptake and wash times
  - Saves at least 45 seconds per sample for all samples
    - Can save up to 90 seconds per sample for clean samples in a batch of dirty samples
  - Means at least 75 minutes less instrument time for 100 samples
- Using an "in-line" autodilutor,
  - Use dilutor to make calibration saves approximately 15 minutes instrument time in each analytical calibration (Blank + 4 standards)
  - Assuming 5% sample failures requiring dilution
    - Saves approximately one hour's labour per batch of 100 samples
  - Reduces total batch cycle time by one day

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#### Comparison of Illinois EPA's Low-Level Mercury Sample Collection Procedures with USEPA Method 1669

#### Michael S. Henebry

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E-mail: mike.henebry@epa.state.il.us

#### ABSTRACT

Over the past year (2003-2004), the Illinois Environmental Protection Agency (IEPA) collected samples for analysis of mercury by EPA Method 1631 from about 40 municipal and industrial wastewater facilities and from about equal numbers of stream and lake sites throughout the state. At 28 sites, samples were collected using both the "clean hands/dirty hands" (CH/DH) collection methods developed by USEPA (Method 1669) and by IEPA's routine sample collection procedures. These comparisons were conducted because the procedures detailed in Method 1669 appeared to be problematic for routine sample collection by both the IEPA and by Illinois wastewater discharge permit holders. Procedures in Method 1669 seemed overly complex, and required at least two persons at every sampling event. The IEPA cannot afford to send more than one technician to collect routine effluent and ambient stream samples.

Mercury concentrations at most ambient sites and in most wastewater effluents were less than the Illinois statewide water quality standard (WQS) of 12 ng/L; many were in the range of 1-5 ng/L, although 454 ng/L was observed in one effluent sample. Field blanks were prepared at the time and site of each environmental sample collection, and generally showed no significant contamination. Sample collection method appeared to have no effect on the reported concentrations of mercury in environmental samples. There were no significant differences in low-level mercury concentrations in replicate samples from either wastewater effluents or from ambient stream water sites whether samples were collected by two people using CH/DH, by one person using clean technique or by one person using IEPA's routine sample collection procedures. It was concluded that one of our routine sample collection staff using reasonable care could collect uncontaminated samples for analysis by Method 1631. Detailed sample collection procedures including photos will be presented.

## Comparison of Illinois EPA's Low-Level Mercury Sample Collection Procedures with USEPA Method 1669

Michael S. Henebry Illinois EPA, Springfield, IL E-mail: mike.henebry@epa.state.il.us

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# Acknowledgements

- Jim Miles, Alyson Grady and Stan Lowe assisted in the study design and sample collection.
- Bob Mosher, Tim Kluge, Gregg Good and Bill Ettinger assisted in the study design.
- Many other Illinois EPA field staff participated in sample collection.

## **Outline of Presentation**

- IEPA's collection methods for effluent and surface water samples for low-level mercury analysis
- Results of effluent and surface water samples
- Comparison of results from different collection methods
- Recommendations for sample collection

## Background on Illinois Mercury Studies

- In the past, Illinois rarely found mercury in ambient surface water and effluent samples.
- IEPA had been using ICP-MS, not EPA Method 1631, for analysis of total mercury in water.
- IEPA had concerns about being able to implement the "clean hands/dirty hands" procedures in EPA Method 1669 because we usually send only one person to collect effluent and stream samples.

# **Changes in Mercury Monitoring**

- Many new NPDES permits
  - require Method 1631
  - first time requirement for mercury testing
  - levels 400 times lower than before
  - 12 ng/L (parts per trillion) water quality standard in Illinois

## **Comparison of Analytical Methods**

Method Number	Descriptive Name	Reporting Level	Cost per Sample
EPA 200.8	Metals in Water by ICP/MS	200 ppt (NEMI) 10 ppt (IEPA)	\$18 - \$30 (IEPA Lab)
EPA 1631	Mercury in Water Using CV-AFS	0.2 ppt (NEMI) 0.2 ppt (EnChem)	\$70 - \$100

## **Study Goals**

- To test whether current IEPA sample collection procedures are adequate for collecting uncontaminated samples for total mercury analysis by EPA Method 1631.
- To evaluate the concentrations of total mercury in Illinois effluents and surface waters and compare them to the Illinois human health water quality standard of 12 ppt

## **Site Selection - Effluents**

- Some wastewater facilities were selected for their potential to have concentrations of total Hg exceeding the water quality standard of 12 ng/L
- Other "background" facilities were not expected to have elevated concentrations of Hg in their effluents.
- Effluent samples were collected from 33 facilities distributed throughout the state.

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## Site Selection – Surface Waters

- We selected stream and lake sites that were part of their respective ambient water quality monitoring core networks that have been sampled over a number of years and are projected to be sampled in the future.
- We collected 52 samples from core stream (AWQMN) stations.
- We collected 32 samples from core lakes and from lakes with fish advisories for mercury.



The analytical laboratory provided sampling kits consisting of:

- Pre-cleaned 500-mL borosilicate glass bottles that were double zip-lock bagged
- Mercury free deionized water for preparation of field blanks at each site
- Bagged, talc free vinyl gloves

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## **IEPA Clean Hands/Dirty Hands**

- The sample collection crew wore clean, unlined nylon windbreaker jackets and vinyl gloves.
- Clean Hands handled the sample bottles and the inner zip-lock bags provided by the analytical laboratory and clean parts of the sample collection apparatus.
- Dirty Hands opened coolers, handled the outer zip-lock bags, and manipulated all potentially "dirty" parts of the sample collection apparatus.

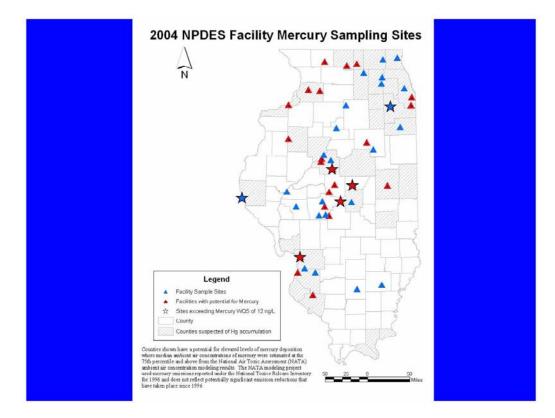
#### **Materials and Methods – Effluents**

- At least one sample of each of the first 33 facility effluents was collected by two persons using IEPA's version of USEPA's CH/DH technique (EPA Method 1669).
- One person using IEPA's "routine" effluent sample collection procedure collected a second (duplicate) effluent sample during the first 19 effluent sampling events for comparison to the results from CH/DH samples.

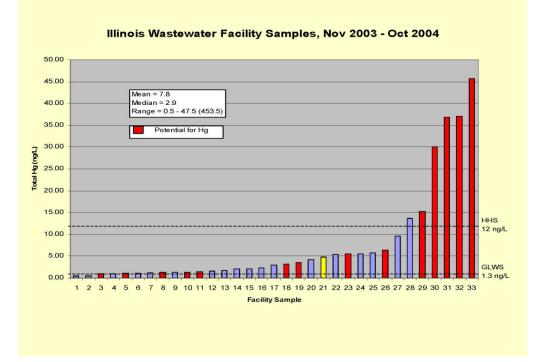
# IEPA "Routine" Effluent Sample Collection Procedures

One person:

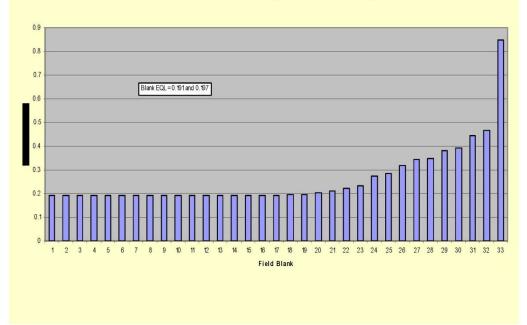
- Performed all the tasks of both Clean Hands and Dirty Hands
- Used relatively clean sample collection apparatus
- Wore a clean, nylon windbreaker jacket and vinyl gloves
- Was aware of and was careful to avoid possible sources of sample contamination

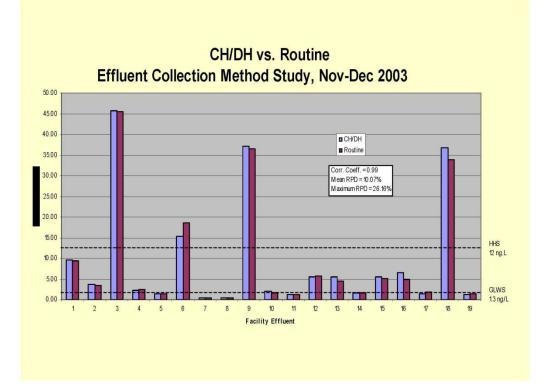


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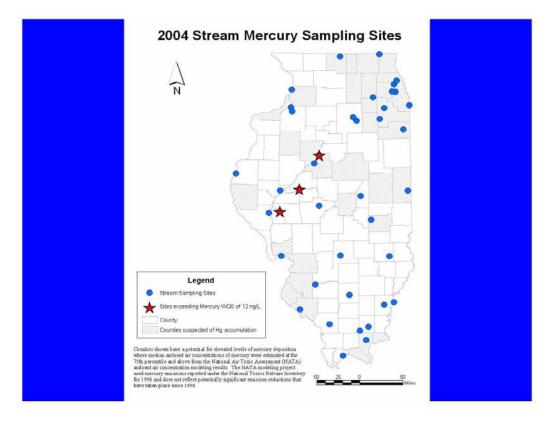
#### Field Blanks for Facility Effluent Samples

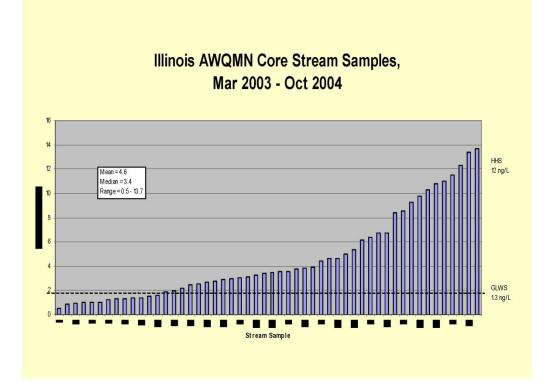


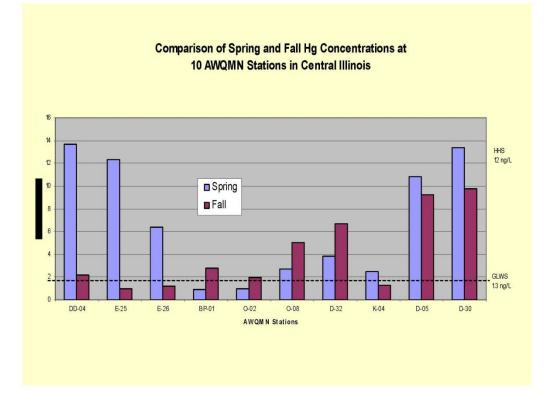


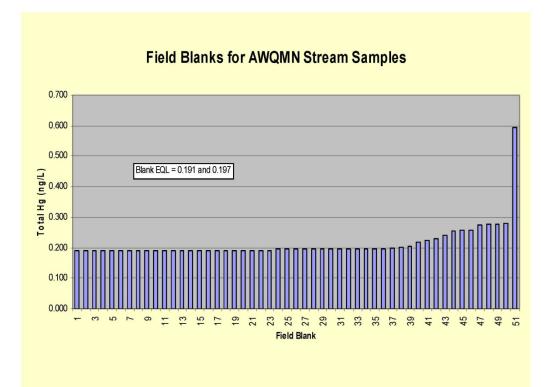
## **Materials and Methods – Streams**

- All AWQMN samples were collected by at least two persons using IEPA's version of USEPA's CH/DH procedure.
- A non-metallic, weighted-bottle sampler was used to immerse the 500-mL sample bottle directly into the top 1-foot of the center of stream flow.



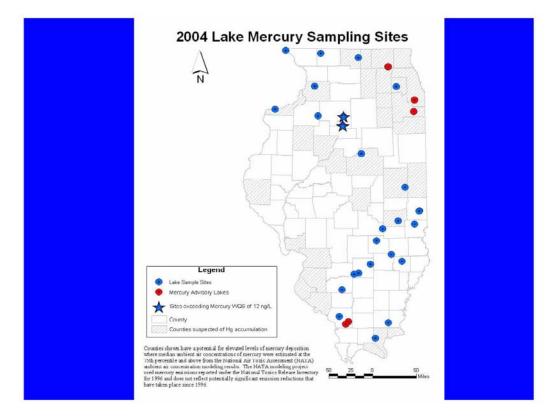




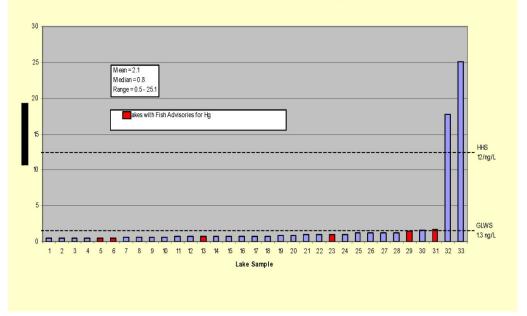


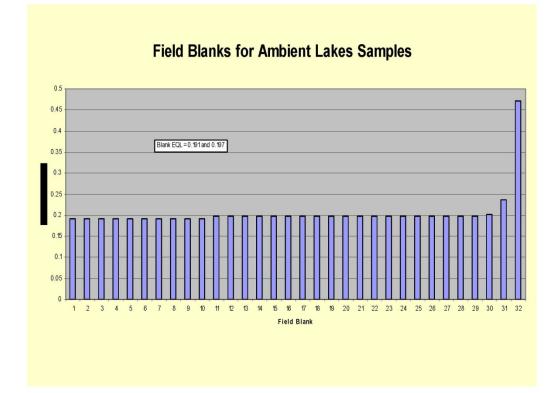
## **Materials and Methods - Lakes**

- Two persons using the IEPA's CH/DH procedures collected all of the ambient lakes samples.
- Clean Hands collected samples from the top 1-foot of lake water by reaching over the side of the boat and immersing the Hg sample bottle until full.



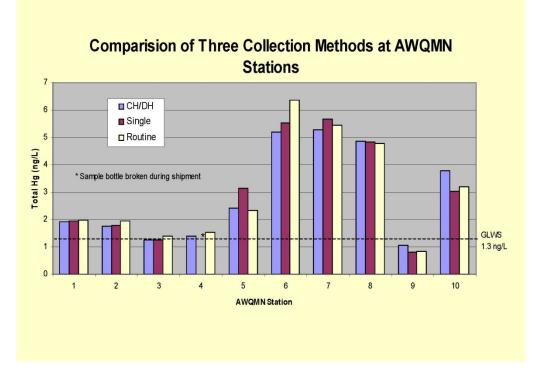
Illinois Ambient Lakes Samples, Aug - Oct 2004

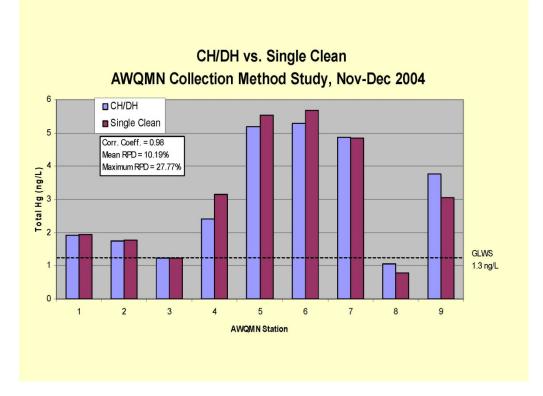


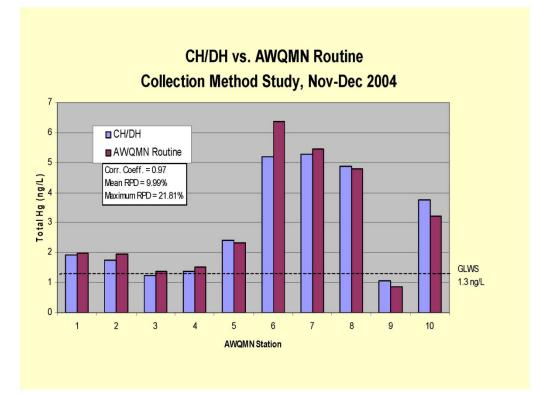


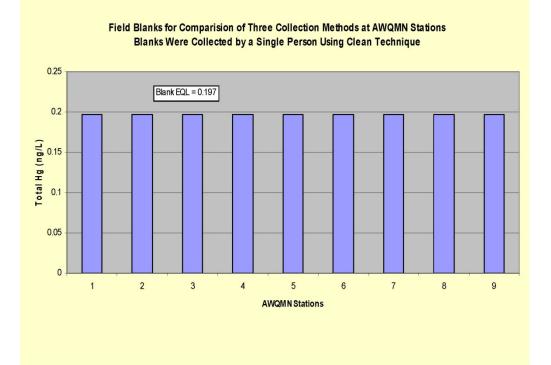
## Comparison of of Stream Sample Collection Methods

- Three sample collection methods were compared at 10 stream stations in central Illinois. Replicate samples at these stations were collected by:
- Two persons using IEPA's CH/DH procedure
- By one person collecting samples using clean technique
- By the usual field staff person collecting the way that AWQMN samples are routinely collected.









## **Summary and Conclusions**

- Our results suggest strict adherence to EPA 1669 is not necessary for the collection of samples for low-level mercury analysis (EPA 1631).
- It appears that samples for low-level mercury analysis can be collected by one of our usual sample collection staff using our routine collection methods without introducing significant levels of mercury contamination.

## **Summary and Conclusions (Cont.)**

- Concentrations of total mercury in most samples of Illinois' facility effluents and surface waters did not exceed the human health standard of 12 ng/L.
- Concentrations of mercury in some effluent and stream samples were greater after rainfall and subsequent runoff events.

## Why Did Collection Method Make No Difference in Our Results?

- We always collected the environmental sample directly into the bottle provided by the laboratory.
- Our sample collection staff was trained to be aware of and to avoid possible sources of sample contamination.

## **Recommendations for Low-Level Mercury Sample Collection**

- 1) Make use of the sample collection kit provided by your laboratory.
- 2) If possible, collect samples directly into the bottles provided by your laboratory (avoid using compositors, plastic bottles or your own "clean" glass bottles).
- 3) Keep bottles uncapped for as short a time as possible.
- 4) Avoid touching the rim of the sample bottle or the inner surface of the bottle cap.

## **Recommendations (Cont.)**

- 5) Be aware of and avoid possible sources of airborne contamination.
- 6) Periodically prepare field blanks with environmental sample collections.
- Duplicate samples can be useful for evaluating the reliability of your sample collection methods.

# **Session 5**

# Advances in Electronic Deliverables and Information Management

#### SEDD – An Overview and Status Report

#### Anand R. Mudambi

US EPA Analytical Services Branch, 1200 Pennsylvania Ave NW, Mail Code 5102G, Washington, DC 20460 Author's e-mail: <u>mudambi.anand@epa.gov</u>: Phone: 703-603-8796

#### ABSTRACT

SEDD (Staged Electronic Data Deliverable) is a program neutral format for the delivery of analytical data. It supports multiple users' needs depending on the level of analytical requirements. The main advantage of SEDD is that once implemented, laboratories do not have to completely overhaul their Electronic Data Deliverable (EDD) generating systems as data requester needs become more complex, but can simply add additional elements to their current system. Using SEDD as the basis of electronic delivery of analytical data will decrease costs by reducing number of EDDs laboratories currently have to support and ease data exchange between various programs and agencies.

At the present time different levels or Stages for SEDD have been developed based on the complexity of data reporting requirements needed.

SEDD files are delivered as XML (eXtensible Markup Language) files. Sponsored by the World Wide Web Consortium (W3C), XML is license free, platform independent, final recommended standard which encapsulates structured data in text files. It is well supported by freely available third party tools.

Delivery of analytical data in the SEDD format is now a requirement in the U.S. Army Corps of Engineers' Formerly Used Defense Sites (FUDS) program and for certain U.S. Environmental Protection Agency contracts including the new Contract Laboratory Program's (CLP) Organic Statement of Work SOM 1.0. Interagency efforts are underway with the U.S. Air Force, U.S. Navy, and the Department of Energy to promote the use of SEDD.

Laboratories and LIMS vendors are gearing up to provide SEDD files as required in the upcoming contracts. Laboratories have already submitted compliant SEDD files as part of the new CLP Organic Solicitation. U.S. EPA is providing tools to assist laboratories in both creating and checking SEDD files prior to submission. Private parties are also evaluating SEDD as a basis for uniform delivery of analytical data.

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#### STAGED ELECTRONIC DATA DELIVERABLE (SEDD) - AN OVERVIEW AND STATUS REPORT

Dr. Anand R. Mudambi Environmental Protection Agency (USEPA)





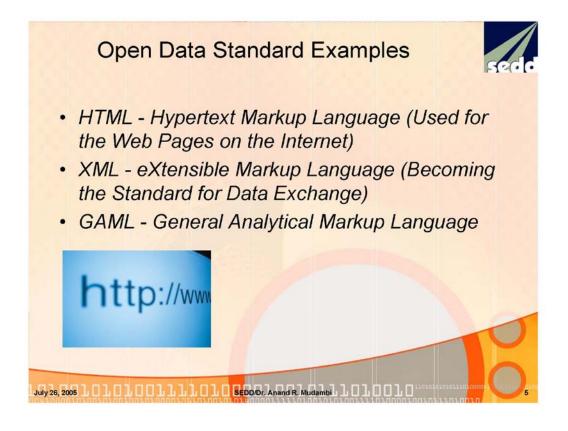
#### Problems with Current Electronic Data

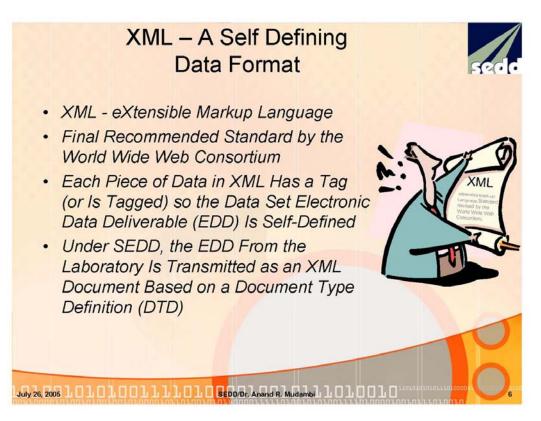


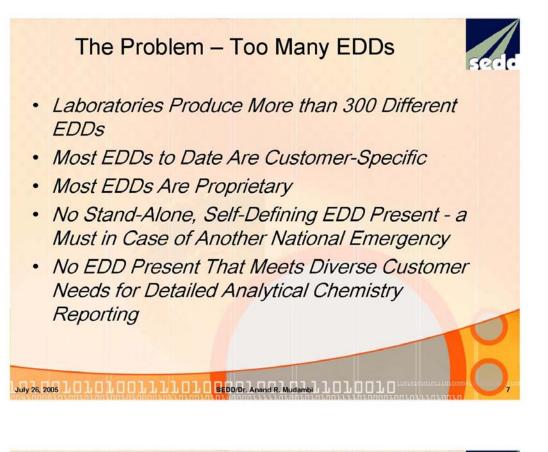
- Most data is received in proprietary formats (e.g., documents in Word or WordPerfect, spreadsheets in Excel or Lotus).
- The business model for proprietary formats is PLANNED OBSOLESCENCE with little or no backward compatibility.
- Thus, most data generated in these formats today will not be accessible 5-10 years from now.

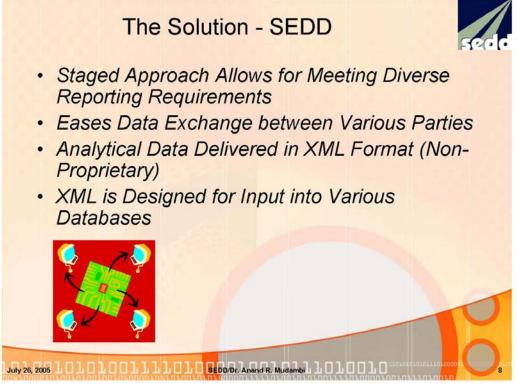
July 26, 2005 J. O. L. O. L. J. L. L. D. L. C. SEDD/Dr. Anand R. Mudambi J. C. L. O. L. D.

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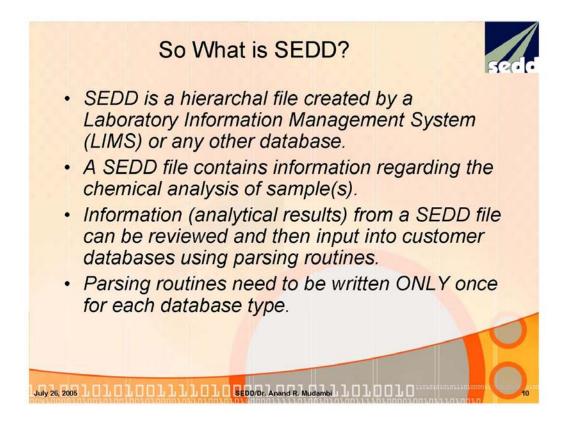




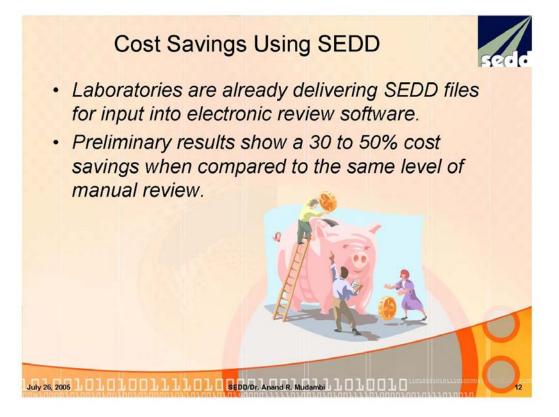












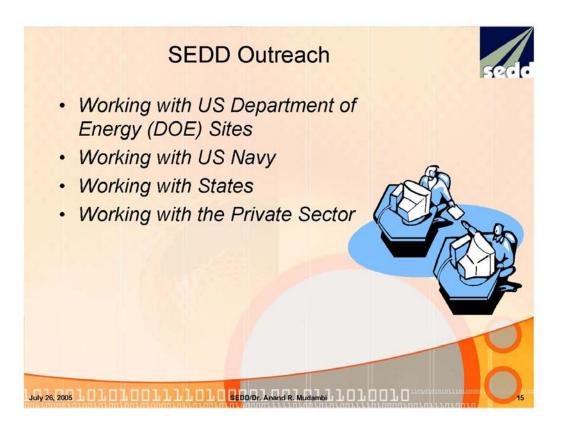
#### **SEDD Status**



- Working on Pilots with Laboratories since 2002
- Laboratories (Including At Least One Major Network) and Two LIMS Vendors Are Already Delivering Compliant SEDD Stage 2 Files
- SEDD Files Being Input and Checked by Automated Data Review Software







### What's New with SEDD!



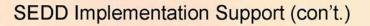
- SEDD is being considered as an American Society for Testing and Materials (ASTM) standard for LIMS-to-LIMS data transfer.
- SEDD is being embraced by the industry as it becomes a contract requirement.

July 26, 2005 L O L O L O L L L L O L O SEDD/Dr. Anand R. Mudambi L O L O L O L O

SEDD Stage 4 is being developed with input from instrument manufacturers.

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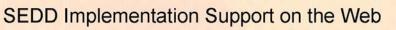


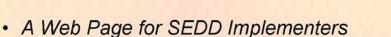


- For Federal Agencies, contract language for implementing SEDD is available.
- For all parties interested in implementing SEDD, two courses are being offered at NEMC on Thurs July 28
  - Morning Technical Implementation of SEDD

July 26, 2005 J. O. L. O. L. J. L. L. D. L. O. SEDD/Dr. Anand R. Mudambi J. O. L. O. D. O. L. O. L. O. D. O. L. O. L. O. D. O.

 Afternoon – SEDD Files: Automating the Parsing and Review of Analytical Data





- A List of Laboratories and Vendors Who Support SEDD (YOUR NAME HERE?)
- A SEDD Q & A Web Page
- A 10 Step Process for SEDD/ADR Implementation

July 26, 2005 L O L O L O L L L L O L O SEDD/Dr. Anand R. Mudambia L O L O L O L O



#### The Technical Components of SEDD Stage 3 Files

#### Joseph Solsky

U.S. Army Corps of Engineers (CENWO-HX-C), 12565 W Center Rd, Omaha, NE 68144 E-Mail: <u>Joseph.F.Solsky@usace.army.mil</u>; Phone: 402-697-2573

#### ABSTRACT

No one single electronic data deliverable format would be able to meet the needs of the multiple data users due to the various levels of data complexity and reporting as required by those users. As a consequence, SEDD accommodates the reporting of data in 'Stages', with each stage building on the next using XML technology. Currently, three stages or unique electronic data deliverable formats have been defined for SEDD. Stage 1 contains the minimum number of analytical data elements to report 'Results Only' data to the end user. Stage 2 builds on Stage 1 and adds method (Stage 2a) and instrument (Stage 2b) Quality Control (QC) data. Stage 3 builds on Stage 2 and adds additional measurement data to allow for the independent recalculation of the reported results.

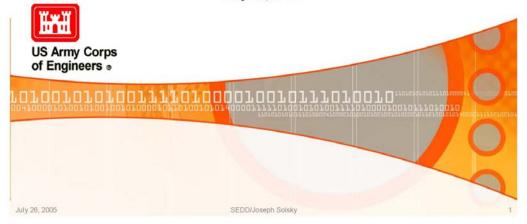
SEDD delivers data in the form of an XML document. A common structure has been developed that will allow for the reporting of all types of data. SEDD allows for the complete linking of all samples to their associated QC samples, the complete linking of all samples to their associated continuing and initial calibration data, and the complete linking of all reported results to the specific analysis that was used to derive that specific result. It is these linkages that allow for the complete and independent recalculation of all reported results within a SEDD Stage 3 file. This independent recalculation is performed by starting with an integrated area count for a typical organic chromatographic method or by starting with a background corrected spectral intensity measurement for a typical inorganic spectroscopic method.

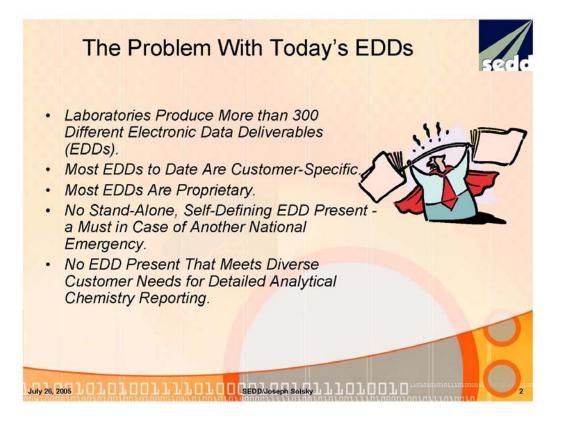
A SEDD Stage 3 file captures and reports all of the data needed to independently recalculate all final results by capturing this data in the manner in which in was generated. By capturing the data in this manner, the data can be reported and reviewed against the requirements of many different programs. For initial calibrations, average calibration/response factors, linear regressions, quadratic regressions, and other techniques can all be used. These calibration strategies can be applied on a per peak basis or applied when peaks are summed together. Various weighting factors can also be used when regressions are performed. In addition, either 'external standard' or 'internal standard' procedures can be used for any analyte using any method. This same type of flexibility that is used for the reporting of initial calibrations is used throughout the sample preparation and analysis process.

(2) I prefer to give an oral presentation.

#### A TECHNICAL OVERVIEW OF STAGED ELECTRONIC DATA DELIVERABLE (SEDD)

Joseph Solsky US Army Corps of Engineers (USACE) July 26, 2005







# XML – A Self Defining Data Format

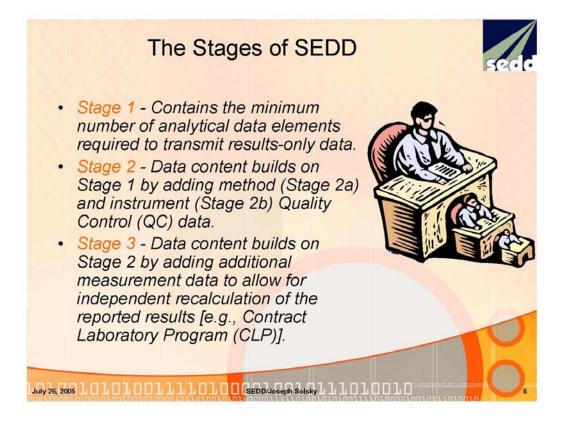


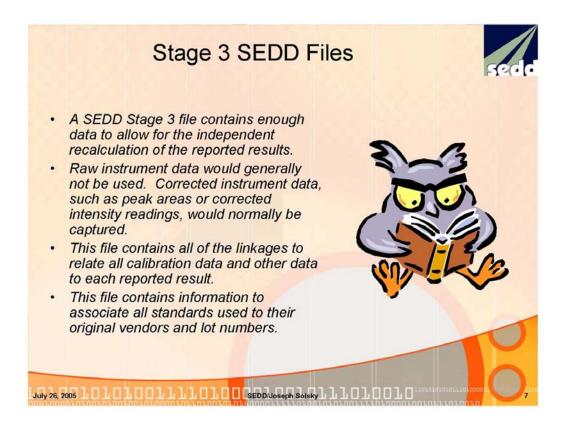
- XML eXtensible Mark-up Language
- Final Recommended Standard by the World Wide Web Consortium
- Each Piece of Data in XML Has a Tag (or Is Tagged) so the Data Set (EDD) is Self-Defined
- Under SEDD, the EDD from the Laboratory Is Transmitted as an XML Document Based on a DTD or Schema

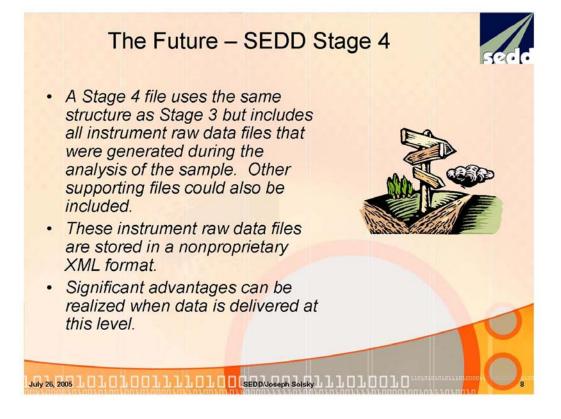
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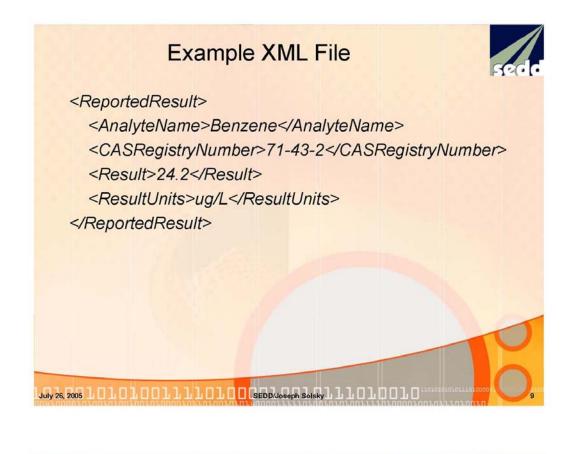


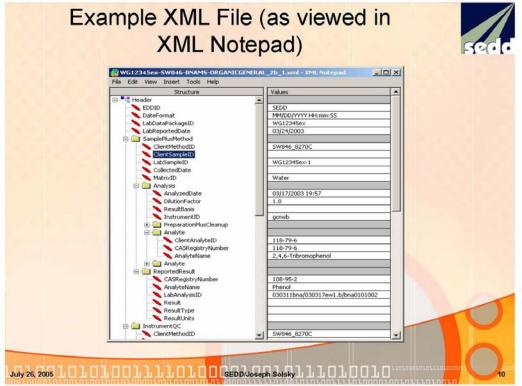


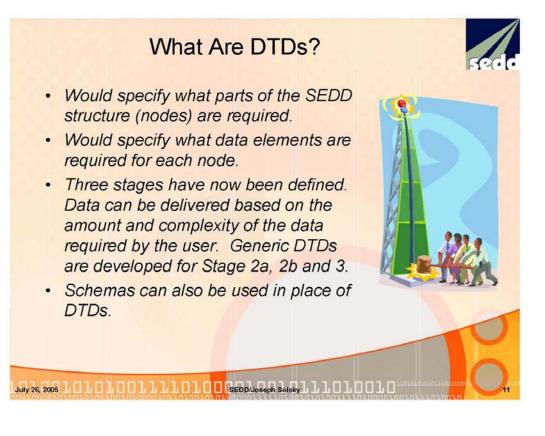


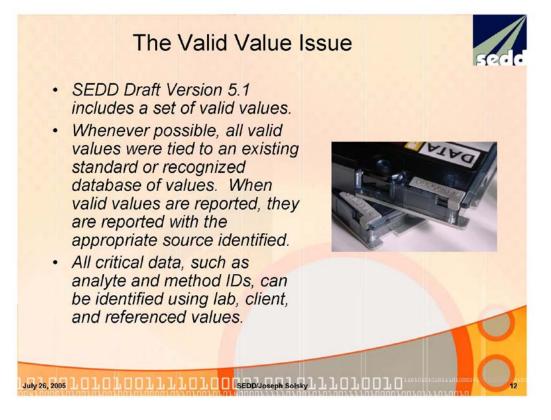


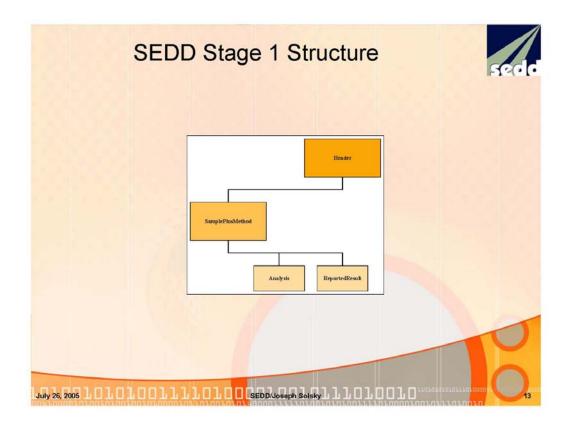


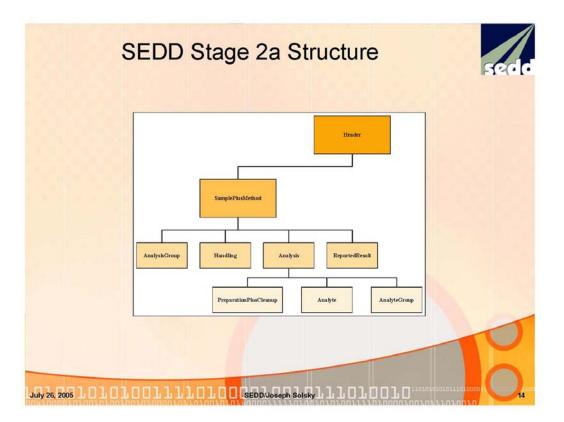


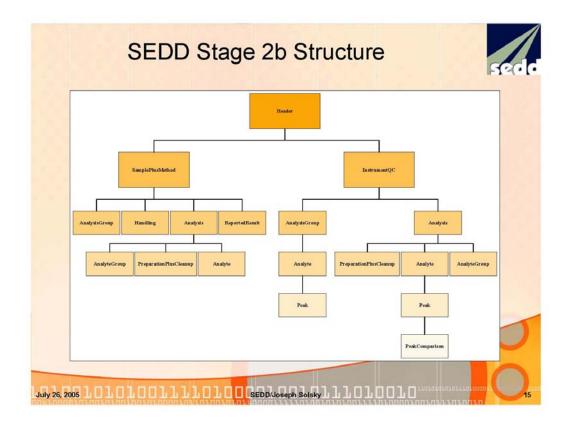


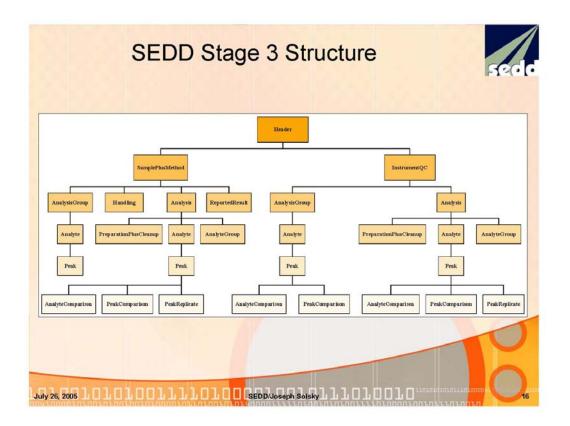














## Automated Generation and Validation of Staged Electronic Data Deliverable in a Commercial Laboratory

Jakub Rehacek, Ph.D. PEL Laboratories, Inc., 4420 Pendola Pt. Rd., Tampa, FL 33619 email: lof@pelab.com, phone: 813-247-2805

#### ABSTRACT

SEDD is an emerging electronic data deliverable offering program-neutral, non-proprietary format for electronic data exchange. PEL Laboratories, Inc., has participated in the Staged Electronic Data Deliverable (SEDD) Pilot study under GEITA T.O. 041, and has successfully delivered all stages of the SEDD.

PEL has developed an in-house data management system (PEL DMS) that automates all aspects of our analytical services. The projects are tracked from the bid stage through bottle kit assembly, sample receiving, prep, and analytical stages to final data reporting in hardcopy and EDD. The system interfaces directly with email and PEL's data driven web site in *real time*. All reviews and releases are done digitally, narratives are automatically generated from the data and analyst feedback, most logbooks and lab notebooks are electronic. A complete Level-IV CLP package is automatically assembled and generated by the PEL DMS. There are several hundred QC and data validation checks performed on each test/method at each review/release level.

Both Windows and Internet-based front-end user interfaces were developed to facilitate automated and streamlined EDD deliverables. The graphical user interface (GUI) allows Project Managers to generate **SEDD** as one of the EDD "flavors" we routinely provide to our clients. The SEDD is generated automatically in conjunction with hardcopy reports <u>directly</u> from our LIMS. Our Lab currently provides over 100 custom EDD formats. The SEDD would greatly simplify our EDD reporting as a single electronic deliverable.

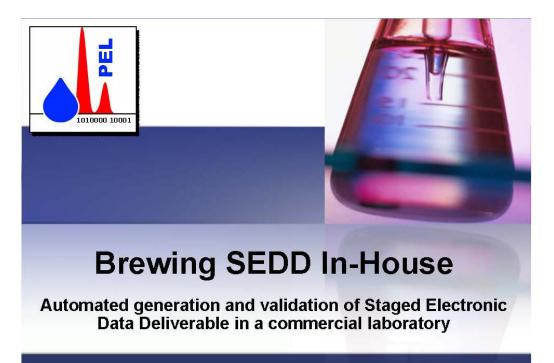
Our Data System has the capability to generate both SEDD and customer specified EDD at the same time in order to ease the transition into the SEDD based deliverables. All necessary information comes from the same data store so we can guarantee that the SEDD, custom EDD, and hardcopy will all have identical values. We can also generate SEDD deliverables for projects that have already been processed through the Lab and were reported in a different EDD format.

#### SEDD Data Review and Validation Tool

One of the modules in our Client Web Portal is an online data review and validation tool capable of reviewing and validating SEDD deliverables. Our clients can upload the SEDD XML file via custom web page and match it with Project specific QC criteria (Project Profile). PEL Data Validation/Review tool significantly reduces the time and cost for a validator to review results delivered in SEDD format. This tool makes it possible to generate reports quickly via our website, including a Summary of all *QC exceptions* with their associated prep and analytical runs, an *Executive Summary* report showing all values detected above the Method Detection Limit, or above custom Contaminant Levels. A comprehensive *Laboratory Review Checklist* (LRC) that quickly summarizes all variances is also available. Analytical runs are cross-

referenced to prep batches and to calibrations. For the SEDD Stage 3, clients have option of independent recalculation of reported results from raw data. The reported results can be reviewed from many angles; all pertinent information is just few clicks away.

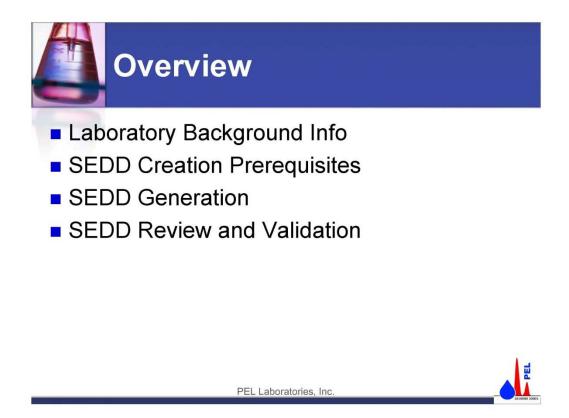
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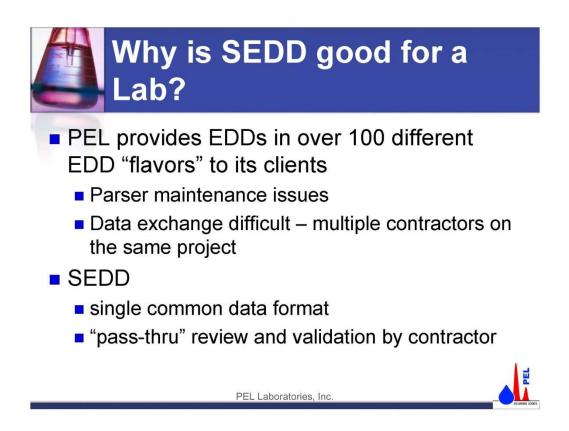


Jakub Rehacek, Ph.D., PEL Laboratories, Inc.

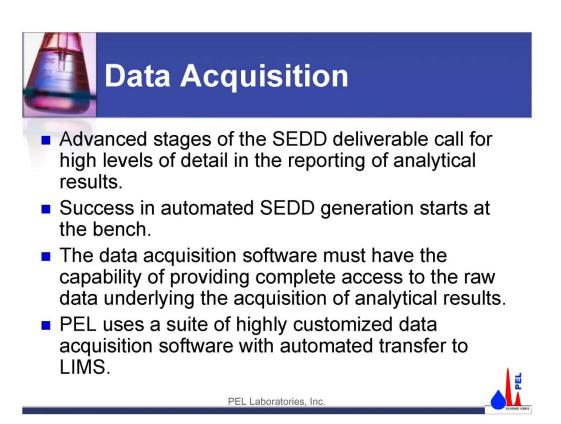


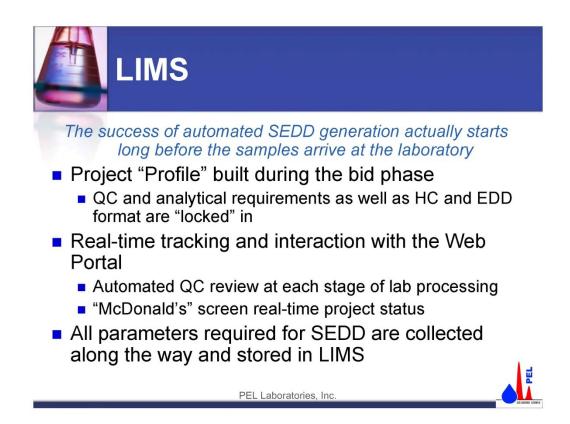
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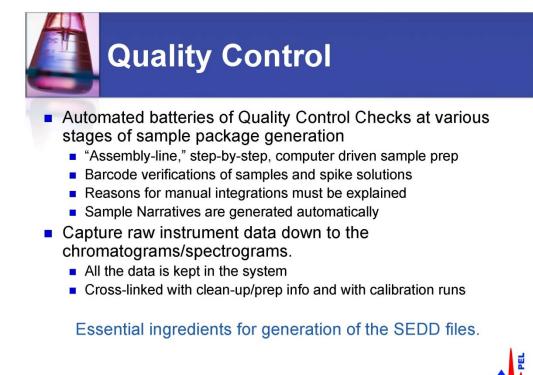


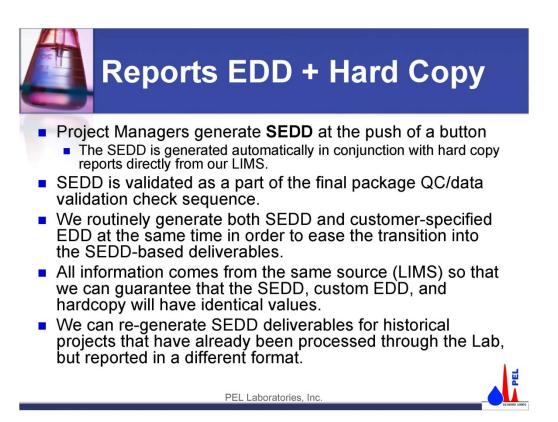


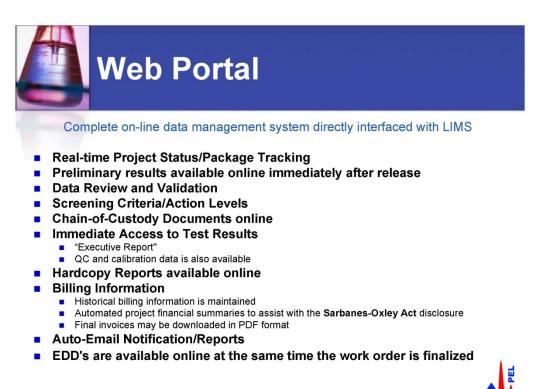














# Automated Generation of SEDD

Available Tools

- SEDD Tool EPA's data converter
- Commercial LIMS
- Off-the-shelf tools (MS SQL Server, .NET)



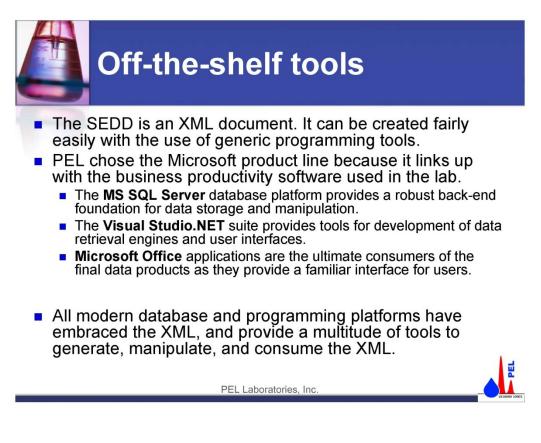


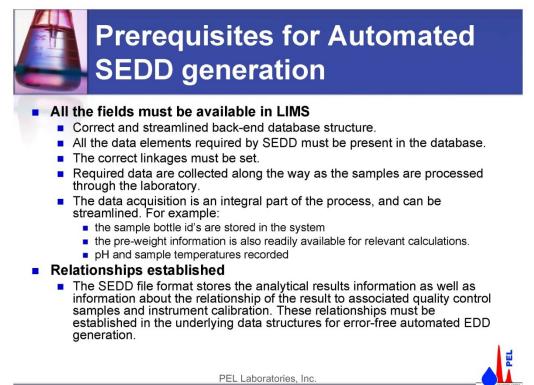
- The SEDD Tool's primary task is to convert local database data into an Extensible Markup Language (XML)-compliant file for delivery to other remote data systems.
- The SEDD Tool addresses the U.S. Environmental Protection Agency's need to receive analytical data electronically from its contracting laboratories.

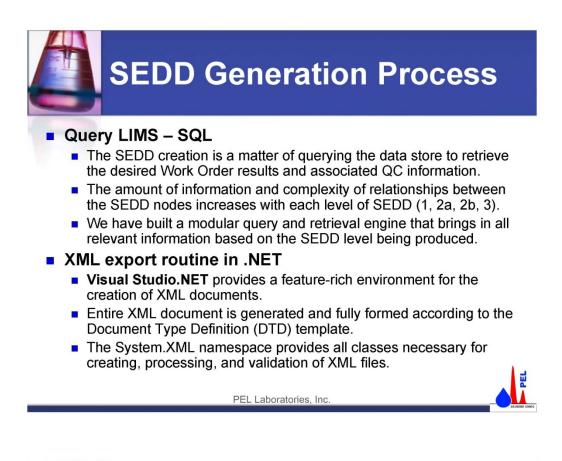


- Several commercial LIMS software vendors have incorporated the SEDD generation into their products.
- Laboratories have to work with the vendor to ensure that their SEDD generators are kept up to date with the latest changes in the SEDD format.







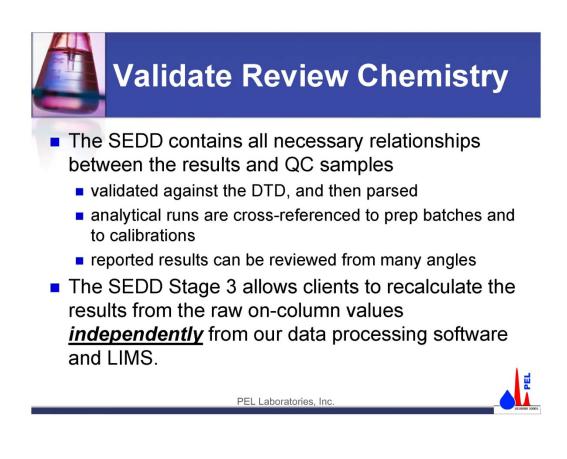


# Validation/Review Tool

PEL's Data Validation/Review tool is an on-line service that assists a project chemist or independent validator with review of our chemistry.

- Data review/Validation
- QC Reports







 Our Review tool makes it possible to generate reports quickly via our website. The reports include an Executive Summary report showing all values detected above the Method Detection Limit, and a comprehensive Laboratory Review Checklist (LRC) that quickly summarizes any exceptions. A detailed QC flagging report can also be created to assist with validation flag applications in our clients' data systems.

 Our system can also export EDDs in multiple formats to facilitate transition from legacy data systems. Clients can upload the SEDD data file and export in ERPMS, ERIS, or any other format that PEL currently supports. PEL Laboratories, Inc., is currently working with other companies to facilitate automated communication in order to provide push-button validation of laboratory results.



Contact info:

Jakub Rehacek, Ph.D. VP Information Systems PEL Laboratories, Inc. *jrehacek@pelab.com* 813-247-2805 <u>www.pelab.com</u>



## Creating SEDD Stage 3 Deliverables: A LIMS Vendor's Perspective

#### **Buddy Wilson**

Promium 22522 29<sup>th</sup> Dr SE, Suite 205 Bothell, WA 98021 <u>Primary Author E-Mail</u>: buddy@promium.com Phone: 425.286.9200

#### ABSTRACT

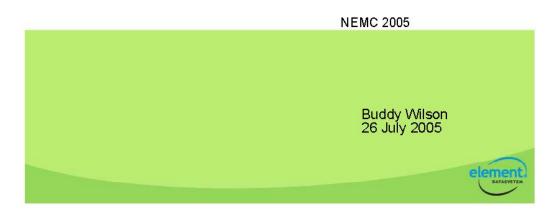
The SEDD (Staged Electronic Data Deliverable) specification provides a common structure and data element dictionary to report a wide variety of data to multiple customers. The SEDD specification is program-neutral and allows for reporting of data in a single deliverable format that contains results ranging from simple sample concentrations (Stage 1) all the way to a CLP (Contract Laboratory Program) type data package (Stage 3). Because of the potential and flexibility of the SEDD format, Promium has actively worked with the EPA and Army Corps of Engineers to support the use of the format by laboratories with Promium's Laboratory Information Management (LIM) system when they perform testing for projects that must be reported to those agencies.

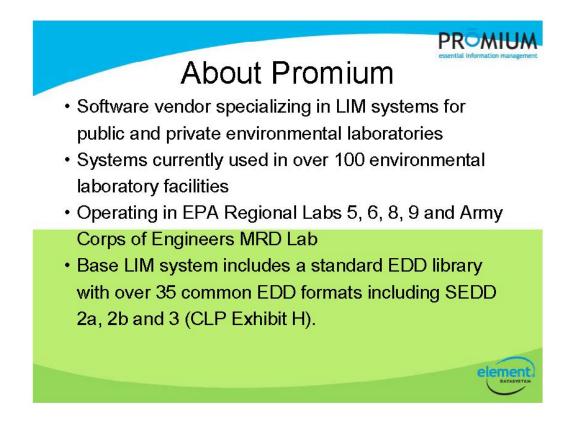
Promium already supports the SEDD format by providing native generation capability for Stage 2a and 2b deliverables within Element DataSystem, its out-of-the-box LIMS for Environmental Testing Laboratories. During 2004, the SEDD format gained further acceptance among federal agencies and was written into the SOM 1.0 Organics CLP Contract. Keeping with its commitment to support this well-designed deliverable, Promium included the most complex form of the SEDD, Stage 3, within its LIMS application during the last half of 2004 and assisted one of its client laboratories in generation and the new CLP Forms and Exhibit H SEDD deliverable for the Performance Evaluation sample submission requirement of the new CLP contract. The process of writing support for this stage of the deliverable presented a different set of issues than previous development efforts. In particular, the company found itself re-designing some aspects of its existing LIMS application to better support the structure and data elements required for a Stage 3 SEDD. The end result was a more comprehensive LIM system for Environmental Testing Labs with a native ability to generate SEDD Stage 3 deliverables for CLP laboratories or for non-CLP laboratories wishing to provide electronic deliverables in this very comprehensive format.

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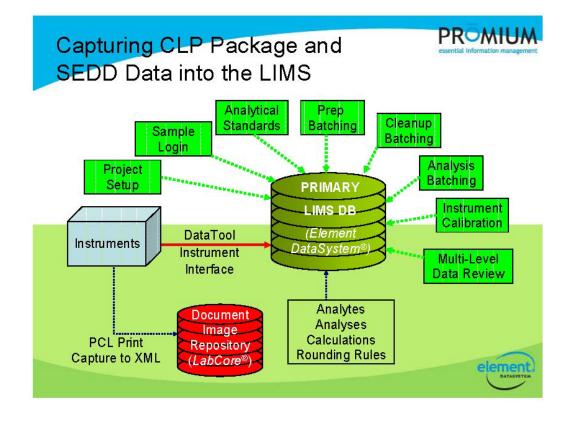


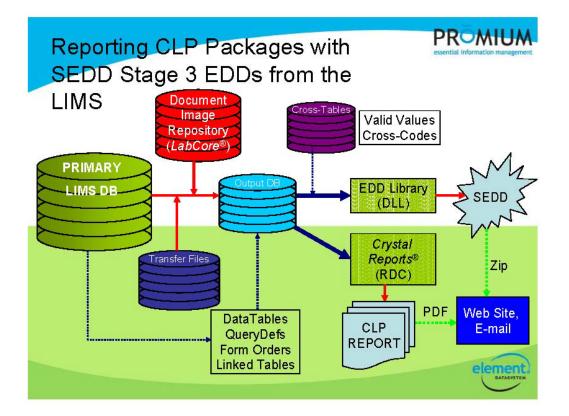
# Creating SEDD Stage 3 Deliverables: A LIMS Vendor's Perspective













- On-line SEDD checking tool was very helpful
- Not something you would want to do manually ...

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VOA Trace	85394	24760 (29.0%)	3.70 MB	
VOA Low	170140	49149 (28.9%)	7.37 MB	
BNAs	151648	44919 (29.6%)	6.55 MB	
Pesticides	96585	28693 (30.0%)	4.19 MB	
Aroclors	41693	12755 (30.6%)	1.81 MB	
Fraction	Data Elements	Data Elements Filled	File Size	

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 Phone: 425-286-9200
 EMail: bwilson@promium.com



### Automating the EDD Designer, Checker, and Generator Process

#### Paul Banfer

Vice President / Product Technology EISC *EISC*, 6767 W. Tropicana Ave, Las Vegas, NV 89103 Primary Author's E-Mail: eisc@eisc.net; Phone: 702-248-1021

#### ABSTRACT

The EDD Designer, Checker, and Generator process provides a unique approach to seamlessly passing analytical data from business to business. This concept is extremely productive, flexible, and manageable while maintaining quality and integrity.

Today's laboratory is becoming more difficult to manage due to the increasing diversity of client deliverables. These diverse deliverables affect a lab's quality, integrity, growth, and productivity...all of which affect revenue.

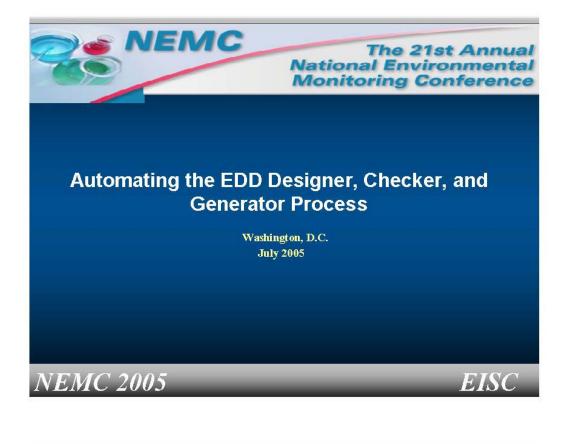
All in all, the most significant result of the industries diverse deliverables has pushed the focus to data deliverables rather than analytical data.

To combat this industry shift many laboratories, engineering firms, and agencies have focused their attention to automate the EDD process through the EDD Designer, Checker, and Generator Process.

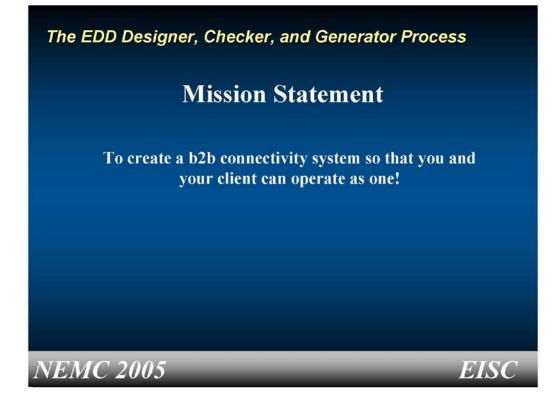
This presentation will focus on example automation models consisting of:

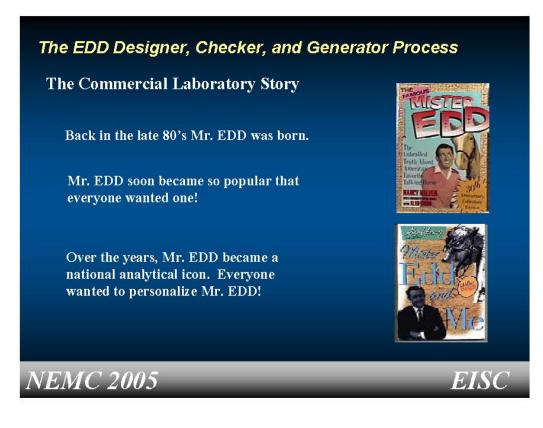
- 1) Commercial Laboratories that have many different EDD formats to produce
- 2) Analytical Laboratories for Water Utilities meeting the 50 state requirements
- 3) Automation of Commercial Laboratory to Data Validation
- 4) An Engineering Firm or Agency model for Data Validation
- 5) XML, SEDD, Superfund, and Stage 4
- 6) XML and the States
- 7) Chaining information sources to present the combined results to the decision maker
- 8) Chaining instrument data sources to present combined data to the decision maker
- (2) I prefer to give an oral presentation.

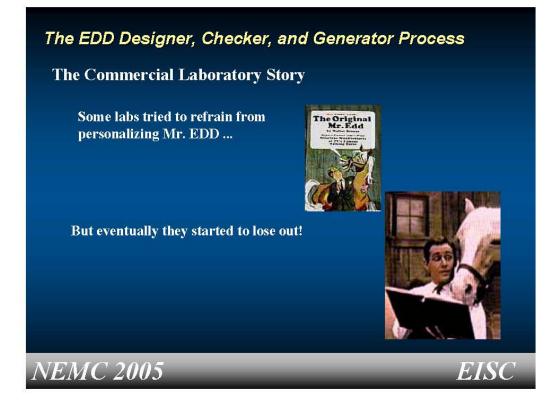
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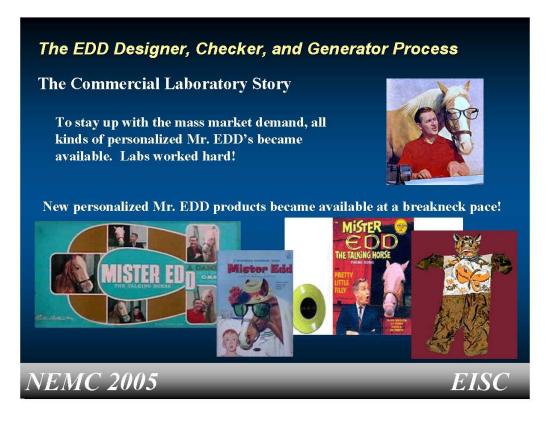


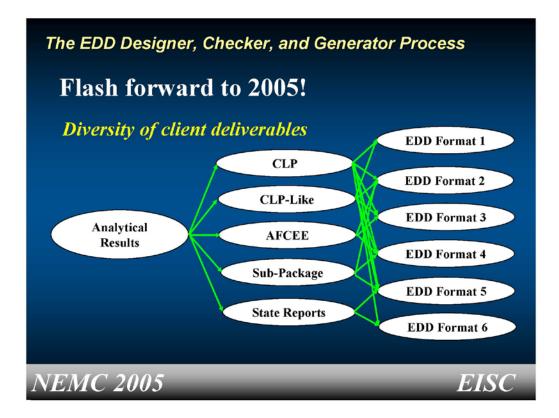
# Overview Management challenge Increased diversity in client deliverables offers management challenges for today's lab > Data Quality > Data Integrity > Growth > Productivity > All effect...Revenue! Meeting the challenge Industry focus on EDD process automation > The EDD Designer, Checker and Generator Challenge Met > EISC's experience and approach across variety of analytical industries **NEMC 2005** EISC

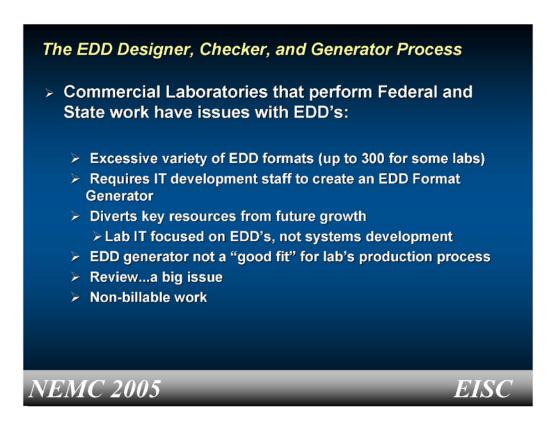


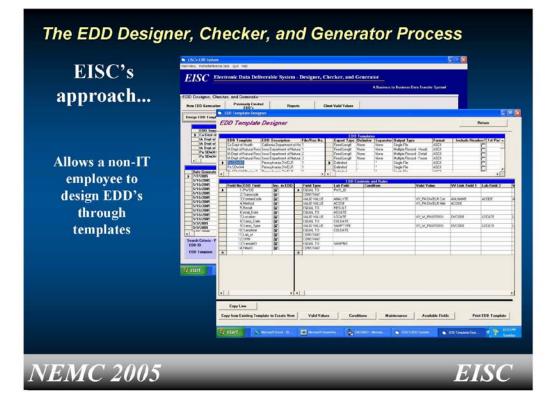


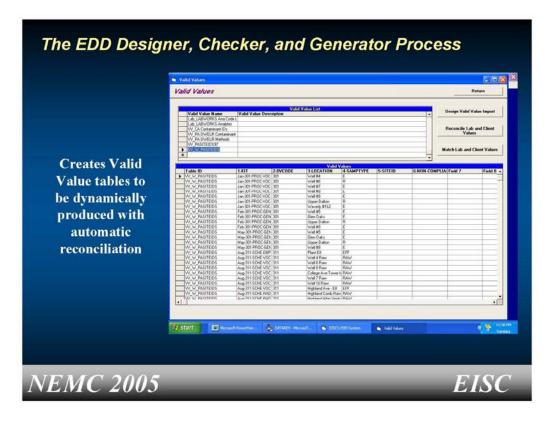






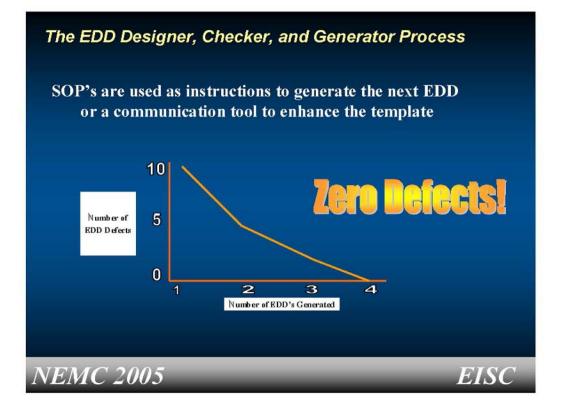


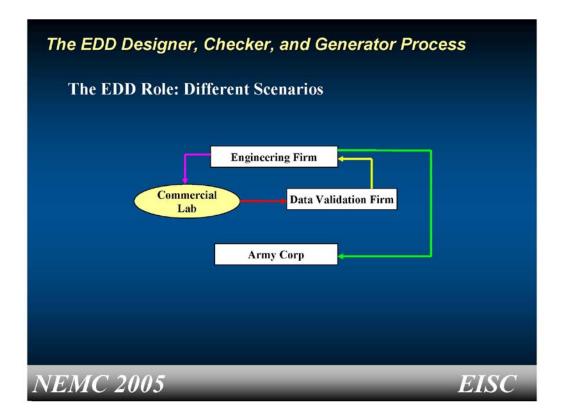


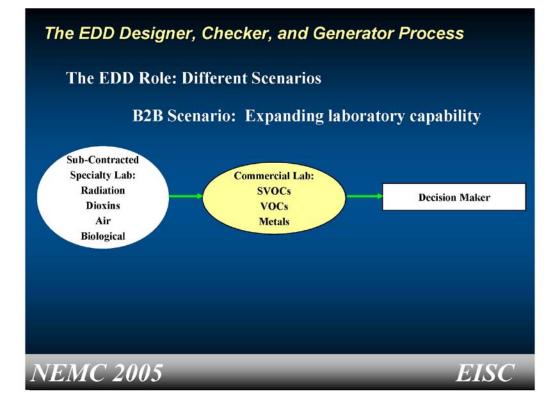


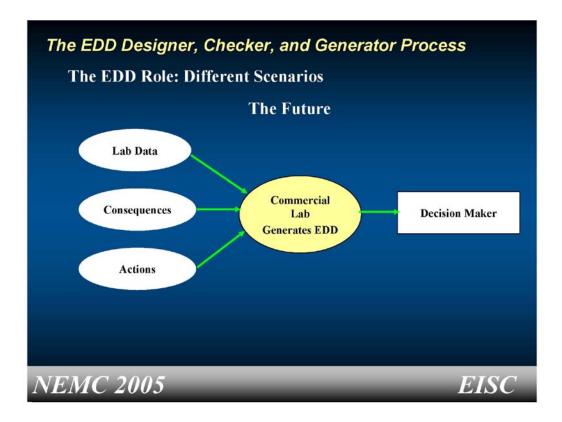
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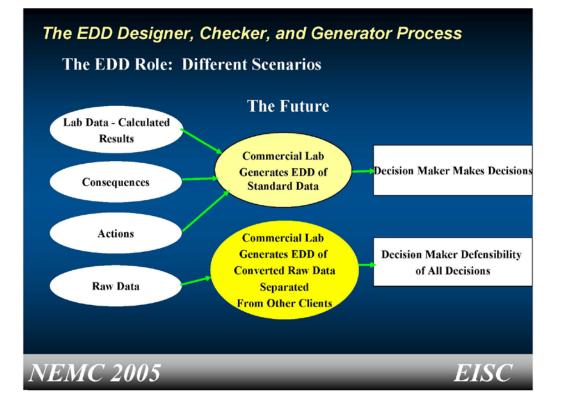
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### Conclusion

### Motivation for the Lab to Produce Quality EDD's

A lab's client will never go to a competitor (not even for price), when:

- Deliverables (report and electronic) are perfect
- Turnaround time is swift

This is a solid business strategy to build a consistent revenue base of return clients (a.k.a. Residual Revenue)!

Happy clients make a happy lab!

Have a great Day!



EISC

NEMC 2005

#### Automated Review of SEDD Stage 3 Deliverables

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#### Alfred Mayo

CSC, 15000 Conference Center Dr., Chantilly, VA, 20151 E-Mail: <u>amayo@csc.com</u> Phone: 703-818-4299

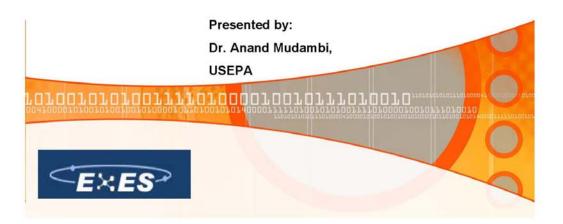
#### ABSTRACT

CSC has developed the EXES (Electronic data eXchange and Evaluation System) software for the USEPA Analytical Services Branch to review SEDD (Staged Electronic Data Deliverable) XML files up through SEDD Stage 3. EXES is designed primarily to review files submitted under the USEPA Contract Laboratory Program (CLP). However, the software has been designed to allow review of data submitted for other programs and methods or for modified CLP analyses. EXES can be used as a stand-alone system or linked to a database system. Over 4000 separate tests can be performed on a data deliverable and the results reported to the data user. The software can recalculate all values derived from the raw (quant report) data along with inspecting each required data element for presence, validity, and correctness. EXES can inspect data directly against a lookup table or by iteration (e.g., no more than x values may exceed a requirement). It also checks analysis sequence. The software provides pre-inspection capability to laboratories to allow problems to be corrected prior to submission.

I prefer to give an oral presentation.

## Automated Review of SEDD Stage 3 Deliverables:

## EPA's <u>E</u>lectronic data e<u>X</u>change and <u>E</u>valuation <u>S</u>ystem



## What is EXES?



- A system developed by CSC for the USEPA Analytical Services Branch (ASB) to evaluate analytical data technical and contractual quality.
- Performs Contract Compliance Screening based on the technical requirements of the appropriate Statement of Work (SOW).

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 Performs technical data qualification based on the National Functional Guidelines.

## Contract Laboratory Program



- Used by USEPA to evaluate samples from Superfund sites.
- In FY04 alone over 126,000 analyses were process through the CLP program.
- High sample volume requires automated review of electronic data deliverables.
- New CLP SOWs require Electronic Data Deliverables (EDD) in SEDD Stage 3.
- EXES can evaluate EDDs from all three stages of SEDD.

### **Review of SEDD Stage 3 Files - Process**



- Labs generate SEDD Stage 3 Files and upload them to the SMO Server via EXES website.
- Files are then evaluated by EXES for the different Test Types.

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• EPA also provides CLP labs (with SOM01.1 Contracts) with a Self Inspection Tool.

## Self-Inspection



- EXES is available on-line to allow CLP laboratories and their software vendors to inspect data prior to submission.
- This approach will allow CLP laboratories to correct all deficiencies found prior to delivery to USEPA.
- Ensures delivery of a complete and technically compliant data to USEPA.
- Does not guarantee that the SEDD files will be compliant with all SOM01.1 requirements.

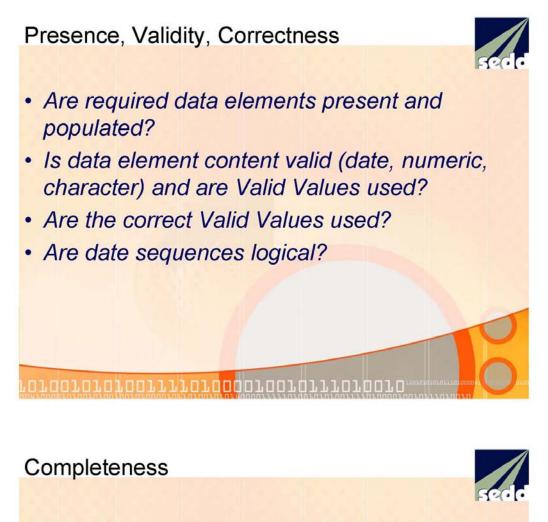
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## Test Types

• Presence, Validity, Correctness.

- · Completeness.
- Sequence and Frequency.
- · Batching.
- Recalculation.





- Are all requested samples, analyses, and analyte results present?
- Are all required dilutions, re-analyses, or repreparations present?

## Sequence and Frequency



- Are specified analytical sequences followed?
- Do QC occur at required frequencies?
- Do no more than "x" values fail to meet specified windows?

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## Batching



- SEDD has 11 batch types, SOW SOM01.1 specifies the use of seven of these.
- All samples must link to their required batches.
- No sample can link to a non-existent batch.
- Batches must contain required QC.

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## Calculation



- Calculates all calibration results from the instrument response.
- Recalculates all final reported results from the instrument response and the recalculated calibration results.
- Recalculates all spikes and additions (Internal Standards, Surrogates, MS/MSD) from standard concentrations, amounts added, and sample volumes/masses.

## 

## Flexibility



- Designed to accommodate Modified Analysis Requests.
  - Modified Quantitation Limits.
  - Modified Analyte lists.
  - Modified matrices and methods.

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 Designed to accommodate Non-CLP data so long as SEDD specifications are met.

## **Custom Reports**



- Provides electronic database ready reports via email summarizing data assessment results.
- All reports are stored at a central database for future references by users.
- Reports can be customized to accommodate each users specific reporting and technical needs.
- Saves cost and time by expediting transfer of data to end users database and eliminating manual processing of hardcopy reports.

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## Stand-Alone Version



 A stand-alone version of EXES is being planned.

- Features:
  - · Users may store results to their own databases.
  - Users may customize checks to meet their program needs.
  - Users may customize analyte lists, reporting limits, and data qualification flags.



# Environmental Data from the Field to the Map, and the Impact of EDD Formats Like SEDD

**Dr. David W. Rich** President Geotech Computer Systems, Inc., Englewood, CO

#### ABSTRACT

#### Introduction

The amount of data being gathered at environmental sites is growing at ever increasing rates. Action levels are becoming more stringent, leading to more exceedences, and the expectations for using the data is also growing rapidly. Most people recognize the need for efficient tools for managing laboratory and field data, and affordable software is now readily available to more efficiently manage data. Advances in electronic data deliverable formats such as SEDD (Staged Electronic Data Deliverable) may remove some obstacles to data exchange, improving efficiency and improving data integrity. Cost savings of 50% or more can be documented resulting from better data management, and these savings can result in a high return on investment for software purchases, staff training, and data conversion. This talk follows the data through such a system from the field to the final uses of the data, and addresses a number of data interchange issues.

#### Gathering Data

Management of groundwater and related data starts in the field, taking physical samples to send to the lab, and gathering field data. The field data is imported into the database, and then associated with the analytical data when it arrives from the laboratory. The data management system should help with all phases of this process.

#### Data Interchange Issues

Efficiently moving data between project participants is a challenging and often time-consuming issue. New data formats like SEDD have the promise of simplifying data transfer by providing a standardized interface between data providers and consumers. This should contribute to data management efficiency and data integrity. As with most things, however, there are some challenges to overcome, some inherent in the process, and some dependent on the implementation of the interchange.

#### Quality Control, Storage and Retrieval

There are many different aspects of quality control that apply to managing environmental data, many of which can be made more efficient through effective use of data management software, and by implementing efficient transfer of EDDs. The software should help with simple statistical tests such as outlier and charge balance calculations. For more rigorous checking, the software

should check holding times, spike recoveries, QC sample frequencies, and other more traditional "validation" activities before the validator makes the final determination of suitability for use. Once the data has undergone the appropriate level of review, it is stored in a central repository, usually in a normalized relational data model. The user interface of the data management system should provide selection and display tools that provide a good level of flexibility, while still being easy to use.

#### **Reports and Graphs**

In the past the primary deliverable for project data has been tabular reports. These displays remain important, and software features such as flexible and automated formatting of results, and automatic comparison to target levels, can make this process much more efficient. With the data stored in a comprehensive data management system, other displays such as time-sequence graphs, also with comparison to limits, are easy to generate, and can tell quite a bit about the site, providing a greater return on the investment in sampling and analysis.

#### **Display Using GIS**

The spatial component of contaminant distribution can be a critical factor in understanding site issues. The spatial component is very difficult to visualize from tables and graphs, but often can be easily understood with one or more maps. Tight integration between the data management system and GIS displays is the key to efficiently generating good maps, and ensuring that the quality of the data is not degraded in the process. Graphically rich displays such as callouts (data tables on the map), graphs on the map, and Stiff water quality diagrams can aid greatly in understanding site conditions and making project decisions.

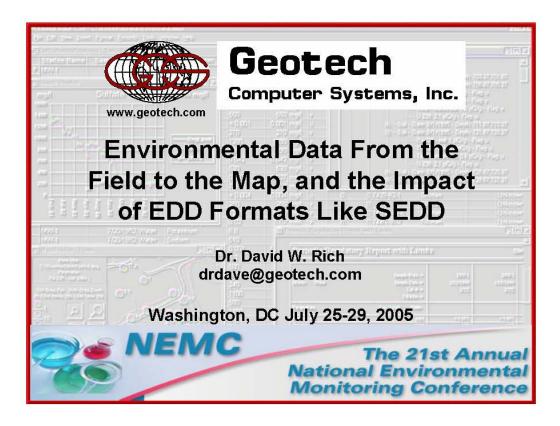
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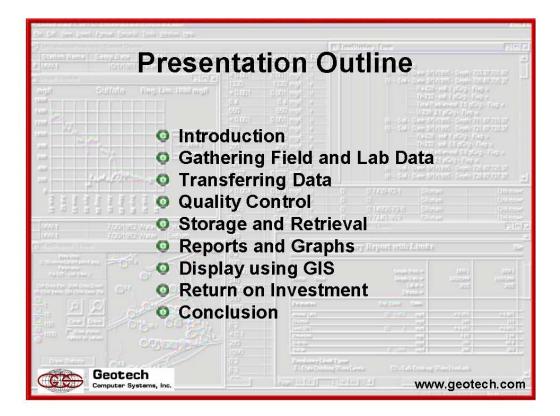
#### Author Biographical Sketch

Dr. David W. Rich has a PhD in geology and over 30 years experience solving earth science computing problems. He is President of Geotech Computer Systems, where he directs their technical and business development efforts, and is the author of *Relational Management and Display of Site Environmental Data* from CRC Press.

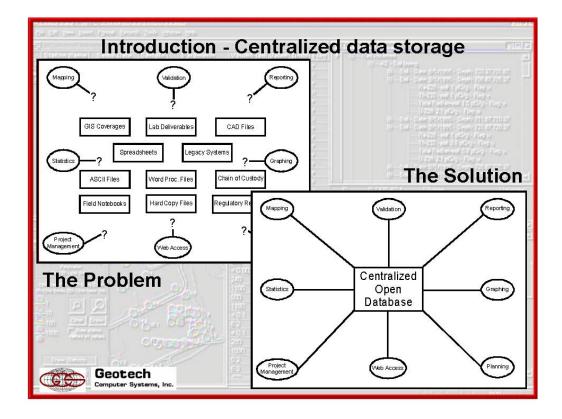
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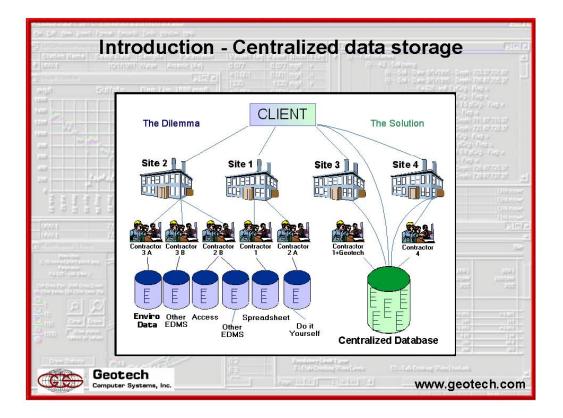
Dr. Dave Rich President, Geotech Computer Systems, Inc. 6535 S. Dayton Street, Suite 2100 Englewood, CO 80111 Phone: 303-740-1999 Fax: 303-740-1990 Email: drdave@geotech.com Web page: www.geotech.com

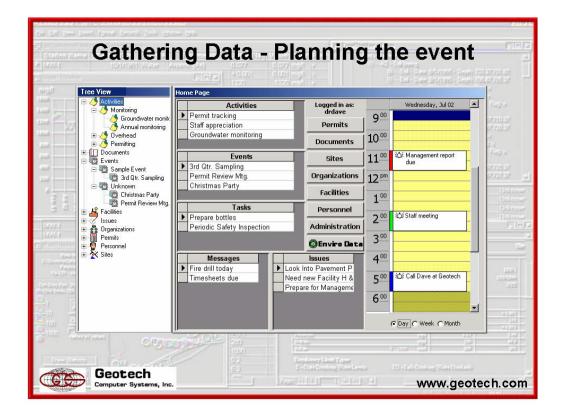








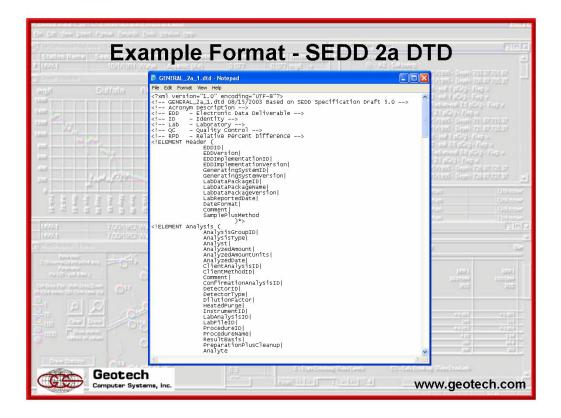




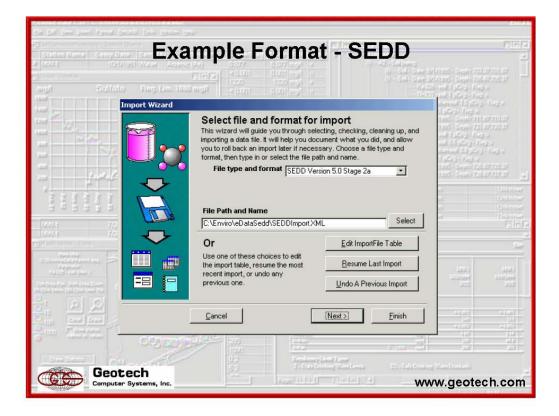
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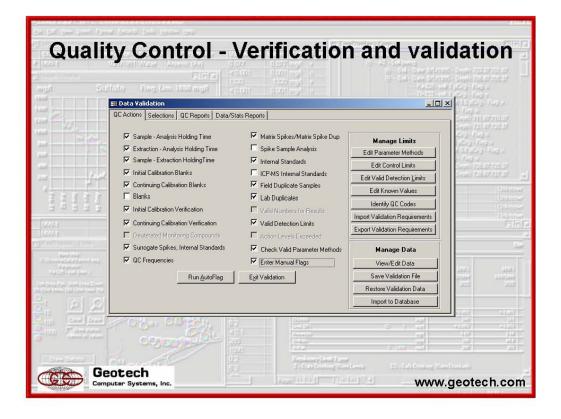


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Analyses.Detect2	ReportedResult ReportingLimit	
Analyses.Detect3	ReportedResult QuantitativeLimit	
Analyses DilutionFactor	ReportedResult DilutionFactor	
Analyses FlagCode	ReportedResult LabQualifiers	
Analyses Lab	SamplePlusMethod LabID	
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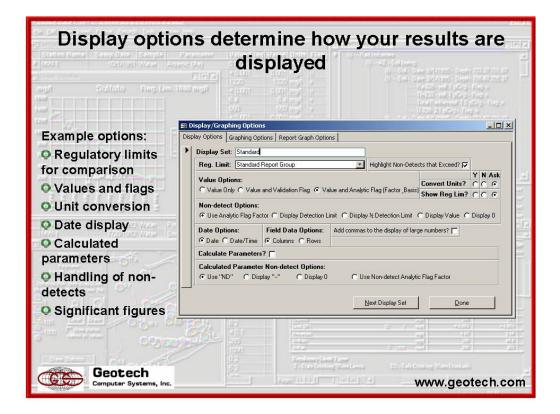
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2.3	02/26/81-05/18/94	0	52	52	0.0	5.5	1.1	1.3	1.8	0.7
6.6	02/26/81-05/18/94	0	53	53	1.1	48.0	5.3	5.2	6.3	0.2
410	02/26/81-05/18/94	0	53	53	96.6	550.0	239.2	290	164.3	1.0
220	02/26/81-05/18/94	0	53	53	120.0	380.0	181.7	190	60.6	0.6
< 4	02/26/81-08/18/93	0	50	53	120.0	380.0	181.7	190	2.1	0.6
920	02/26/81-05/18/94	0	53	53	190.0	1400 D	503.3	670	410.9	1.0
7.1	02/26/81-05/18/94	0	53	53	7.0	8.0	7.4	7.4	0.3	1.1
2070	02/26/81-05/18/94	0	53	53	850.0	2813 D	1505.2	1630	714.7	0.8
P ODI	112			Linds			ing the	(Jame)		
	A La LL									
	LAB 1 11/21/85 RESULT 160 2.3 6.6 410 220 < 4 920 7.1	RESULT         PERIOD OF DATA           160         02/26/81-05/18/94           2.3         02/26/81-05/18/94           6.6         02/26/81-05/18/94           410         02/26/81-05/18/94           2.20         02/26/81-05/18/94           4.0         02/26/81-05/18/94           2.0         02/26/81-05/18/94           2.1         02/26/81-05/18/94           2.20         02/26/81-05/18/94           2.1         02/26/81-05/18/94           2.00         02/26/81-05/18/94	LAB NO: 11/21/85 COMPARISON RESULT PERIOD OF DATA Code 160 02/26/81-05/19/94 O 2.3 02/26/81-05/19/94 O 6.6 02/26/81-05/19/94 O 410 02/26/81-05/19/94 O 410 02/26/81-05/19/94 O 410 02/26/81-05/19/94 O 40 02/26/81-05/19/94 O 40 02/26/81-05/19/94 O	LAB NO:         COMPARISON PERIOD OF DATA         QC Code         N           11/21/85 RESULT         COMPARISON PERIOD OF DATA         QC Code         N           160         02/26/81-05/18/94         O         63           2.3         02/26/81-05/18/94         O         63           6.6         02/26/81-05/18/94         O         63           410         02/26/81-05/18/94         O         63           420         02/26/81-05/18/94         O         63           410         02/26/81-05/18/94         O         63           520         02/26/81-05/18/94         O         63           7.1         02/26/81-05/18/94         O         63           2070         02/26/81-05/18/94         O         63	LAB NO:         STATION:           11/21/85 RESULT         COMPARISON PERIOD OF DATA         QC Code         N         # OF DET           160         02/26/81-05/18/94         O         63         63           2.3         02/26/81-05/18/94         O         63         63           410         02/26/81-05/18/94         O         53         63           410         02/26/81-05/18/94         O         53         63           420         02/26/81-05/18/94         O         53         63           420         02/26/81-05/18/94         O         53         63           410         02/26/81-05/18/94         O         53         63           200         02/26/81-05/18/94         O         53         63           920         02/26/81-05/18/94         O         53         63           2070         02/26/81-05/18/94         O         63         63	LAB NO:         STATION/MIV-1           I1/21/85         COMPARISON RESULT         QC         # OF DET         MIN           160         02/26/81-06/18/94         0         53         63         105.0           2.3         02/26/81-06/18/94         0         53         63         1.01           410         02/26/81-06/18/94         0         53         63         1.11           410         02/26/81-06/18/94         0         53         63         120.0           <4	LAB NO:         STATION:MVV-1           11/21/85         COMPARISON PERIOD OF DATA         QC Code         # OF DET         MIN         MAX           160         02/26/81-06/18/94         0         53         53         105.0         210.0           2.3         02/26/81-06/18/94         0         52         52         0.0         5.5           6.6         02/26/81-06/18/94         0         53         63         1.1         48.0           410         02/26/81-06/18/94         0         53         53         106.6         50.0           220         02/26/81-06/18/94         0         53         53         120.0         380.0           44         02/26/81-06/18/94         0         53         63         190.0         1400.0           7.1         02/26/81-06/18/94         0         53         53         7.0         8.0           2070         02/26/81-06/18/94         0         53         53         860.0         2813.0	LAB NO:         STATION:MW-1           11/21/85         COMPARISON RESULT         QC PERIOD OF DATA 2000         % OF DET         MIN         MAX         MEAN           160         02/26/81-06/18/94         0         63         63         105.0         210.0         145.3           2.3         02/26/81-06/18/94         0         62         52         0.0         5.5         1.1           6.6         02/26/81-06/18/94         0         63         63         1.1         48.0         5.3           410         02/26/81-06/18/94         0         63         63         120.0         380.0         181.7           44         02/26/81-06/18/94         0         63         63         120.0         380.0         181.7           920         02/26/81-06/18/94         0         63         63         7.0         8.0         7.4           2070         02/26/81-06/18/94         0         63         63         20.0         2813.0         1605.2	LAB NO:           STATION/MW-1           I1/21/85         COMPARISON RESULT         COMPARISON RESULT         YOF         MIN         MAX         MEAN MEDIAN           1100         02/26/81-05/18/94         0         63         53         105.0         210.0         146.3         131           2.3         02/26/81-05/18/94         0         63         53         1.1         48.0         5.3         5.2           410         02/26/81-05/18/94         0         63         53         120.0         380.0         181.7         190            4         02/26/81-05/18/94         0         63         53         120.0         380.0         181.7         190            4         02/26/81-06/18/94         0         63         53         190.0         1400.0         603.3         670           7.1         02/26/81-06/18/94         0         63         53         60.0         2813.0         1605.2         1630	LAB NO:         STATION:MV/-1           11/21/85         COMPARISON PERIOD OF DATA         QC Code         # OF DET         MIN         MAX         MEAN         MEDIAN         SD           160         02/26/81-06/18/94         0         53         63         105.0         210.0         146.3         131         33.4           2.3         02/26/81-06/18/94         0         52         52         0.0         5.5         1.1         1.3         1.8           6.6         02/26/81-06/18/94         0         53         63         1.1         48.0         5.3         5.2         6.3           410         02/26/81-06/18/94         0         53         63         120.0         380.0         181.7         190         60.6            44         02/26/81-06/18/94         0         53         53         120.0         380.0         181.7         190         2.1           920         02/26/81-06/18/94         0         53         53         140.0         503.3         670         410.9           7.1         02/26/81-06/18/94         0         53         53         7.0         8.0         7.4         7.4         0.3           2070<

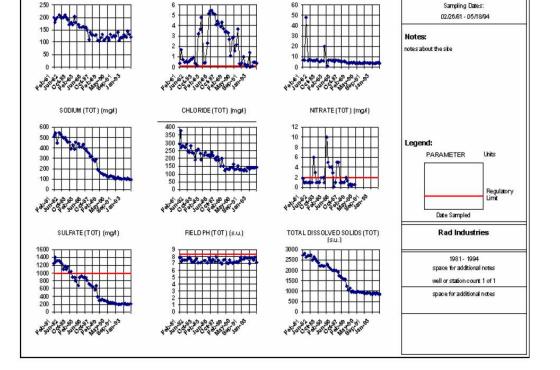
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Antiparten (a)	Use Sample Events Number of Analyses: 237	Use Station Parameters	Valid. Cd. • • - QC • • - Batch • • -		
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Dispid	y options	an			d	DetectionL	imit times ReportingFa	ictor and Flag	100
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				Site ->	Forest Products Co.	Forest Products Co.	Forest Products Co.		
			Sample I	Point ->	MWD-19	MWD-23	MWD-38		
333333	Matrix: Water		Sample	Dane ->	2/16/1998	2/16/1998	2/17/1998		
				.ab#->	XYZ	XYZ	XYZ		
			Dib	ition->	1	1	1		
(A) 7/2	Parameters	Reg. Li	mit	Units					
961 JL1	Arsenic (As)	SD	0.002	mg/l	0.0300	< 0.0050	0.0074 J		
CONTRACTOR .	Boron			mg/l	0.21 B	0.18 B	0.32 B		
And had to be to	Chronium	S	0.01	mg/l	< 0.0066	< 0.0066	< 0.0066		
inmaids fullies -	Cobalt			mg/l	< 0.0058	< 0.0058	0.0072 J		
The state of the s	Copper	PR	0.05	mg/l	< 0.0045	0.01 J	< 0.0045	1991	110
the first till beautions	Iron (Ferrous)	PR	0.1	mg/l	2.61 B	< 0.01	0.93 B		11010
the Distance in the	Margarese	SD	0.02	mg/l	0.5100	< 0.0031	1.0200		
101	Molyb denu m			mg/l	0.0100	0.0055	0.0096		
22	Nickel			mg/l	U 80.0	0.01 J	0.05		
	Selenium			mg/l	< 0.0037	0.0059 J	< 0.0037		
m Land Lind	Silver			ppm	< 0.0047	< 0.0047	0.009 J	14.0	
(12) June dames	Zinc	FM	0.05	mg/l	0.02	0.06	0.06	La.	
Canal of Links	Fluoride			mg/l	4.22	6.71	12.5	1.00	i.
	Nitrate/Nitrite	FM	0.4	mg/l	< 0.03	4.26	< 0.03		
traditions -	Sulfate	S	1000	mg/l	283	129	510		
George	tech	63			State Controls	Sand Level S	the first state of the		
Geor	CECN ar Systems, Inc.		-		2010 (0 () T			geoted	

Site: AA									
This report s limits at one		compa	rison to	multiple	Cluster -> Sample ID -> Date -> Depth ->	ZZ AA-BB-003 10/9/2001	ZZ AA-BB-005 10/25/2001	ZZ AA-BB-006 10/17/2001	Z AA-BB-00 1/14/200
Analyte	Units	Base Background	BTAG Sediment	Industrial Sediment RBCs	Residential Sediment RBC				
Pesticides	_			ocumentores					
,4-DDD	ug/kg	8.3	16	NA	NA	29 v / 20	60 U / 60	50 J / 7.5	2
,4'-DDE	ug/kg	11	2.2	ΝΛ	ΝΛ	37 v / 20	60 U / 60	31 v / 7.5	-
,4'-DDT	ug/kg	15.4	1.58	NA	NA	20 U / 20	60 U / 60	7.5 U/7.5	-
LDRIN	ug/kg	NA	NA	340	38	10 U/10	29 U/29	3.7 U/3.7	-
ALPHA-BHC	ug/kg	NA	NA	NA	NA	10 U / 10	29 U/29	3.7 U/3.7	
ALPHA-CHLORDANE	ug/kg	5	NA	NA	NA	10 U/10	29 U/29	3.7 U/3.7	
ETA-BHC	ug/kg	NA	NA	NA	NA	10 U/10	29 U / 29	3.7 U/3.7	
ELTA-BHC	ug/kg	NA	NA	NA	NA	10 U / 10	29 U / 29	3.7 U / 3.7	
DIFLORIN	ug/kg	NA	NA	360	40	20 U / 20	60 U / 60	7.5 U / 7.5	
NDOSULFAN I	ug/kg	NA	NA	NA	NA	10 U / 10	29 U/29	3.7 U/3.7	-
ENDOSULFAN II	ug/kg	NA	NA	NA	NA	20 U/20	60 U / 60	7.5 U / 7.5	-
NDOSULFAN SULFATE	ug/kg	NA	NA	NA	NA	20 U / 20	60 U / 60	7.5 U / 7.5	
NDRIN	ug/kg	NA	NA	610000	23000	20 U / 20	60 U / 60	7.5 U / 7.5	
NDRIN ALDEHYDE	ug/kg	NA	NA	NA	NA	20 U/20	60 U / 60	7.5 U/7.5	-
NDRIN KETONE	ug/kg	NA	NA	NA	NA	20 U/20	60 U / 60	7.5 U / 7.5	-
amma-BHC (Lindane)	ug/kg	3.14	NA	NA	NA	10 U/10	29 U/29	3.7 U/3.7	-
JAMMA-CHLORDANE	ug/kg	NA	NA	NA	NA	10 U / 10	29 U / 29	3.7 U/3.7	-
IEPTACHLOR	ug/kg	3.14	NA	1300	140	10 U / 10	29 U/29	3.7 U/3.7	
IEPTACHLOR EPOXIDE	ug/kg	NA	NA	630	70	10 U/10	29 U/29	3.7 U/3.7	-
<b>JETHOXYCHLOR</b>	ug/kg	NA	NA	1E+07	390000	100 U / 100	290 U / 290	37 U/37	
OXAPHENE	ug/kg	NA	NA	52000	580	200 U / 200	590 U / 590	74 U / 74	-
CBs									
AROCLOR-1016	ug/kg	NA	22.7	82000	5500	20 U / 20	29 U / 29	37 U / 37	-
ROCLOR-1221	ug/kg	NA	22.7	2900	320	20 U/20	29 U / 29	37 U / 37	-
AROCLOR-1232	ug/kg	NA	22.7	2900	320	20 U / 20	29 U/29	37 U / 37	
ROCLOR-1242	ug/kg	NA	22.7	2900	320	20 U/20	29 U/29	37 U / 37	-
ROCLOR-1248	ug/kg	NA	22.7	2900	320	20 U / 20	29 U/29	37 U / 37	
ROCLOR-1254	ug/kg	NA	22.7	2900	320	20 U/20	29 U/29	37 U / 37	-
ROCLOR-1260	ug/kg	NA	22.7	2900	320	20 U / 20	29 U/ 29	37 U/ 37	

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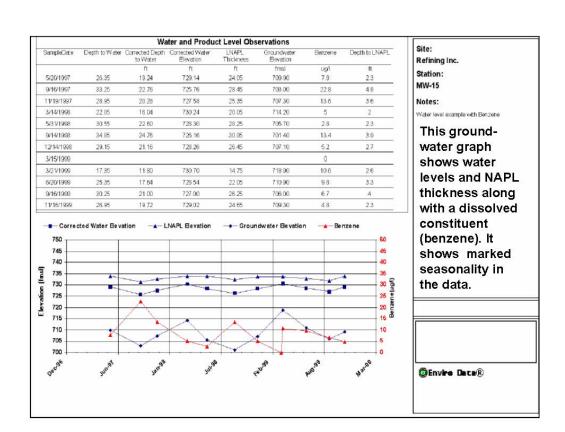


POTASSIUM (TOT) (mg/)

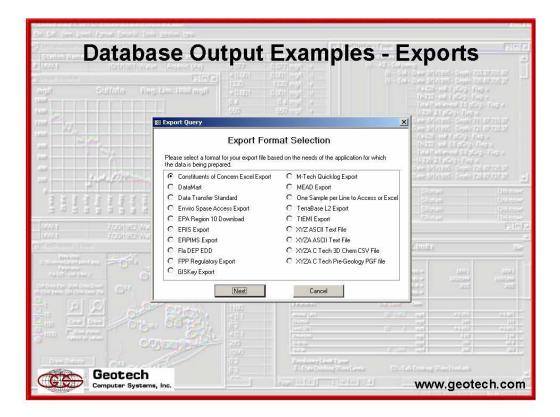
SOURCE: MW-1

IRON(FERROUS)(TOT) (mg/l)

CALCIUM (TOT) (mg/)



ListSelectedAnaly:	ses : Select Que						ES Tre	eDisplay : Form		
Station Name	Samp.Date	Sample	Parameter	Value/Flag	Value Unit	s Flag		- Bad Industries		
MW-1	10/1/1981		Arsenic (As)	0.077	0.077 ma/l	v		A2 - Soil borin	a	
	101111001	THATCH		< 0.001	0.001 ma/l	u		i Soil∙	Date: 9/1/1995 - Depth	703.37:701.87
			_ 🗆 🗵	1320	1320 mg/l			⊜Soil+	Date: 9/1/1995 - Depth	
ng/l	Sulfate	Reg. Li	im. 1000 mg/l	< 0.001	0.001 ma/l				-Ra-226 · soit 1 pCi/g ·	
00 1 1 1 1				6.4	6.4 mg/l	v			Th-232 - soit 1 pCi/g -	
00 -				550	550 ma/l				-Total Radium-soit 1.5	
				< 0.001	0.001 mg/l	u		H-Sol-	U-238:2.1 pCi/g - Fla Date: 9/1/1995 - Depth	
00				270	270 mg/l	v			Date: 9/1/1995 - Depth Date: 9/1/1995 - Depth	
00		-	RegLimit	1280	1280 mg/l			5.00	-Ra-226 - soit 1 pCi/g -	
00				290	290 mg/l				-Th-232 - soit 0.6 pCi/g	
00	<b>N</b> .			0.042	0.042 mg/l	v			Total Radium-soit 0.8	pCi/g - Flag: u
				530	530 mg/l				-U-238: 2.1 pCi/g - Fla	
00				0.027	0.027 ma/l				Date: 9/1/1995 - Depth	
10			13	7.3	7.3 mg/l			⊡ — Sol •	Date: 9/1/1995 - Depth	x 728.87:727.37
0	·			< 0.004	0.004 ma/l	u	0	0 7439-92-1	Silurian	Unkno
5 2 8 A	M. Se	7 2		250	250 ma/l		0	0	Silurian	Unkno
Apr-85 Nov-83 Jul-82 Feb-81	Sep-90 May-89 Jan-88 Aug-86	Jun-93 Feb-92		1240	1240 mg/l		0	0 14808-79-8	Silurian	Unkno
	* * 2 5	NÜ		0.02	0.02 mg/l		0	0 7440-38-2	Silurian	Unknow
M/8/-1	7/20/1982	Water	Potassium	6.8				port with Limits		- 0
MW-1	7/20/1982		Sodium	510	Contraction of the local division of the loc		COLUMN ST			
MapDisplay   Form			_ [D] ×	0.001	C Pe	riodic I	2emila	tory Report with I	imite	S
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Parameter: Ra-220 - soil (Max.)	· · · · ·	-		< 0.001	C			Sample Po	MW-1	MW-1
		0	11	240	Matri	x: Water		Sample D		
rt-Drag:Pan Shift-Drag: Click Menu Dbl-Click Ve			OYL	1100				Li Diha	b#-> XYZ	: XVZ
LINE MENU DOI-CHOK WE	and and	100		500	_	27				
-1 p	$\mathcal{Q}$	Ora	Omul	1180	Pare	neles		Reg. Linuit	Civitis	
-10		12		< 0.1		ř (&)		SD 0.002	mgi <0.001	
- 100	)raw	14 30.1		6.2	Chlor			SD 5	mgi 140 mgi <0.005	
-1000 Show sta		0	O'AN .	< 0.1	Potas			au 3	mga ~0.003 mgl 4.03	
names or v	Alles	OOK	TO BARRA	280	Soda	m			mg1 100	96.6
	1	-00		1090	Sulfa			S 1000	mgl 200	205
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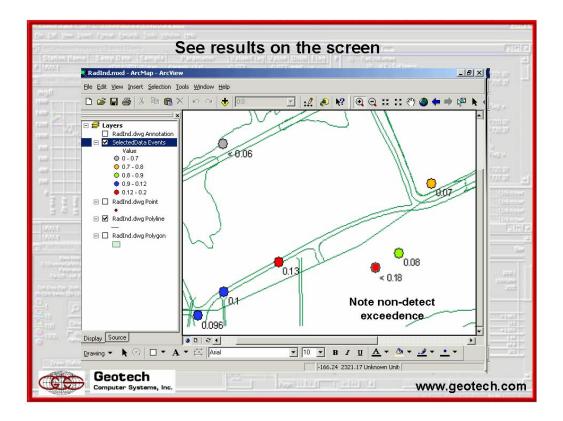


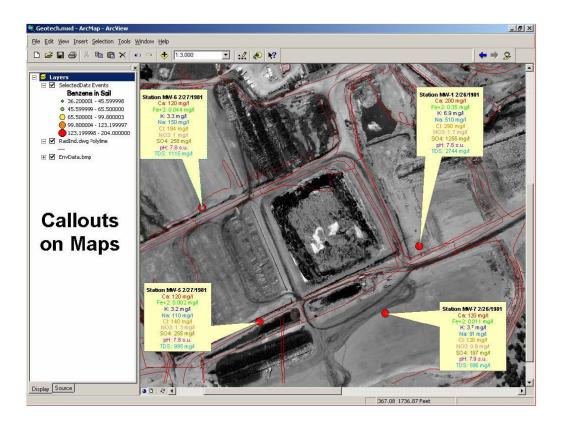
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ACADM FI		0000 ww 0		L HCS	EPA 826 NONE	None	1 MTLNCL	
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		0000 ww 0	0 2	L HCS	EPA 826 NONE	None	1 DBCME	

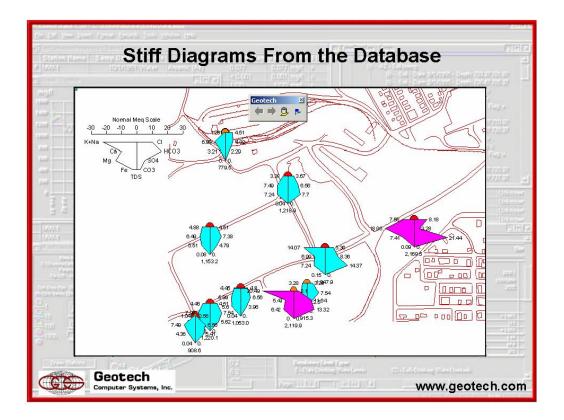
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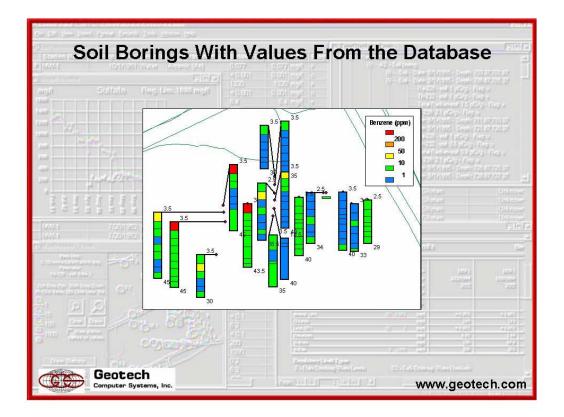
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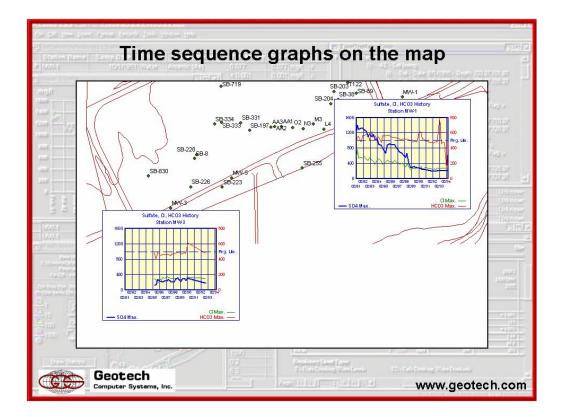
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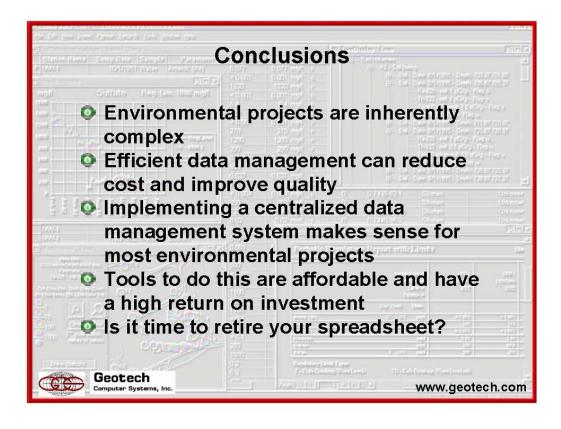


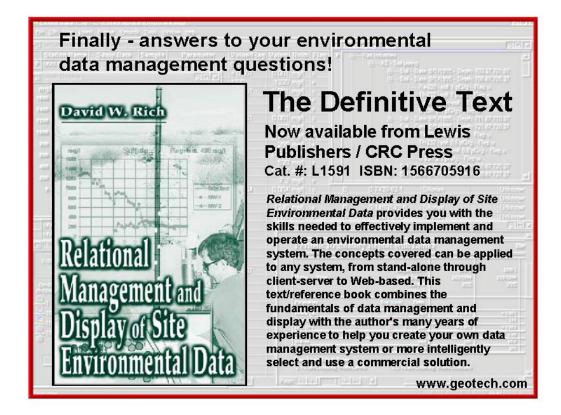


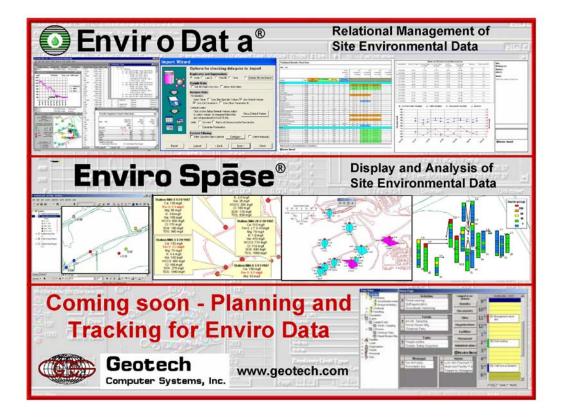




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Cost items Software	\$4.000	
Support (3 years)	2,400	(
Total cost	\$6,400	5/ 2/
Cost savings		
Data loading – save 50% of 4 days per year at \$80 per hour, for 3 years	\$3,840	0 <u></u>
Analysis – save 50% of 4 days per year at \$80 per hour, for 3 years	\$3,840	
Reporting – save 50% of 4 days per year at \$80 per hour, for 3 years	\$3,840	
Total savings	\$11,520	a <del></del>
Payback – \$11,520 ÷ 6,400	1.8:1	3.
Plus intangibles	Work quality	
	Client satisfaction Staff morale	







### Remote LIMS Access from Sample Login to Result Retrieval

#### Rebekah Johnson and Christine Paszko, Ph.D.

Accelerated Technology Laboratories, Inc., 496 Holly Grove School Road, West End, NC 27376

Primary Author's E-Mail: rjohnson@atlab.com; Phone: 910-673-8165

#### ABSTRACT

In today's complex and dynamic business environment, laboratories are focused on maximizing customer lifetime value, providing those customers with real-time access to data and controlling the cost of service. To survive in this environment, laboratories must offer remote Laboratory Information Management System (LIMS) access solutions to their customers. This presentation will describe a remote access system that will allow remote sample login functionality and result retrieval.

Request Point<sup>™</sup> is a web based system that provides remote LIMS access for sample login. Request Point<sup>™</sup> allows customers of a laboratory to submit order requests over the Internet. Instantly, the laboratory is notified of the request and the laboratories' LIMS can be configured to log in the order. The system also allows customers to review the status of their orders submitted online from login to results. With this power of remote login, the order process is streamlined for laboratories and their customers.

Result Point<sup>™</sup> is a system for remote LIMS access for result retrieval. This system allows the customer, who has a valid username and password, to login and view the status of their samples from pending entry to pending approval, as the samples travel through the analysis process. The power of Result Point<sup>™</sup> is realized through the access of real-time results. When results are entered into the LIMS, customers will instantly be able to view their results. Not only will customers be able to view results, they will be able to generate reports. Reporting functions currently provided in the LIMS are extended to the customer by offering complete analytical reports with customized letterhead, if desired. With the power of remote results retrieval, laboratories can offer instant results and satisfaction to their customers.

Reducing overhead and maximizing the customers experience with a contract analytical laboratory are some of the advantages resulting from remote LIMS access. These systems allow users to extend the power of the LIMS from the laboratory to their customers.

(2) I prefer to give an oral presentation.

#### Application of Electronic Data Verification with Data Validation to Site Characterization Projects to Maximize Efforts

**Stephen T. Zeiner, CEAC, CPC, Ruth L. Forman, CEAC, and David R. Blye, CEAC** Environmental Standards, Inc. 1140 Valley Forge Road, Valley Forge, PA 19482-0810 Primary Author's E-mail: <u>Szeiner@EnvStd.com</u> Phone: 610-935-5577

#### ABSTRACT

Data validation has traditionally been used to determine the usability of the reported analytical results for site characterization and site remediation. In the recent past, data validation was required to be performed on 100% of the data for federal and state-led project sites and litigious sites. There is a growing trend to use electronic data verification (EDV) to assess data usability. Although it is time-efficient and cost-effective to utilize the automated EDV process to evaluate project data, relying on EDV alone can result in increased costs based upon decisions made using incorrect or incomplete information. Utilizing data validation and EDV has proven to be a very powerful combination in understanding site characterization data.

Electronic data verification is an automated process by which the quality assurance/quality control (QA/QC) results supplied in an electronic data deliverable is utilized to assess the usability impact of select QA/QC information and to subsequently apply applicable data qualification codes to the associated investigative sample results. EDV generally evaluates only a subset of the QA/QC that is evaluated during the data validation process. EDV assumes that the reported analytical results and associated QA/QC results are correct as reported by the laboratory. EDV is quicker and less expensive than data validation because EDV is an automated process.

Data validation, which must be performed by an experienced chemist, is a process by which the data package deliverable is reviewed relative to the following areas: correctness of the reported analytical results; completeness of the hardcopy data package deliverables to substantiate the reported analytical results; compliance with the associated analytical method and/or site-specific project requirements; and usability of the analytical results. Data validation examines all aspects of the data from sample receipt through data reporting, inclusive of the raw data for investigative samples, QA/QC samples, and calibrations. Data validation does not make any assumptions relative to the correctness of the information provided in the hard copy data package deliverable. Data validation requires more time and is more expensive than EDV due to the extensive labor involved.

This presentation will briefly describe the items reviewed during the data validation and EDV processes. In addition, this paper will present case studies involving large environmental investigations that utilized a combination of EDV and data validation to assess data usability. These case studies will provide examples of "issues" identified in EDV that were not issues when data validation was performed and, conversely, "issues" that were not identified in EDV and were issues when data validation was performed. These case studies will provide a demonstration of how data validation and EDV work together to improve the understanding of the site characterization data.

I prefer to give an oral presentation.

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## Application of Electronic Data Verification With Data Validation to Site Characterization Projects to Maximize Efforts

Stephen T. Zeiner, CEAC, CPC; Ruth L. Forman, CEAC; and David R. Blye, CEAC



## **Electronic Data Verification**

- Electronic Data Verification (EDV) is:
  - A program add-on to a data base
  - An automated process
  - Evaluates results based on programmed logic using data from an EDD





## **Typical EDV Components**

- Typical QA/QC elements evaluated:
  - Holding Times;
  - Percent Solids:
  - Method/Field/Trip blank results;
  - Matrix spike/matrix spike duplicate results;
  - Laboratory/Field duplicate results;
  - Surrogate compound recoveries; and

3

– Laboratory control sample results.



## **EDV Advantages**

- Automated process
- Error reduction
- Speed of assessment
- Lower overall cost





## EDV Disadvantages

- Evaluates a limited set of QC data
- · Set up cost for smaller projects
- Modification costs for project specifics
- Assumes reported data in the EDD are correct





## What Is Data Validation?

- Typical data validation includes a review of a hardcopy data package for:
  - Correctness (qualitative ID and quantitation) of reported data;
  - Completeness of deliverable;
  - Method compliance; and
  - Usability of the results.



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## **Data Validation Components**

- Data validation includes all of the EDV elements.
- · Additional elements:
  - Sample receipt information;
  - Initial and continuing calibrations;
  - Internal standard results;
  - Instrument check results;
  - Instrument blank results; and
  - Evaluation of raw data.



## **Data Validation Advantages**

- Complete evaluation of all results
- · Data base not required
- · No size restriction on project
- · Raw data review for interferences





# Data Validation Disadvantages

- Time consuming
- Requires expert knowledge
- · Higher cost than EDV
- Potential for human error





# Case Study 1 - Background

- Characterize soil and monitor groundwater and surface water
- Perchlorate and 1,4-dioxane drivers
- Four laboratories
- QAPP
  - Defined Data Quality Objectives
  - Reporting/Method Guidance



# Case Study 1 - Process

- Analytical data in a single data base
- EDV performed on all results
- 10% of soil underwent data validation
- Majority of aqueous underwent data validation



# Case Study 1 - Results

- All EDV qualifications were reflected in the data validation reports
- Data validation identified several issues that were not covered under EDV
  - Improper quantitation
  - False positives
  - Incorrect sampling and analysis dates
  - Calibration Issues
  - Chromatographic interferences



## Case Study 1 - Summary

- Data validation was able to identify and correct systematic issues such as:
  - Reporting errors
  - Quantitation and initial calibration issues
- Laboratories were able to adjust processes to improve data reliability
- EDV and data validation addressed a budget issue without breaking the bank

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## Case Study 2 - Background

- Characterize large sediment site
- Analyte list is short but PCB driver
- Five laboratories splitting analytical load
- QAPP includes:
  - Data quality objectives
  - SOPs for preparation and analysis



# Case Study 2 - Process

- Field and analytical data in a single data base
- EDV performed on all results
- First year: each group has PCB audit sample
- Remaining years: 6% of PCB data
- Random selection of two delivery groups for other analytes
- Total of 20% of data underwent validation



## Case Study 2 - Results

- All EDV qualifications were reflected in the data validation reports
- First year: data validation identified several issues that were not covered under EDV
  - Reporting errors
  - Method compliance issues
- Remaining years, data validation did not identify issues outside of EDV qualifications



# Case Study 2 - Summary

- First year, data validation was able to identify and correct systematic issues
- Remaining years EDV and data validation identified same issues
- EDV and data validation were used to increase the confidence in entire data set



# Case Study 3 - Background

- Characterize numerous small sites
- One laboratory
- Large range of analytes including:
  - Metals/mercury
  - Volatile organic compounds
  - GRO/DRO/RRO
  - $-CI/SO_4$
- No QAPP



# Case Study 3 - Process

- Analytical data in a single data base
- EDV performed on all results
- About 20% of samples underwent data validation
- Utilized laboratory limits for data evaluation in EDV and data validation



## Case Study 3 - Results

- First couple of years laboratory had issues with EDD and EDV was not possible
- · Laboratory erratically updated limits
- Data validation identified several issues that were not covered under EDV
  - Hard copy did not match EDD
  - Large disparity between total and dissolved metals
  - Elevated temperatures upon receipt



# Case Study 3 - Summary

- Inability to produce an EDD eliminated the cost savings of the central DMS and prevented EDV for the first couple of years
- Data reporting issues further reduced the efficacy of the DMS and EDV
- Laboratory was able to fix the issues



# Case Study 4 - Background

- Characterize and Remediate many sites
- Large number of analytes
- Seven laboratories providing analyses
- · Sites tied to a single central document
- QAPP includes:
  - Data quality objectives
  - Analytical method guidelines
  - Reporting requirements



# Case Study 4 - Process

- Field and analytical data in a single data base
- EDV performed on all results
- Random selection of 10% of total samples collected underwent data validation



# Case Study 4 - Results

23

- All EDV qualifications were reflected in the data validation reports
- Data validation identified several issues that were not covered under EDV

- Chromatographic interferences
- ICP response suppression
- Reporting errors
- Method compliance issues
- Sample receipt issues



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# Case Study 4 - Summary

- Data validation was able to identify and correct systematic issues such as:
  - Reporting errors
- Data validation was able to identify matrix issues such as
  - Chromatographic interferences
  - ICP response suppression
- EDV and data validation were used to increase the confidence in entire data set



# Conclusion

- · Laboratory performance is critical to success
- EDV and data validation can be used to improve laboratory performance and data quality over time.
- The combination of EDV and data validation enhance the advantages of both while minimizing the disadvantages.





## Setting the Standards for Innovative Environmental Solutions



## Modernization of EPA's Superfund Contract Laboratory Program (CLP) through Method Customization, Electronic Data Delivery, and Client Support

### **Bruce Means**

US EPA 1200 Pennsylvania Ave., Mail Code 5102G Washington DC 20460 E-Mail: <u>means.bruce@epa.gov</u> Phone: 703-603-8815

## ABSTRACT

Since its inception in the 1980s, Superfund's Contract Laboratory Program (CLP) has provided EPA's regional Superfund community analytical data of known, documented, and court defensible data. Originally designed to offer analytical support for the more "routine" site projects, the CLP has now evolved into a complete turn- key, full customer support mechanism, offering: Ability to log field sample information using CLP's Field Operations and Records Management System (FORMS II Lite); sample scheduling with contemporary Organic and Inorganic Environmental Testing Laboratories via the Sample Management Office (SMO); flexibility in requesting analyses for specific analytes, reporting levels, and quality assurance requirements; data delivery through Staged Electronic Data Deliverable format (SEDD), data assessment using the CLP Data Assessment Tools (DAT), and invoice processing through use of our Web Based Invoicing System (WIS). The "new" CLP was purposely re-designed to provide maximum flexibility, while providing the highest level of legal defensibility. The combination of these services has proven to be better, faster, and more cost effective for HQ EPA and its customers.

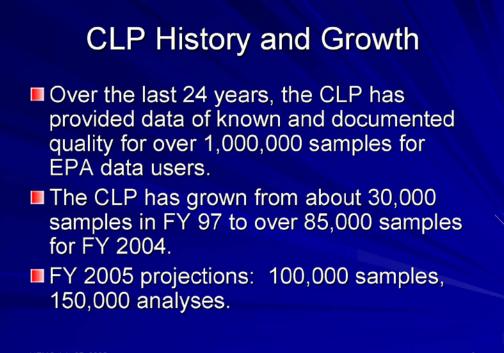
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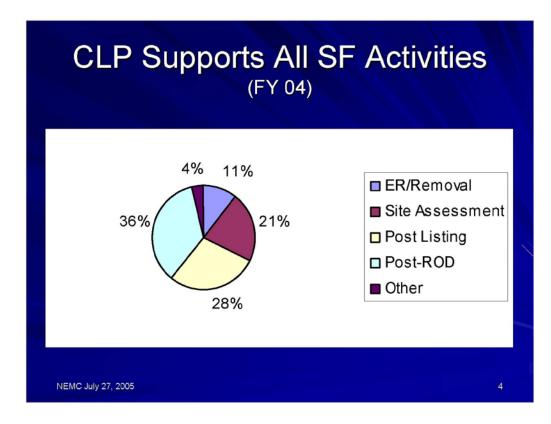
## Modernization of the Superfund Contract Laboratory Program

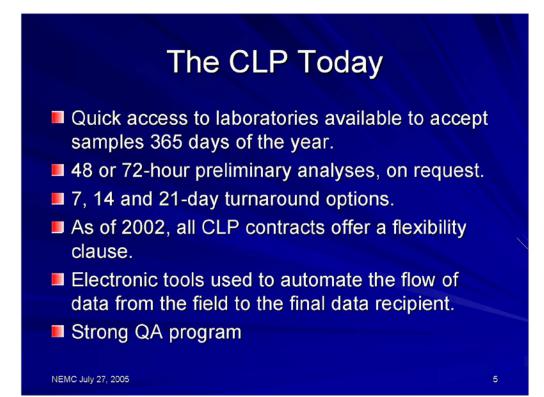


NEMC 2005 Bruce Means US EPA Analytical Services Branch

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## The New CLP Process

Field personnel coordinate sample shipment to CLP labs through Superfund's Sample Management Office contractor (SMO).

SMO assigns a lab(s) to each site project based upon scope of project, lab capacity, and the labs prior performance ranking.

Measuring Lab Performance	
<ul> <li>SMO monitors 100% of generated data:</li> <li>Timeliness;</li> <li>Completeness;</li> <li>Adherence to the SOW;</li> <li>Accuracy.</li> </ul>	
~3500 separate data checks are made for each group of laboratory data. NEMC July 27, 2005	

## **Rewarding Good Performance**

Laboratory performance results are then weighted and considered with their prices to come up with a final ranking.

Lab rankings are performed on a monthly basis.

Top performers are considered first in assigning new work.

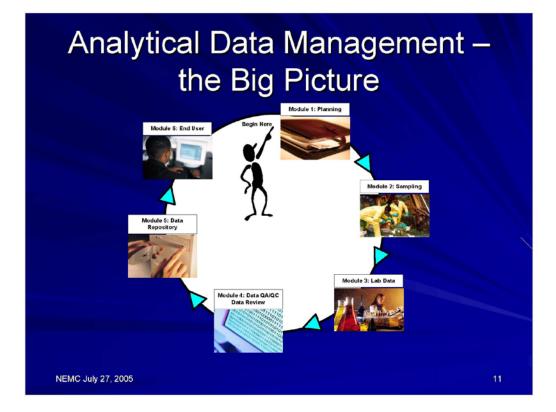
NEMC July 27, 2005

## Support for Regional Data Review and Validation

Performance monitoring results delivered to the data user within 24-48 hours of receipt of data from the lab.

Results are used for Regional data validation.

On request, SMO provides specialized computer aided data review of data formatted to meet user needs.



## **CLP Electronic Tools**

Field Operations Records Management System (FORMS) II Lite

Data Assessment Tool (DAT)

Web Contract Compliance Screening

Web Invoicing System (WIS)

## FORMS II Lite

A flexible, stand-alone, Windows-based software that automates documentation for CLP and non-CLP samples.



13

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DRP20	Soil/Sediment / Frankie Jewell	L/G	BNA (21), PEST (21), VOA (21)	T4-096 (ice Only), T4- 097 (ice Only), T4-098 (ice Only) (3)	EM-06-S	D 10/13/1999	19:00	MDRP20	
DRP21	Soll/Sediment / Frankie Jewell	L/G	BNA (21), PEST (21), VOA (21)	T4-100 (Ice Only), T4- 101 (Ice Only), T4-102 (Ice Only) (3)	EM-07-S	D 10/13/1999	18:30	MDRP21	
DRP22	Soil/Sediment / Frankie Jewell	UG	BNA (21), PEST (21), VOA (21)	T4-103 (ice Only), T4- 104 (ice Only), T4-105 (ice Only) (3)	EM-08-S	D 10/13/1999	18:15	MDRP22	
DRP23	Soil/Sediment / Frankie Jewell	UG	BNA (21), PEST (21), VOA (21)	T4-107 (loe Only), T4- 108 (loe Only), T4-109 (loe Only) (3)	EM-09-S	D 10/13/1999	18:00	MDRP23	
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## **FORMS II Lite Advantages**

Generates tags, bottle labels, Traffic Reports, and chain of-custody records.

- Facilitates electronic transfer of sample information to other databases (XML).
- Saves up to 10-15 minutes of work per sample (~ \$1M/year)

## Data Assessment Tool (DAT)

- Assesses >3,500 contract compliance and QC parameters within 24 to 48 hours of receipt.
- Provides customized electronic deliverables for direct input into client databases.

>\$17 million in savings.

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## Web-Based Contract Compliance Screening

- Web-based tool designed to check deliverables prior to submission.
- Ensures accurate and complete data packages.
- Significantly reduces errors in final data deliverables.
- Greatly facilitates on-time payment.

## Web-Based Invoicing System



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- Allows CLP contractors to electronically bill EPA for work performed.
- Billing is done in safe and secure environment.
- Invoices built on information already present in EPA Databases.

## WIS - Advantages

- Eliminates paper invoices
- Eases monitoring of invoices needing payment
- Eliminates re-keying of invoice data
- Reduced the number of disallowed invoices by 96%.



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Awards: Summer 2005

- Statement of Work (SOM 1.1) details methods consistent with SW 846.
- Requires use of the Staged Electronic Data Deliverable (SEDD Stage 3).
- Contact: Anand Mudambi
  - 703-603-8796
  - Mudambi.anand@epa.gov

NEMC July 27, 2005



## Staged Electronic Data Deliverable (SEDD)

- New electronic data deliverable format developed jointly with USACE.
- Open, non-proprietary.
- Uses XML to transmit the data.
- New EPA RAC and START contracts also require SEDD.

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## **SEDD Benefits**

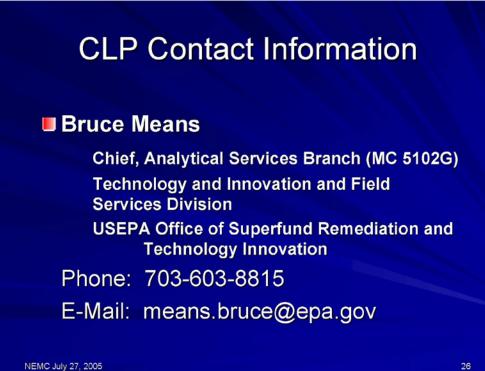
- Will reduce industry-wide EDD confusion.
- Will improve compatibility and versatility in electronic data reporting, review, handling, and archiving.
- Pilots suggest data review time and cost savings of 30-50%.
- Interagency effort: EPA, USACE, Air Force, Navy, others working to implement.

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## **Other National Non-Routine Analytical Services**

Air analysis by TO-14, TO-15 Dioxin by High Resolution GC/MS. PCB congeners (all 209) via analytical protocol developed from OW Method 1668A.

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## **New Jersey Beach Monitoring Solution**

## **Robert Peeples, PE**

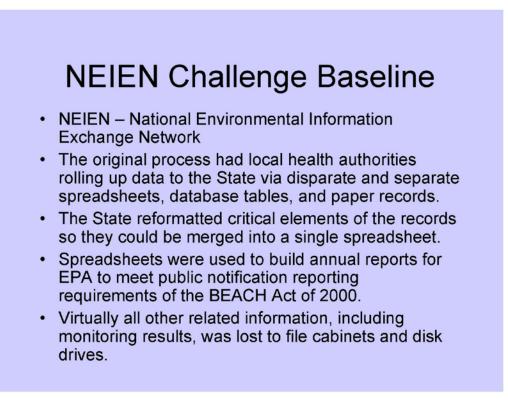
BMS Project Manager Earth 911

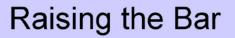
## ABSTRACT

Developed for the State of New Jersey, this monitoring solution package is a fully paperless Electronic Data Delivery (EDD) system from beach sampling field data to EPA reporting. Our work with New Jersey and four other states on the 2003 NEIEN Challenge Grant taught us that in order to send quality data to EPA, data quality must remain in control starting from the shoreline. Handheld and tablet computers were used to demonstrate a wireless web system for field data entry, and web-based forms for laboratory results posting and risk-based decisions. The system automatically updates both the New Jersey Web site (www.NJBeaches.org) and the Earth 911 Web site (www.Earth911.org) and retains the information necessary to send reports to the EPA activity tracking database (PrAWN) and the EPA water quality database (StoRet) through the state exchange node or Earth 911's virtual state node, which we have registered with the Central Data eXchange (CDX). This year we added handheld XML-based entry systems to act as a temporary solution when wireless communications are not available or costeffective. The system is automatically updated on return to the office by synchronizing the handheld database across the Internet. New Jersey is offering this system free of charge to anyone who may wish to implement it in their state. Earth 911 is also available to help with the implementation of this new EDD system.

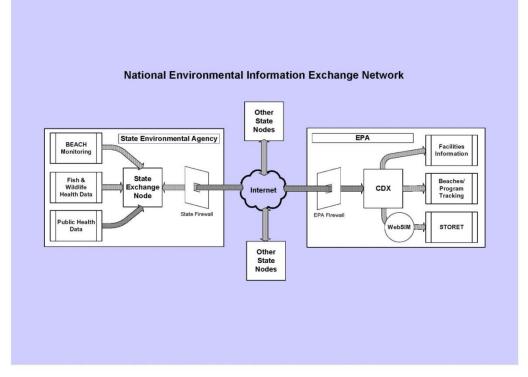
## New Jersey Beach Monitoring Solution: Lessons Learned in the NEIEN Challenge Process

Robert S Peeples, PE Earth 911



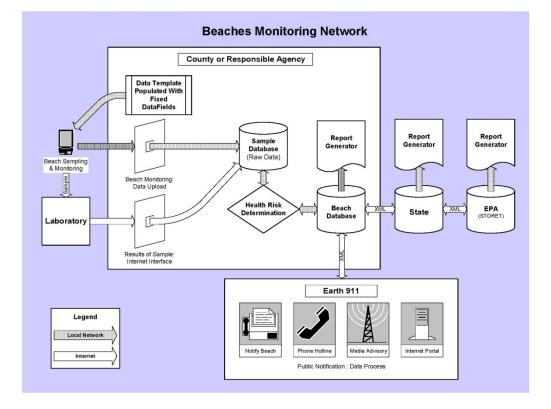


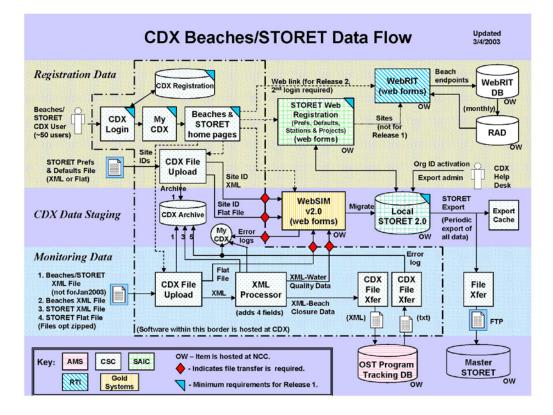
- EPA decided to add monitoring data reporting requirements to the BEACH program.
- EPA's oldest database, StoRet, was to be used for reporting monitoring data.
- StoRet required substantial modification in order to accept modern data transfer techniques.
- The NEIEN was in development as a method of automating reporting through a Central Data eXchange (CDX).

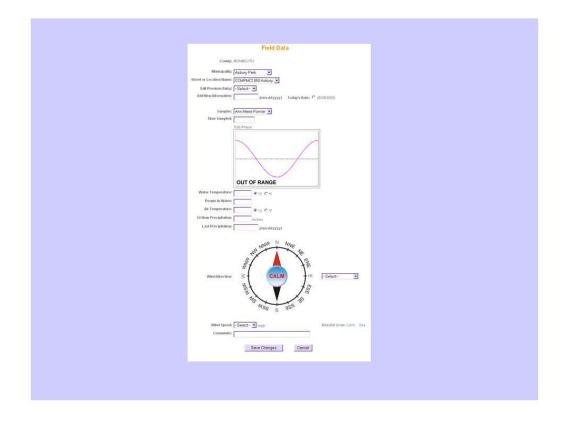


## The Obvious Solution?

 Control the data flow from generation to reporting in a single, coherent data management system.







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# **Questions?**



## **Contact Information**

- Bob Peeples
  - Telephone Number: 480-889-2650
  - E-Mail: bpeeples@earth911.org
  - www.earth911.org



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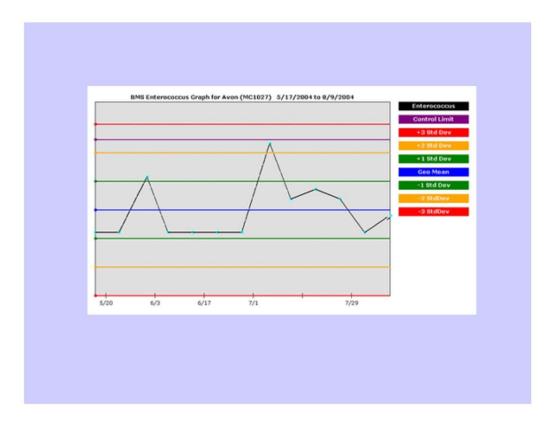


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# Crossing the Digital Divide: Looking to the Future through Quality Assurance of Records Management

#### Mary Thomas Sullivan, MLS, CRM, CQIA

Associated Records and Information Services, P.O. Box 937, Caddo Mills, TX 75135 Email: <u>aris@associatedrecords.com</u> Phone: 903-527-2156

#### ABSTRACT

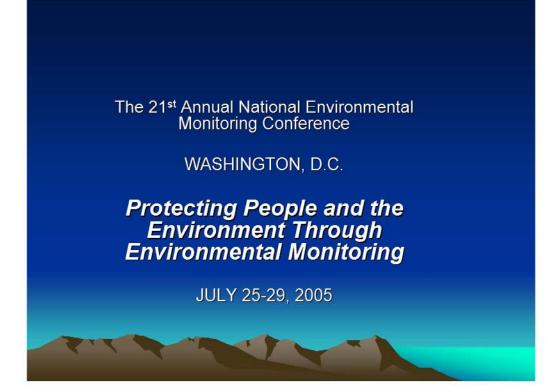
Documents that are not filed correctly run the risk of not being retrieved. This principle applies to electronic files as well as paper files. This presentation will

- Go beyond offering a surface view of the various aspects of records management as it pertains to industry, laboratory and homeland security.
- Show ways information may be saved and migrated as software and hardware technologies advance.
- Present future methods of saving electronic information.
- Review methods of preventing loss of information due to a disaster as well as preventing the loss.
- Include scenarios to illustrate information included in the presentation.
- Enable the attendee to learn how knowledge management will increase the viability and credibility of the organization as a whole.

We are pioneers standing on the new frontiers of quality. Quality defines the information gained from processes in the organization and ensures the management of such information through the quality assurance of records management. Quality assurance of information is a strong defense for the country. As we cross this new frontier let us ensure the high standards of quality are maintained at all levels of research and policy implementation.

Join others at this presentation to learn the benefits of good records management and the problems associated with poor records management. Learn where disaster begins when quality assurance is left out of records management.

I prefer to give an oral presentation.





## **ITEMS OF CONCERN**

- Management of Files
- Management of Indexing
- Management of Computerized Documents
- Management of Email
- Management of Information

## MANAGEMENT OF FILES

- File Naming
- File Indexing
- File Arrangement
- Protection of Information

## MANAGEMENT OF FILES

- Consequences of Poor Management of Files:
- Increase in costs
- Loss of Credibility
- Higher Audit Fees
- Slower Disaster Recovery

## MANAGEMENT OF INDEXING

- Purpose of Retention Plan
- Indexing = Ease of Retrieval
- Metadata Controlled Vocabulary
- Content Management
- Knowledge Management

### MANAGEMENT OF COMPUTERIZED DOCUMENTS

- Naming Responsibilities
- Uniform Filing
- Saving of Document
- Backup of Files



## MANAGEMENT OF EMAIL

- Guidelines
- Vulnerability of Information
- 90 Day Policy
- Training Print out records need to be maintained as part of records management procedures

### MANAGEMENT OF INFORMATION

### Security:

- Common Sense Application
- Vital Records
- · Continuity of Operations
- Security of Information



- Information Responsibilities:
- Accuracy on part of creator of document
- Accuracy on filing method and retrieval of document

### MANAGEMENT OF INFORMATION

- Training:
- Naming responsibilities
- Ethical responsibility
- Business continuity practices
- Storage responsibilities

### MANAGEMENT OF INFORMATION

- ISO 15489:
- Clause 4: "Integrate records management into business systems and processes"
- Crucial in meeting goals through best practice in managing information assets

### MANAGEMENT OF INFORMATION

- Sarbanes Oxley:
- Refers to Risk Management
- Not a Document Management regulation have a "domino" effect on document management

### MEASUREMENT OF INFORMATION

- Tape Measure / Ruler / Yardstick
- Databases Production Charts
  - Software Applications:
  - Access
  - Excel
- Quality Management Tools
  - Relationship to Records Management

## MANAGEMENT OF DIGITAL INFORMATION

- Migration of Information
- Software
- Hardware
- Non-electronic methods:
  - Microfilm
  - Imaging



## MANAGEMENT OF IMAGING INFORMATION

- Information = Selective Process
- Indexing = Ease of Retrieval
- Image:
  - For documentation
  - For convenience
  - Not to save space

## FUTURE OF INFORMATION

- Portal = an opening / Business Portal = an electronic opening to a private Internet cache of information / sometimes found as an Extranet.
- Problems with portals: finding the right software company to support the operation / allowing only specific individuals access to the portal.

# FUTURE OF INFORMATION

- Benefits of portals: cuts down on communication costs / cuts down on travel.
- Future of portals: Pratt & Whitney sees a federated portal coming where an aircraft mechanic at American Airlines can access information from Pratt & Whitney on a part for the plane and Boeing for a delivery date on a new model.

## FUTURE OF INFORMATION

- Data Mining similar to Portals, similar to an Internet search. A term is entered and various companies reply with answers.
- Data Warehouses the Internet site for storage of information, releasing earthbound servers from saving unlimited amounts of information.

## FUTURE OF INFORMATION

- New Employment Positions:
  - KO Knowledge Officer
  - –Army leads the way with an AKO (Army Knowledge Online) – useful in the field – allows all Army personnel to know where everyone is located at an exact moment.

- CIO - Chief Information Officer

Mary T. Sullivan, MLS, CRM, CQIA Associated Records and Information Services Phone: 214-675-9598 FAX: 903-527-0326 aris@associatedrecords.com www.associatedrecords.com



## **Session 6**

# **Analysis for Emerging Chemicals**

#### Use of Non-Standard Mass Spectrometric Techniques to Solve Analytical Problems for Emerging Contaminants

#### **Richard Burrows**

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#### ABSTRACT

Analysis of low levels of some emerging contaminants in complex matrices can be challenging, and in some cases requires the use of more advanced mass spectrometric techniques to assure sufficiently low quantitation limits and freedom from matrix interference. This presentation provides some examples, including:

- N-Nitrosodimethylamine by GC/Chemical ionization MS/MS
- Explosives by LC/MS/MS
- PBDEs
- PFOA

Limitations of the standard methods along with performance details of the new methods will be presented.

NEMC 2005 Proceedings



### Use of non-standard Mass Spectrometric Techniques to Solve Analytical Problems for Emerging Contaminants

Richard Burrows Severn Trent Laboratories

#### National Environmental Monitoring Conference July 2005





#### Advantages

- Powerful separation
- Structural information from fragmentation
- Affordable instrumentation

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Universal detector (if the analyte gets to the MS)

#### Disadvantages

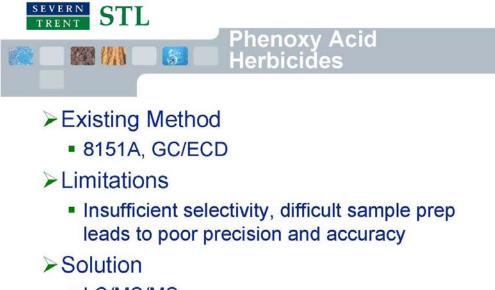
- Most organic compounds will not go through a gas chromatograph
- Some compounds fragment too much
- No selectivity

#### SEVERN TRENT STL

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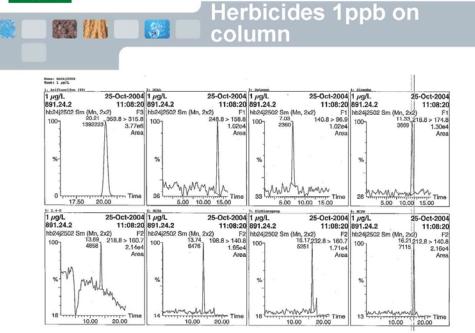
# Desirable method characteristics

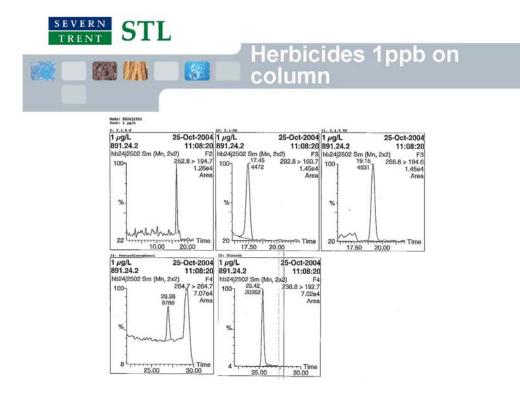
- Linearity predictable instrument response
   Precision reproducibility of results
   Accuracy proximity of results to true value
   Sensitivity low concentration reliably detected
  - Selectivity ability to differentiate compound of interest from interferences
  - Robustness ability of method to work properly in a variety of types of samples



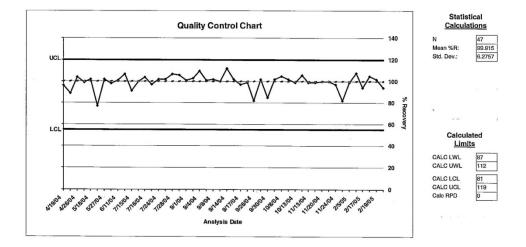
LC/MS/MS



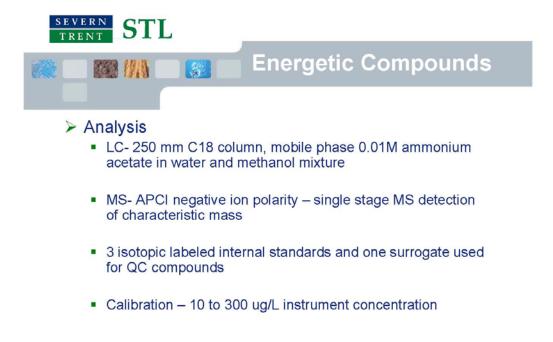














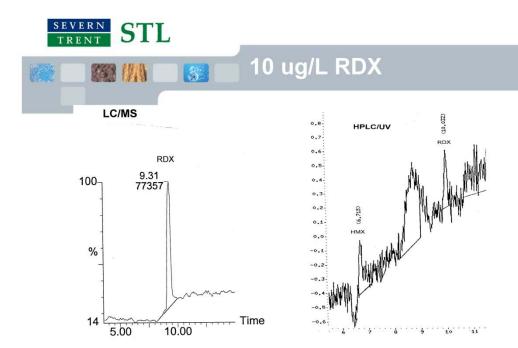
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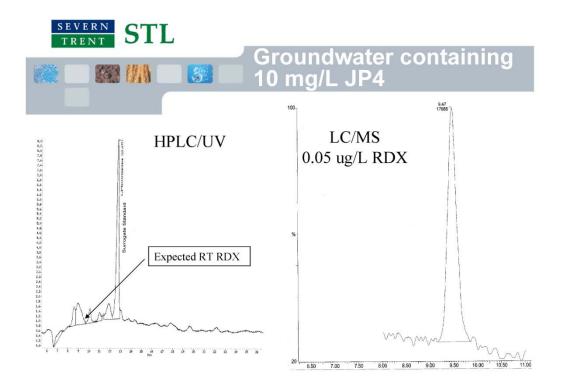
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### Detection limits, LC/MS vs. LC/UV

Analyte	LC/UV MDL	LC/MS MDL	Factor
1,3,5-Trinitrobenzene	0.037	0.015	2
1,3-Dinitrobenzene	0.065	0.008	8
2,4,6-Trinitrotoluene	0.047	0.015	3
2,4-Dinitrotoluene	0.068	0.013	5
2,6-Dinitrotoluene	0.075	0.013	6
2-Amino-4,6-dinitrotoluene	0.058	0.012	5
2-Nitrotoluene	0.065	0.022	3
3-Nitrotoluene	0.034	0.016	2
4-Amino-2,6-dinitrotoluene	0.028	0.015	2
4-Nitrotoluene	0.042	0.014	3
HMX	0.068	0.015	4
Nitrobenzene	0.096	0.020	5
Nitroglycerin	0.374	0.039	10
PETN	0.529	0.016	33
RDX	0.098	0.006	18
Tetryl	0.084	0.010	9

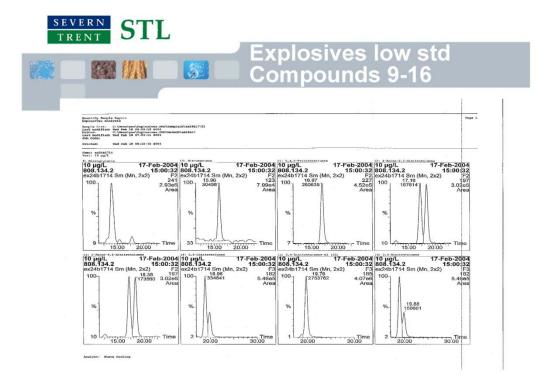




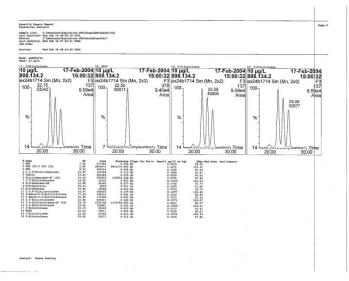


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10 µg/L 308.134 x24b17	. 17	7-Feb-2004 15:00:32 2) F1 355 4.01e6 Area	10 µg/L 808.134	4.2 14 Sm (Mn, 9.90 9955472	17-Feb-2004 15:00:32 2x2) F1 284 1.78e7 Area	808.134		17-Feb-2004 15:00:32 2x2) F1 281 8.17e5 Area	10 µg/L 808.134.2 ex24b1714 §	17-F	eb-2004 15:00:32 F1 213 2.17e5 Area
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%			%			%			%		
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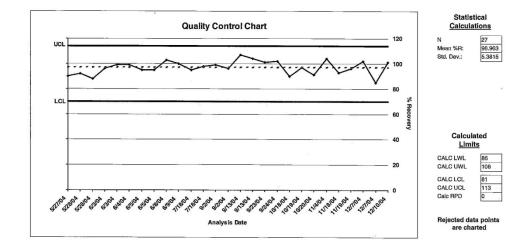




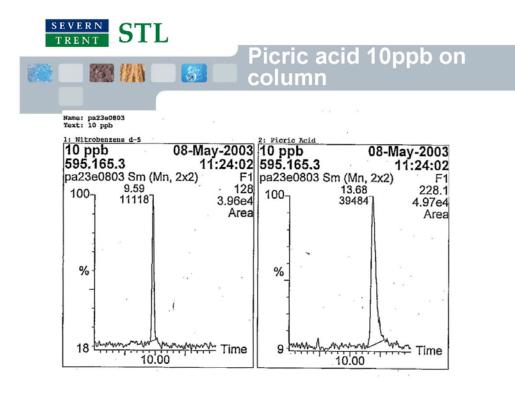


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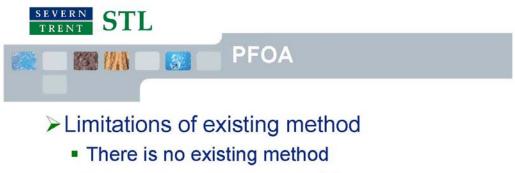








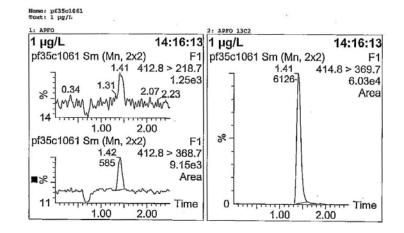
- Used in the manufacture of fluoropolymers nonstick cookware, water and stain resistant finishes, fire resistant finishes
- Persistent in the environment
- Related compounds, Perfluorooctyl sulfonate (PFOS) and perfluorooctanesulfonic acid (PFOSA) can be analyzed using the same method



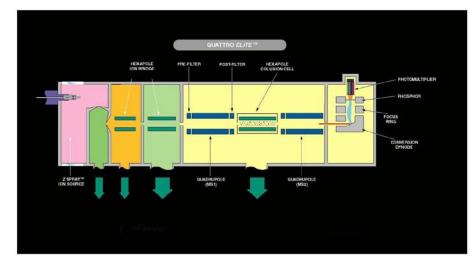
- Not a good compound for GC
- No strong chromophore
- > Solution
  - LC/MS/MS

SEVERN STL
<ul> <li>Extraction</li> <li>Aqueous – SPE extraction using C18 cartridge</li> <li>Solids - 10g sonicated with methanol</li> </ul>
LC – 250 mm C18 column, aqueous formic acid and methanol mobile phase
MS – ESI negative ion MS/MS detection
<ul> <li>C13 labeled PFOA used as an internal standard and PFNA (closely related cmpd) used for a surrogate</li> </ul>
Calibration - 1 to 50 ug/L instrument concentration

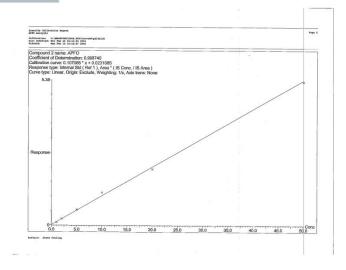




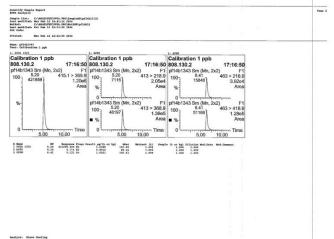


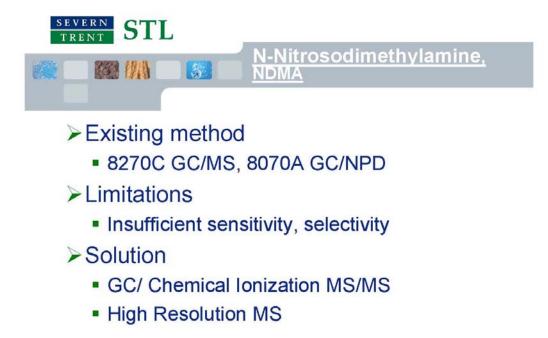






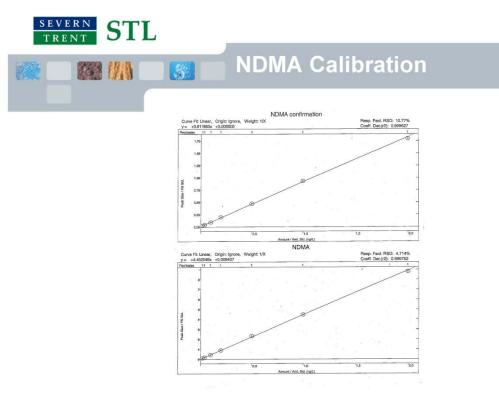




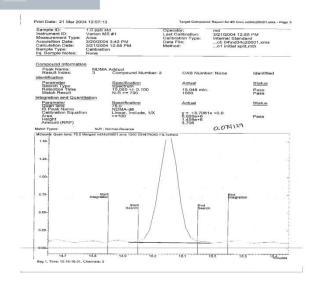


#### SEVERN TRENT STL

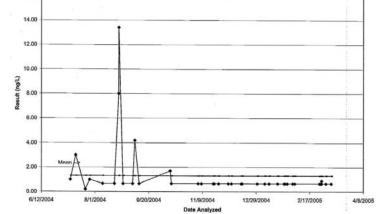
	N-Nitrosodimethylamine, NDMA
A	GC/CI/MS/MS positive ion analysis <ul> <li>CI gas – ammonia</li> </ul>
4	Extraction – CLLE of 1L water with $CH_2CI_2$ , concentration to 1.0 ml final volume
4	624 type capillary column with helium carrier gas
	Cryogenic cool on-column injection
A	NDMA-d6 used for an isotope dilution standard
4	Concentration – 1.0 to 100 ug/L instrument concentration



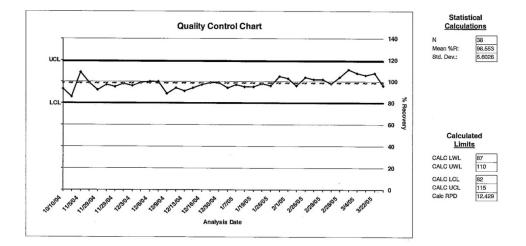


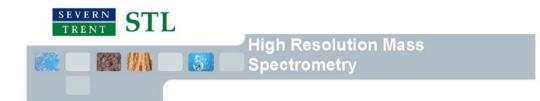




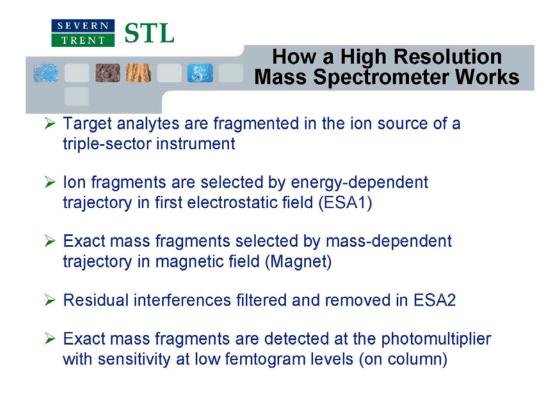


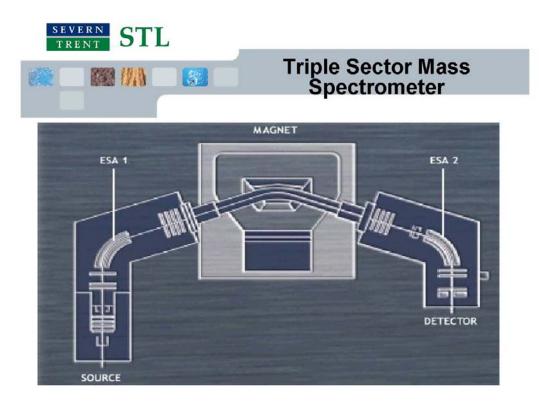






More than just Dioxins!



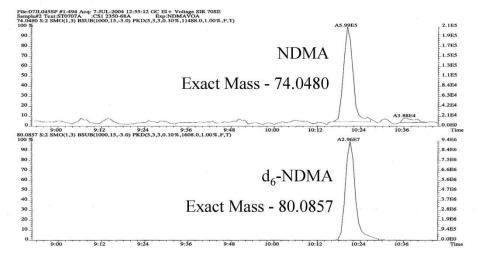


### TRENT STL



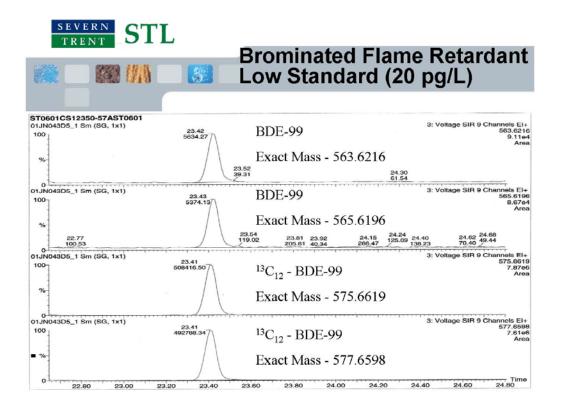
- A target analyte's exact mass is highly characteristic of its identity
- Mass resolution measures the ability of the instrument to isolate and detect a particular exact mass
- Triple sector instruments operate at mass resolution of ~10,000 (high) vs ~100 (low) for quadrupole instruments.
- High Res analyses are nominally 100 times better at filtering interferences than conventional Low Res analysis
- High Res analyses offer improved sensitivity, selectivity, and robustness.

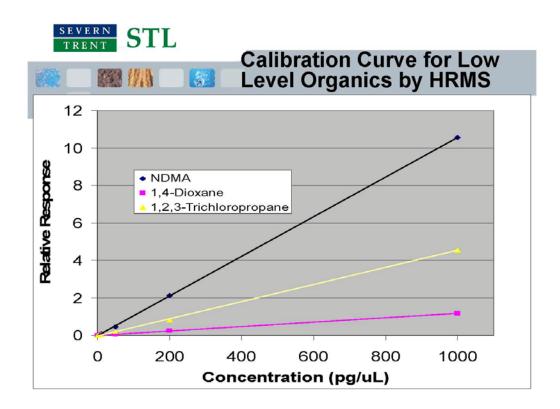






- None, GC/MS amd GC/ECD are possibilities
- Limitations of standard methods
  - Insufficient sensitivity and selectivity
- Solution
  - High Resolution GC/MS





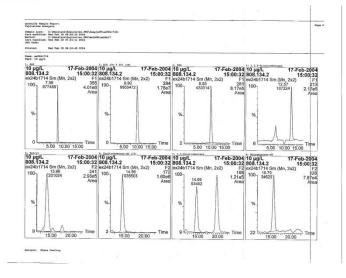




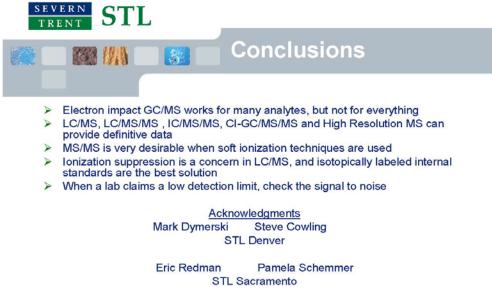
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- Electron impact GC/MS works for many analytes, but not for everything
- LC/MS, LC/MS/MS, IC/MS/MS and CI-GC/MS/MS and High Resolution MS can provide definitive data
- MS/MS is very desirable when soft ionization techniques are used
- Ionization suppression is a concern in LC/MS, and isotopically labeled internal standards are the best solution
- When a lab claims a low detection limit, check the signal to noise!









 Further Information

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# GCxGC-ECD of Chlorination Disinfection Byproducts, Chlorinated Solvents, and Halogenated Pesticides in Drinking Water

#### Jack Cochran\*

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#### Frank Dorman

Restek Corporation 110 Benner Circle Bellefonte, PA 16823 \* presenting author

#### ABSTRACT

Comprehensive two-dimensional GC (GCxGC) is a powerful technique where two independent separations are employed in one analysis for the entire injected sample. In a typical GCxGC setup, a thermal modulator separates press-fitted serial columns of differing phases. One separation is performed on the first column (usually a "boiling point" type), and its effluent is continually focused and "injected" onto the second column (most often a polar or selective phase), where another separation occurs. By keeping the second column short, a series of high-speed chromatograms are generated, and the first column separation is preserved. Separation results can be plotted as a retention plane (column 1 time x column 2 time), also known as a contour plot, or a surface plot, which is a 3-dimensional representation of x (column 1 retention time), y (column 2 retention time), and z (intensity of peaks).

GCxGC produces chromatographic peaks that range from 50 to 500 ms wide. Only a few detectors are available that have the necessary acquisition rates to define peaks this narrow. For example, when mass spectrometry is used, only time-of-flight (TOF) that can record hundreds of spectra per second will work. Of the other detectors used for GCxGC, the flame ionization detector (FID) and electron capture detector (ECD) are most represented in the scientific literature.

The potential of GCxGC-ECD is extremely attractive for environmental analysis since many environmental contaminants are halogenated, including polychlorinated biphenyls (PCBs), chlorinated dioxins and furans, brominated flame retardants, chlorination disinfection byproducts, chlorinated solvents, and pesticides. The sensitivity of the ECD towards halogenated compounds, and its selectivity against those that do not contain halogens, are well known, but both, sensitivity and selectivity, can be enhanced through GCxGC.

A new, commercially available GCxGC-ECD system with a quad-jet, dual-stage modulator was used to demonstrate the potential of the system for the analysis of chlorination disinfection byproducts, chlorinated solvents, and halogenated pesticides (the compounds in US EPA Method 551.1) in drinking water. Separations, instrumental detection limits, and quantification will be discussed.

### GCxGC-ECD of Chlorination Disinfection Byproducts, Chlorinated Solvents, and Halogenated Pesticides in Drinking Water

Jack Cochran LECO Corporation, Las Vegas, NV

Frank Dorman Restek Corporation, Bellefonte, PA

## Outline

- □ Current EPA 551.1 methodology
- GC-TOFMS analysis of standards
  - 551 GC conditions for retention time mapping
- Introduction to GCxGC-ECD
- Preliminary results using GCxGC-ECD
  - Chromatography of standards
  - Analysis of Las Vegas drinking water

## US EPA Method 551.1

- For chlorination disinfection byproducts, chlorinated solvents, and halogenated pesticides in drinking water
- Pentane or MTBE shake extraction
- GC-ECD analysis
  - 30 m x 0.25 mm x 1.0 μm DB-1
  - 30 m x 0.25 mm x 1.0 μm Rtx-1301
- Method detection limits range from ~ 2 to 200 ppt depending on analyte



- 30 m x 0.25 mm x 1.0 µm Rtx-1 (Restek) – He carrier, 0.7 mL/min constant flow
- Split injection of standard
  - 1 μL, 250°C, split ratio 40:1
- Oven program (according to Method 551.1)
  - 15°C, 2°/min, 50° (10 min)
  - 10°/min, 225° (15 min)
  - 10°/min, 260° (30 min)

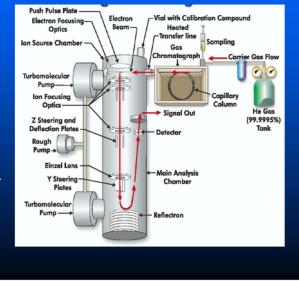
#### Run time = 93.5 min

## **TOFMS Conditions LECO Pegasus III**

- Source temperature: 225°C
- Electron ionization: 70 eV
- Stored mass range: 35 to 450 u
- Acquisition rate: 2 spectra/sec

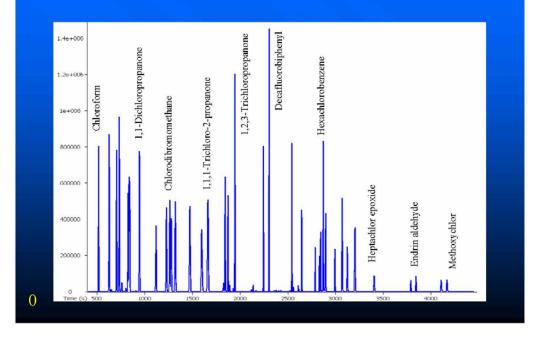
## **LECO Pegasus III GC-TOFMS**

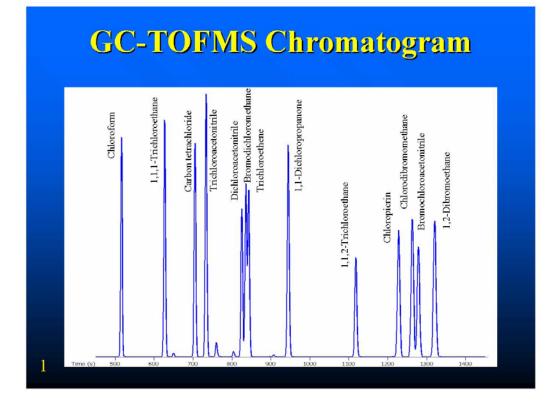
- Make ions
- Pulse them down a flight tube
- Arrival at detector is by time-of-flight
  - Low mass = faster
  - High mass = slower
- Detect ions

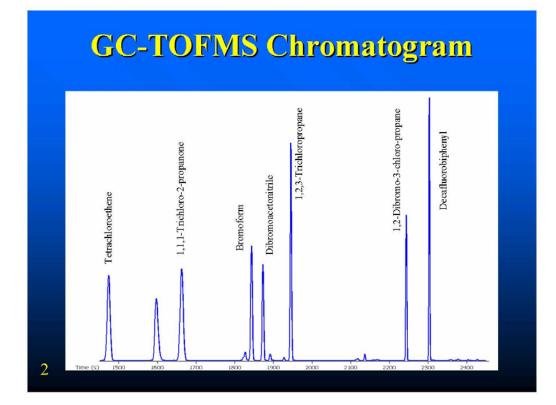


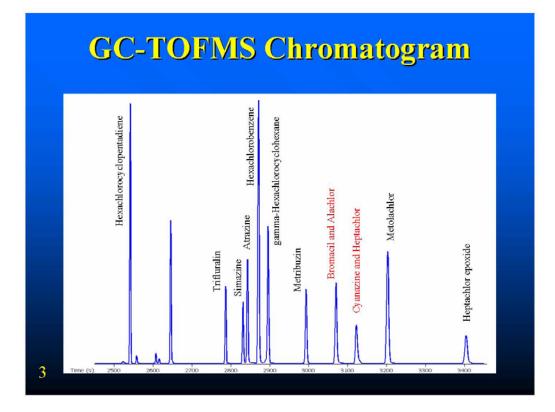
Compound	RTEPA	RT <sub>TOF</sub>		Compound	RT <sub>EPA</sub>	RT <sub>TO</sub>
Chloroform	8.4	8.6		Decafluorobiphenyl	39.5	38.4
1,1,1-Trichloroethane	10.3	10.5		Hexachlorocyclopentadiene	43.9	42.4
Carbon tetrachloride	11.6	11.8		Trifluralin	49.0	46.4
Trichloroacetonitrile	12.0	12.2		Simazine	50.1	47.2
Dichloroacetonitrile	13.5	13.8		Atrazine	50.4	47.4
Bromodichloromethane	13.7	13.9		Hexachlorobenzene	51.1	47.9
Trichloroethene	13.9	14.1		gamma-Hexachlorocyclohexane	51.7	48.3
1,1-Dichloropropanone	15.6	15.7		Metribuzin	54.0	49.9
1,1,2-Trichloroethane	18.6	18.6		Alachlor	55.7	51.2
Chloropierin	20.5	20.5		Bromaeil	55.9	51.2
Chlorodibromomethane	21.0	21.1		Cyanazine	57.0	52.0
Bromochloroacetonitrile	21.3	21.3		Heptachlor	57.2	52.1
1,2-Dibromoethane	22.0	22.0		Metolachlor	59.1	53.4
Tetrachloroethene	24.8	24.6		Heptachlor epoxide	62.5	56.7
1,1,1-Trichloro-2-propanone	27.9	27.7		Endrin	68.0	63.2
Bromoform	31.0	30.7	10	Endrin aldehyde	69.3	64.0
Dibromoacetonitrile	31.5	31.2		Endrín ketone	75.7	68.4
1,2,3-Trichloropropane	32.8	32.4		Methoxychlor	77.0	69.5
1,2-Dibromo-3-chloropropane	38.3	37.4		w.		

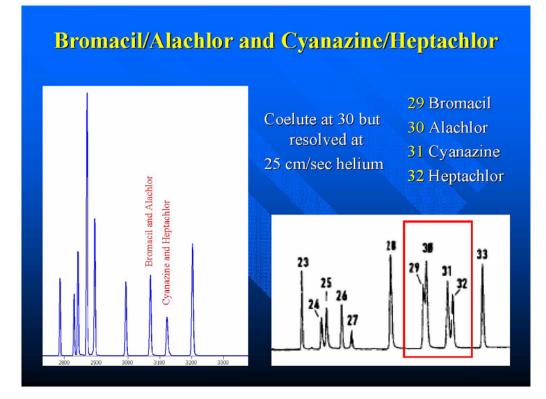
# **GC-TOFMS** Chromatogram

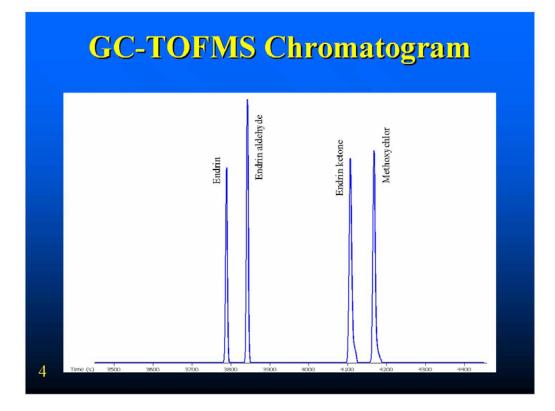


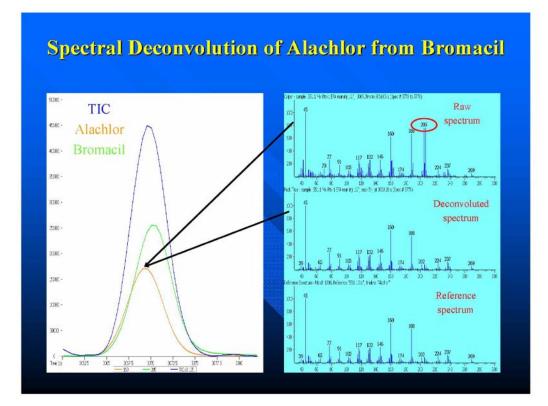


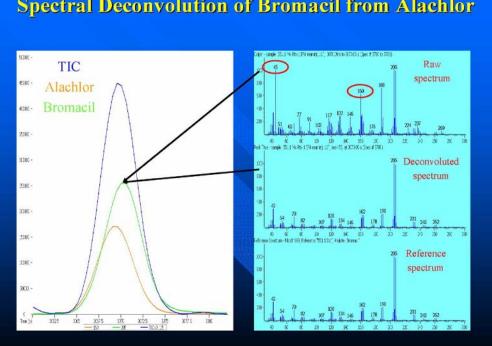


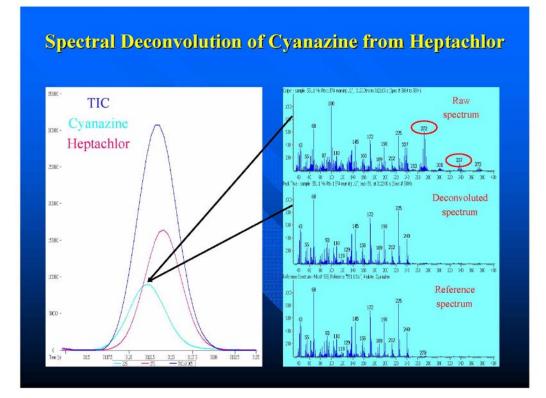










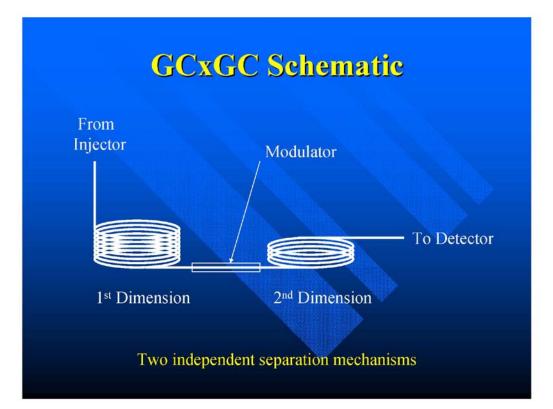


## **Spectral Deconvolution of Bromacil from Alachlor**

## **Typical GCxGC Setup**

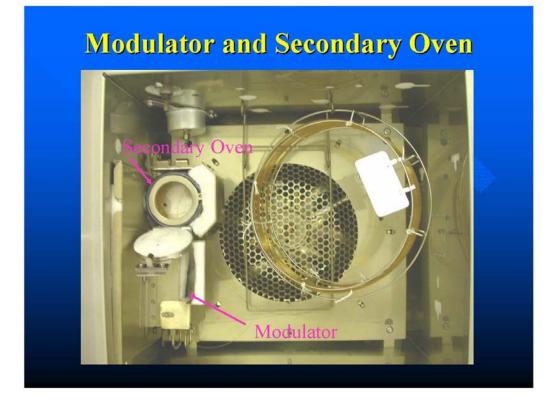
#### ■ Primary column (1<sup>st</sup> dimension)

- Longer, wider bore, thicker film
- Non-polar
- Modulator
  - Thermal in nature
  - Focuses effluent from primary column
  - "Injects" this effluent onto secondary column
- Secondary column (2<sup>nd</sup> dimension)
  - Very short, narrow bore, thinner film
  - Polar or selective











## **GCxGC Columns**

- 30 m x 0.25 mm x 1.0 µm Rtx-1 (Restek)
   100% dimethyl polysiloxane
- 1 m x 0.18 mm x 0.10 μm Rtx-35 (Restek)
   35% diphenyl 65% dimethyl polysiloxane
- Constant flow He at 1.0 mL/min

## Split injection

- 1 μL at 250°C, 10:1 ratio



#### Primary oven

- 30°C (4 min), 2°/min, 50° (10 min)
- 10°/min, 225° (15 min)
- 10°/min, 260° (30 min)

#### Modulator

- Temperature offset: 30°C
- Modulation time: 3 sec

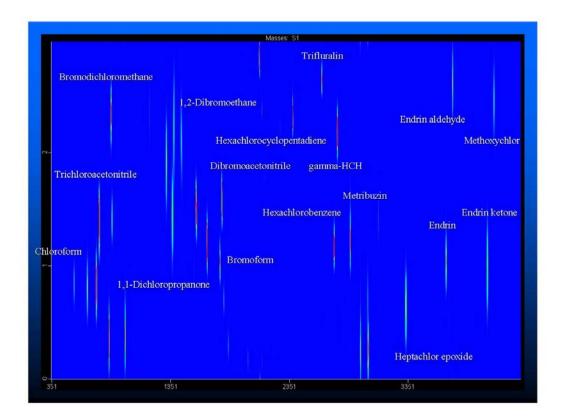
#### Secondary oven

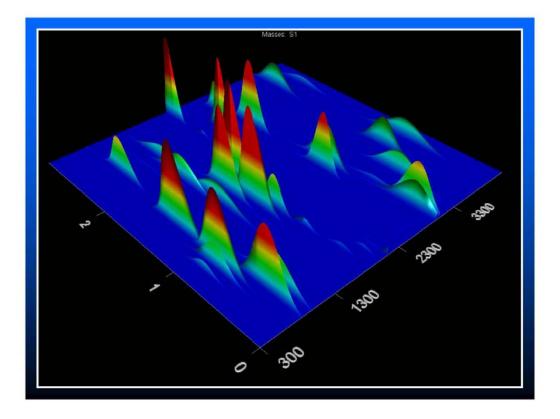
- 5°C offset from primary oven

#### Detector

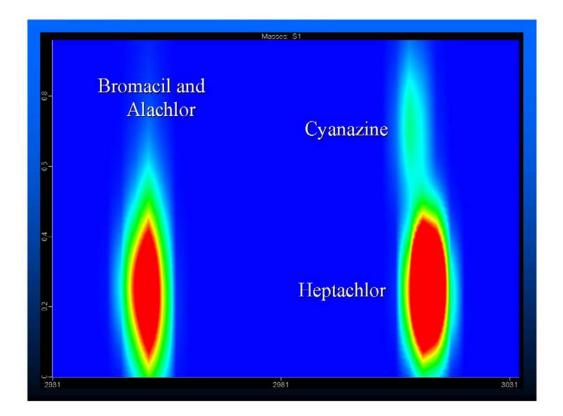
- ECD, 325°C, N<sub>2</sub> makeup gas at 150 mL/min, 50 Hz

Run time = 90 min



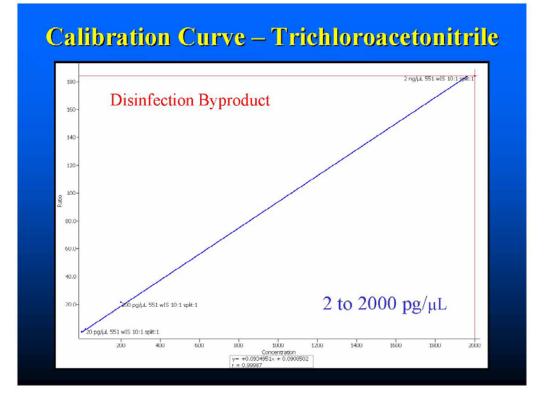


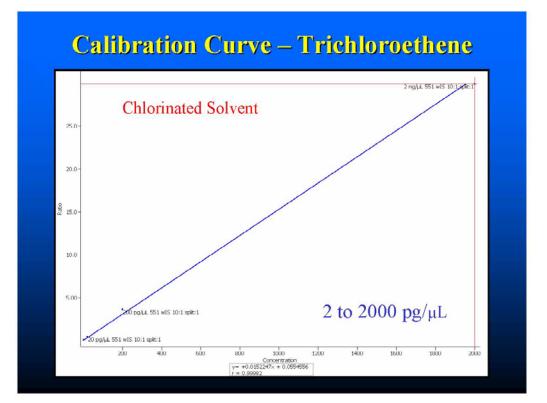
Compound	$-\mathbf{RT}_1$	RT <sub>2</sub>	Compound	RT <sub>1</sub>	RT <sub>2</sub>
Chloroform	540	0.84	1,2-Dibromo-3-chloropropane	2100	2.86
1,1,1-Trichloroethane	651	0.82	Decafluorobiphenyl	2148	1.78
Carbon tetrachloride	729	0.86	Hexachlorocyclopentadiene	2382	2.32
Trichloroacetonitrile	753	1.42	Trifluralin	2628	2.66
Dichloroacetonitrile	834	0.38	Simazine	2682	1.68
Bromodichloromethane	852	2.34	Atrazine	2694	1.52
Trichloroethene	861	1.42	Hexachlorobenzene	2727	1.18
1,1-Dichloropropanone	969	0.38	gamma-Hexachlorocyclohexane	2754	2.20
1,1,2-Trichloroethane	1179	2.34	Metribuzin	2865	1.24
Chloropierin	1317	1.92	Alachlor	2952	0.26
Chlorodibromomethane	1368	1.46	Bromacil	2952	0.26
Bromochloroacetonitrile	1377	2.22	Cyanazine	3009	0.72
1,2-Dibromoethane	1443	2.14	Heptachlor	3012	0.28
Tetrachloroethene	1569	1.56	Metolachlor	3099	1.42
1,1,1-Trichloro-2-propanone	1767	1.26	Heptachlor epoxide	3333	0.28
Bromoform	1782	1.06	Endrin	3669	0.66
Dibromoacetonitrile	1839	1.62	Endrin aldehyde	3726	2.64
1,2,3-Trichloropropane	1839	0.30	Endrin ketone	4020	1.00
p-Bromofluorobenzene	1869	2.98	Methoxychlor	4071	2.46

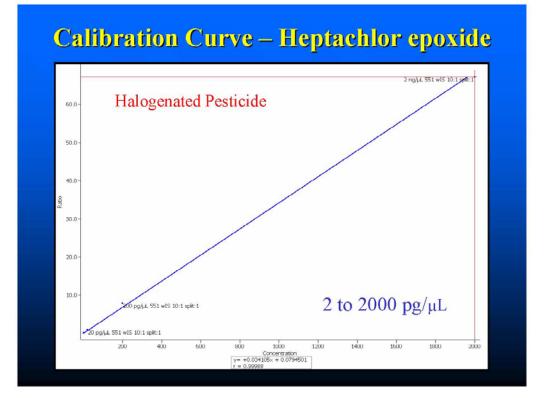


## Calibration and Quantification with GCxGC-ECD

- Analyze standards and sample
  - Internal standard
    - » p-Bromofluorobenzene
- Find peaks and combine slices for peak areas
  - Automatic through software
- Use retention time for identification
- Quantify 551 method analytes against prepared calibration curves







#### GCxGC-ECD Screening Results for Las Vegas Drinking Water

- Pentane extraction
- Only disinfection byproduct and chlorinated solvent results shown here
- Trihalomethane results are similar to previous determinations with SPME GC-TOFMS

Compound	ppb
Chloroform	17
1,1,1-Trichloroethane	0.26
Carbon tetrachloride	0.21
Trichloroacetonitrile	0.11
Dichloroacetonitrile	0.90
Bromodichloromethane	14
Trichloroethene	0.25
1,1-Dichloropropanone	1.3
1,1,2-Trichloroethane	ND
Chloropicrin	ND
Chlorodibromomethane	9.1
Bromochloroacetonitrile	0.63
1,2-Dibromoethane	ND
Tetrachloroethene	0.16
1,1,1-Trichloro-2-propanone	1.4
Bromoform	2.2
Dibromoacetonitrile	0.77
1,2,3-Trichloropropane	0.15
1,2-Dibromo-3-chloropropane	0.29

## Conclusions

- GCxGC-ECD offers selectivity not available with conventional, one-dimensional GC-ECD
  - Always two retention times for each compound
  - Separation of compounds from each other and from matrix interferences
- Calibration and quantification was demonstrated for 551 compounds

## **Acknowledgment**

The LECO thermal modulator was built by LECO under license from Zoex Corporation.

#### Analysis of PCB Congeners by GC-MS-MS As Compared to Aroclor Analysis

#### Ms. Pamela Hamlett\* and David Klein, Ph.D.

Texas Parks & Wildlife Environmental Contaminants Lab 505 Staples Road San Marcos, TX 78666 512-353-3486 pamela.hamlett@tpwd.state.tx.us \* presenting author

#### ABSTRACT

The analysis of legacy pollutant PCBs continues to be of interest. Many organizations have historical data that was compiled as Aroclor fractions or even "Total" Aroclors. We have been working on a novel method to revise this "semi-quantitative" approach. Analytical chemistry works well when specific compounds are measured. When data is multiplied by some "factor" and a "selected" number of analytes are used to represent a larger group, the quantitative analysis becomes less and less accurate. By employing the readily available technique of tandem mass spectrometry we have found a good method to obtain reliable data with acceptable sensitivity.

#### Accelerated Solvent Extraction (ASE) as a Sample Preparation Technique for Polybrominated Diphenylethers (PBDEs) in Environmental Samples

#### Sheldon Henderson\*, Richard Carlson and Bruce Richter

Dionex Corporation 1515 W. 2200 S., Suite A Salt Lake City, Utah 84119 801-972-9292 <u>sheldon.henderson@dionex.com</u> \* presenting author

#### ABSTRACT

Accelerated solvent extraction (ASE) is an innovative approach to liquid-solid extraction. It is accepted under Method 3545A for the extraction of environmental toxins such as PCBs, dioxins, PAHs, and pesticides. This technique uses elevated temperature and pressures to achieve analyte extractions from solid or semi-solid matrices in about 15 minutes and with small volumes of solvents. For example, a 10-g sample can be extracted in about 12 minutes and with about 15 mL of solvent.

There has been increasing concern from scientists and public health officials about polybrominated diphenylethers (PBDEs). PBDEs are part of a class of brominated flame retardants used in the manufacturing of textiles, furniture, polymeric parts in consumer electronics, polyurethane foams, and other polymeric materials. These compounds have been shown to enter the biosystem and have been found in drinking water, animal tissues and human breast milk. They have been found in all animal tissues examined including samples taken from Arctic regions. Their concentration in the environment is increasing annually, and as a result of this increase and concern over possible health effects, their production and use have been banned or soon will be banned in both North America and Europe.

Traditional methods such as Soxhlet have been used to extract samples containing PBDEs. These methods require long periods of time (16 hours) and large volumes of solvent (300 mL). ASE can perform these extractions in short periods of time and with small amounts of solvent. This presentation will discuss the use of ASE for the extraction of several environmental matrices for PBDEs including sediments, fish tissue and human breast milk. Comparisons to traditional methods of extraction will be presented. Accelerated Solvent Extraction (ASE) as a Sample Preparation Technique Polybrominated Diphenylethers in Environmental Samples

S. Henderson, R. Carlson and B. Richter Dionex Corporation, Salt Lake City, Utah



21226

### This Is the Toxic Substance You Can't Avoid; Chemical Residue from Flame Retardants Is Nearly Everywhere

"Created by chemical companies to make hard plastic and polyurethane foam less flammable, polybrominated diphenylethers or PBDEs, are added to computers, TVs, furniture cushions, upholstery textiles, carpet backings, mattresses, cars, buses, aircraft and construction materials."

"The flame retardants have been detected in virtually every person and animal tested, even newborns and fetuses, around the world, including Australia, Arctic Canada and Svalbard, Norway, near the North Pole. Amounts in people and wildlife are doubling in North America every four to six years, a pace unmatched for any contaminant in at least 50 years."

Los Angeles Times, June 20, 2004

#### Introduction

- Polybrominated diphenyl ethers (PBDEs) were developed in the early 1970s
- PBDEs are manufactured as flame retardants for consumer products
- Discovered in European waterways in the 1980s
- Production and use banned by European Union
- Ban to take place in California in 2006
- Toxicity has yet to be determined
  - Evidence suggests it may compromise endocrine or hepatic functions

20083

### **EPA Proposed Rule**

- Tetra-, penta-, hexa-, hepta-, octa-, and nonabromodiphenyl ether
- Manufacturers and importers have to inform EPA at least 90 days before commencing the manufacture or import of any one or more of these compounds on or after January 1, 2005 for any use
- Released December 6, 2004

### Background

- Penta-, octa-, and deca-BDE are most commonly produced
- Found in consumer products
  - Clothing, furniture, plastics
  - Up to 28% by weight in seat cushions
- Disposed in landfills
- Find their way into biosystem
- Accumulation in biosystem is on the rise
- Flame retardants do prevent fire-related deaths

20084

### **Analytical Techniques**

#### ♦ GC/ECD

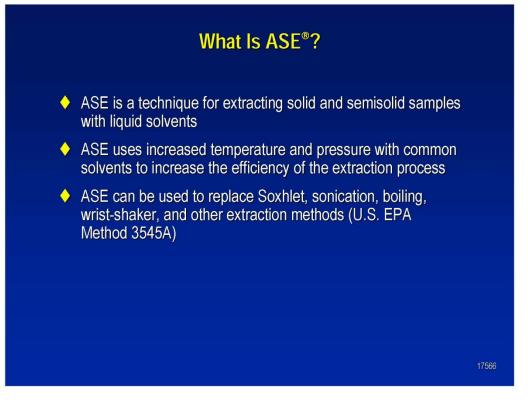
- High-molecular-weight compounds require special high-temperature columns
- Electron capture detector may require second column confirmation

#### GC/MSD or GC/HRMS

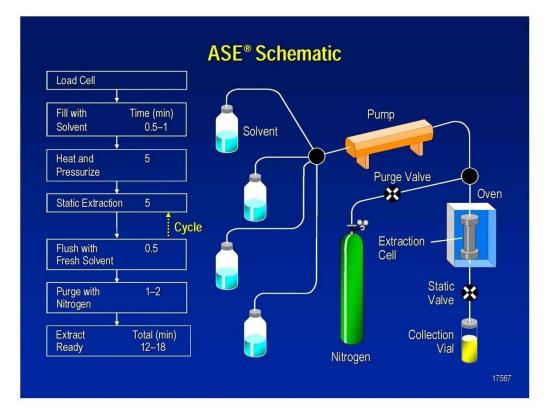
- High-molecular-weight compounds require special columns
- Long run times (approximately 1 h)
- High-temperature column
  - 380 °C temperature limit
  - Separates 20 PBDE congeners, including DBDE 209

### Sample Preparation Techniques for Solids

- Methods for extracting polybrominated diphenyl ethers
  - Sonication
  - Soxhlet
  - Shake or soak
  - Accelerated Solvent Extraction (ASE<sup>®</sup>, PLE, or PFE)
  - Silica gel cleanup may be necessary for environmental samples
    - » SW 846 8290 procedure







## **ASE® 200**

- Automated extraction of 24 sample cells
- Sample cell sizes of 1-, 5-, 11-, 22-, and 33-mL internal volume
- Typical extraction times of 15 min per sample
- Extraction solvent volumes of 10–50 mL

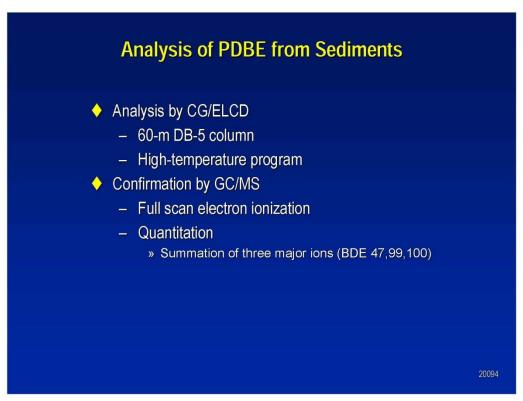


A Faster, More Efficient Way of Sample Preparation



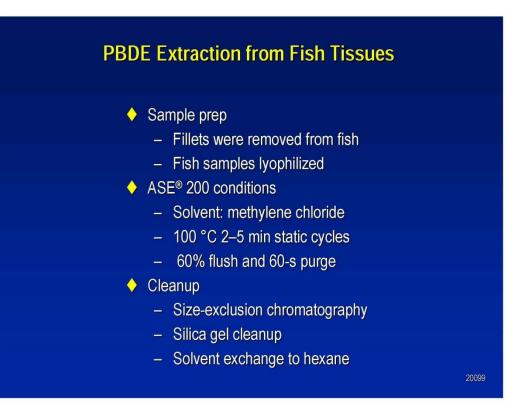
### **PBDE from Sediments**

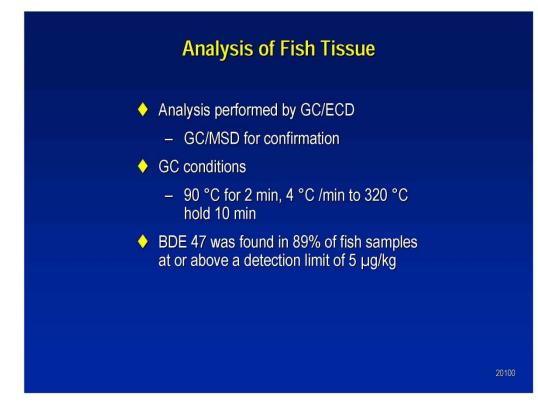
- Samples were dried prior to extraction
- ASE<sup>®</sup> 200 conditions
  - Solvent: Methylene chloride
  - 100 °C 2-5 min static cycles
  - 60% flush 60-s purge
- Solvent exchanged to hexane
- Cleanup
  - GPC and silica gel

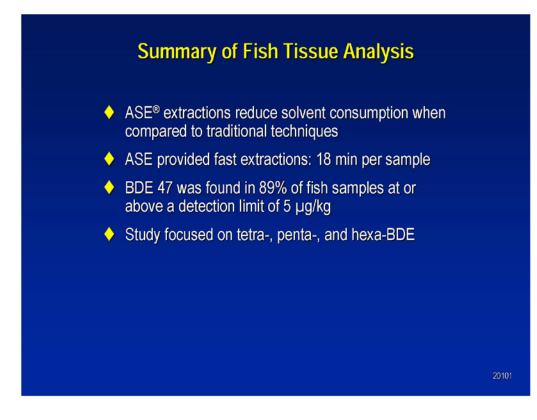


### Summary of PBDE from Sediments

- Fast extractions using ASE<sup>®</sup>: 18 min per sample
- Detection limit of 0.5 µg/Kg
- BDE 47 detected in 22% of samples
- Bottom feeding fish (catfish) had a PBDE profile similar to river sediment

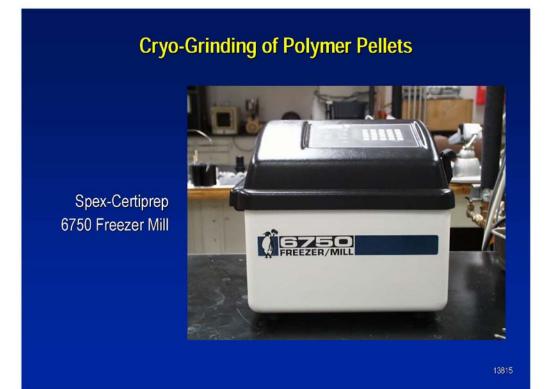






## **PBDE from Polymer**

- Grind pellets to powder
- Add sample to thimble then place in stainless steel cell
- ♦ ASE<sup>®</sup> conditions
  - Solvent: Isopropanol or THF
  - 80 °C, two 10-min cycles
  - 70% flush 60-s purge
- ♦ HPLC/UV or GC-MS



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## **Cryo-Grinding of Polymer Pellets**

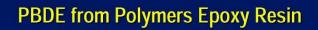


Grinding greatly enhances surface area and extraction efficiency

> Essential for fast, quantitative extraction

## Analysis of Polymer for PBDE

Matrix	Compound	ASE <sup>®</sup> Recovery (% of Soxhlet)
ABS	Polybromodiphenylether	81.3
SB	Polybromodiphenylether	76.3
SB	Polybrominated Biphenyl	76.2





- Solvent: Toluene
- 120 °C, two 5-min cycles
- 100% flush 60-s purge
- ♦ GC-MS analysis

## PentaBDEs in Polymers ASE<sup>®</sup> Comparison with Soxhlet

	BDE 100	BDE 99	BDE 85
Soxhlet			
Avg. (Wt%)	0.21	0.87	0.04
RSD (%)	7.58	4.74	7.75
ASE			
Avg. (Wt%)	0.20	0.94	0.05
RSD (%)	2.76	6.73	3.04
% Soxhlet	95.2	108	125

### Summary of Polymer Extraction

- Recovery of PBDE is similar to Soxhlet
- Reduced solvent consumption
  - 30 mL versus 70 mL
- Faster extraction time
  - 25 min versus 3 h or more

#### PBDE from Human Breast Milk

- ♦ Samples are freeze dried
  - 3.50 g mixed with Ottawa sand in a 22-mL ASE<sup>®</sup> cell with filters
- ASE conditions
  - Solvent: hexane, methylene chloride, methanol 5:2:1
  - 80 °C 3–5 min extraction cycles
  - 60% flush and 240-s purge
- Concentration by evaporation
- Extract cleanup
  - Silica gel and GPC

## Analysis of Human Breast Milk

Human Breast Milk Sample	BDE 77	BDE 153	BDE 209
Mean Recovery as a % of Spike	79.9	76.8	85.7

20092

## Summary Human Breast Milk Extraction GC/HRMS

- ♦ AOAC method is liquid–liquid
  - Sep funnels will clog
- Comparison of liquid milk and dried milk samples
  - No difference when extracted by ASE®
- ♦ ASE extraction
  - 25 min
  - 30 mL of solvent
  - UV protection is important
    - » Use amber collection vials

17574

## Conclusion

- Sample preparation
  - ASE<sup>®</sup> is a fast and effective tool for extraction of PBDE
  - ASE provides automation for sample preparation
- ♦ Analysis
  - GC/MS (1000 amu)
  - GC/ECD
  - Requires high-temperature column





## Acknowledgements

- Dr. Jian Wen, Dr. Arthur Holden, and Margaret Sharp from California EPA, Berkeley, CA
- Mark Olson, Underwriters Laboratories, Northbrook, IL

20104

# RapidMS Chromatography and Tandem Mass Spectrometry for Trace Determination of Brominated Flame Retardants

#### **Robert Brittian**

Varian, Inc. 2700 Mitchell Drive Walnut Creek, CA 94598 925-942-4857 Presented by: Ed George, <u>ed.george@varianinc.com</u>

### ABSTRACT

Polybrominated diphenyl ethers (PDBEs) have been widely used since the 1960s as flame retardants in a variety of products such as clothing, furniture, carpets, electronic components, and plastics. At every stage, from production, to use, to disposal or recycling, PDBEs are released into the air, water, and soil. These compounds will bioaccumulate in fatty tissues and are known to have endocrine disrupting effects, particularly affecting thyroid function. Production of some commercial mixes of PDBEs has halted, but worldwide production of these reported in 1999 was 67,000 metric tons.

A sensitive and selective method for the detection of PDBEs in environmental and food samples will be described. It involves the use of a column known as Rapid-MS combined with tandem mass spectrometry. Rapid-MS vacuum chromatography accomplishes efficient transfer of even the heaviest deca-BDE into the mass spectrometer while maintaining excellent chromatographic resolution in a very short run time. Traditional Selected Ion Monitoring (SIM) methods are very sensitive, but lack the ability to separate the target compounds from complex matrices. Tandem ion trap mass spectrometry ensures that complex matrix interferences are eliminated providing accurate quantitation at sub-pg detection limits.

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#### RapidMS Chromatography and Tandem Mass Spectrometry for Trace Determination of Brominated Fire Retardants

Robert D. Brittain

Varian, Inc.

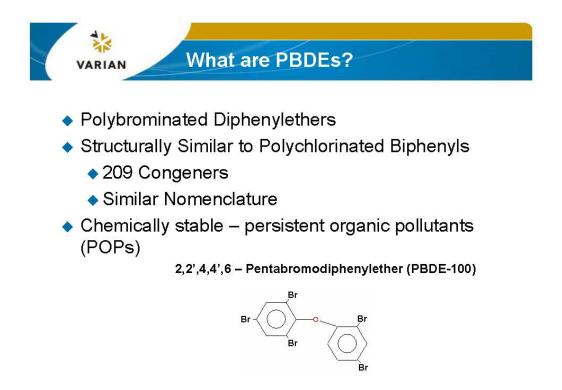
21<sup>st</sup> Annual National Environmental Monitoring Conference Washington, DC July 25 – 29, 2005

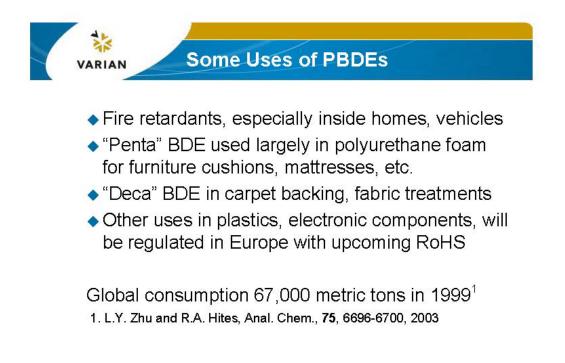


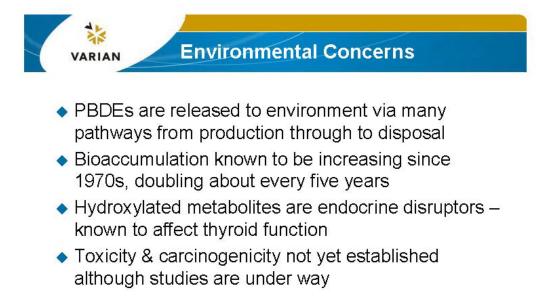
- PBDEs as Persistent Organic Pollutants
  - Polybrominated Diphenylethers
  - Many congeners: mono- to deca-BDE
- The Chromatographic Challenge
  - High Molecular Weights (> 950u)
  - Thermal Instability

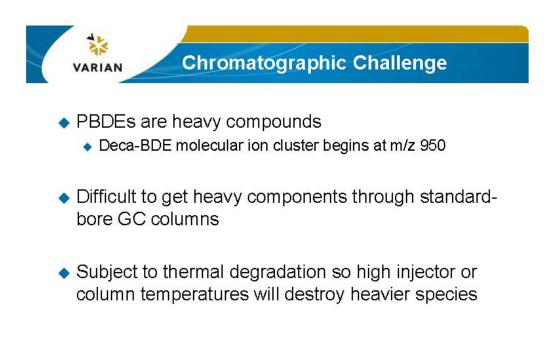
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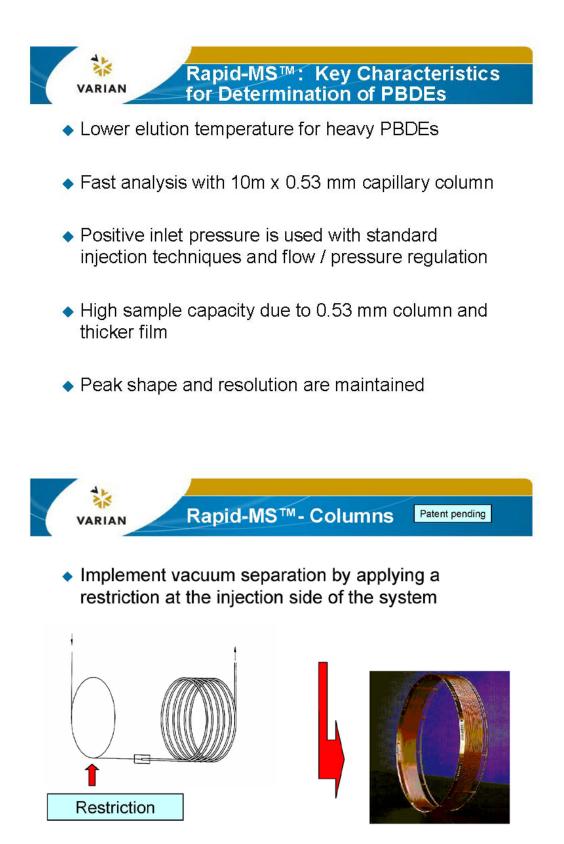
- Vacuum Chromatography for better separations
- Sensitivity/Selectivity with MS/MS Detection
- Calibration and Detection Limits
- Sample Analyses

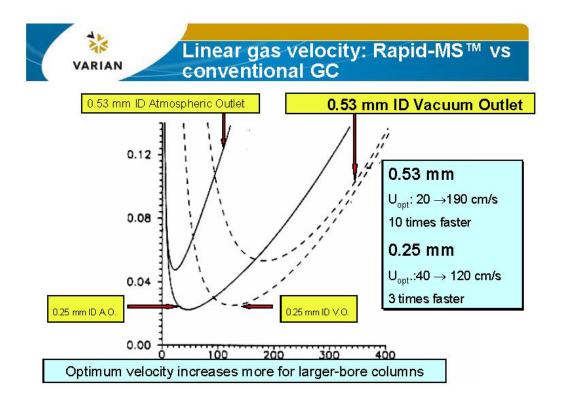




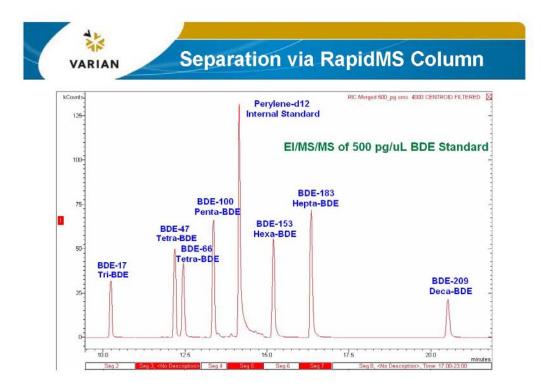






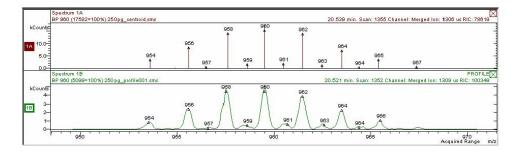


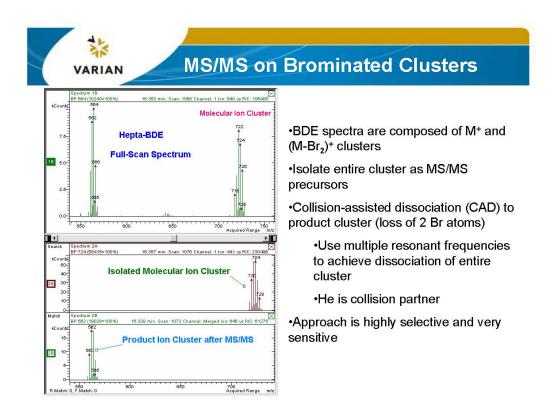






#### Shown in Centroid mode (Above) and Profile mode (Below)





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## External Ionization – 4000 GC/MS

- 2 μL pressure-pulsed, splitless injections (8400 AutoSampler)
- Siltek-coated, fritted liner, 1177 Injector @ 260C
- Rapid MS Column CP-Sil8 (10m x 0.53mm ID x 0.25df)
  - 80C (hold 1.5min) // 250C @ 12C/min // 300C @ 25C/min // Hold 5.33 min (Total Run Time 23.00 min)
- MS/MS
  - External Ionization Configuration 2.5 ml/min He damping
  - Temperatures: Transfer Line 280C/Source 225C/Ion Trap 100C
  - Electron Ionization 70eV Pulsed ON only during ionization
  - Multiplier 10<sup>5</sup> gain + 300V
  - AGC Target 5000
  - Filament Delay 9.00 min
  - MS/MS Time-Programmed for each BDE component
  - I.S. Perylene-d12 acquired in SIS (263-265u)

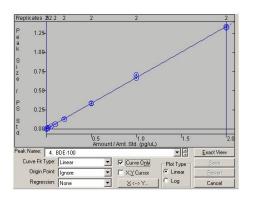


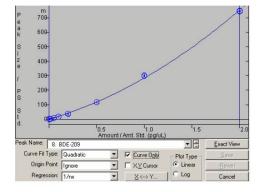
## Calibration Levels (pg/µL)

Calibration Level	Deca-BDE pg/µl	All Others BDEs pg/µا
1	2	1
2	4	2
3	10	5
4	20	10
5	50	25
6	100	50
7	200	100
8	500	250
9	1000	500
10	2000	1000

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## **Representative Calibration Plots**







Range 1 – 1000 pg/µl

Deca-BDE Response was Quadratic Range 2 – 2000 pg/µl

VARIAN		Calibration D (Deca-BDE fr		Contraction and the second
Compound	#Br	Corr. Coef.	%RSD	Type Fit
BDE-17	3	0.9986	8.77	Linear
BDE-47	4	0.9997	7.31	Linear
BDE-66	4	0.9994	8.79	Linear
BDE-100	5	0.9994	6.27	Linear
BDE-153	6	0.9986	16.15	Quadratic
BDE-183	7	0.9980	15.47	Quadratic
BDE-209	10	0.9993	42.81	Quadratic

VARIAN

## **Method Detection Limits**

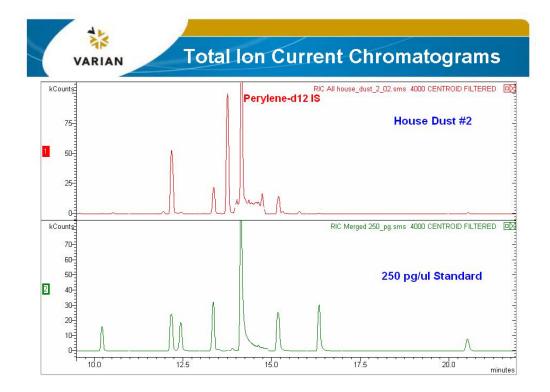
Compound	MDL (pg/uL)
BDE-17	0.309
BDE-47	0.225
BDE-66	0.165
BDE-100	0.207
BDE-153	0.233
BDE-183	0.263
BDE-209	0.764

Based on 10 replicates @ 2 pg/uL

MDL =  $\sigma$  x {Student's t(99% conf.)}



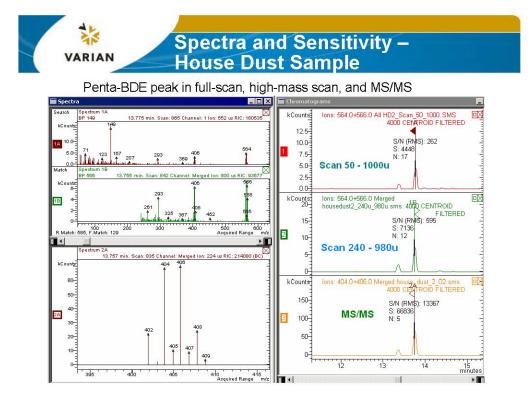
- Dust removed from vacuum cleaner bags
  - Two different houses
  - First had hardwood floors, second was carpeted
- Sonicate 1 g dust in 10 ml hexane 5 min
- Filter
- Add perylene-d12 Internal Standard @ 500 pg/μl
- Run samples in EI/MS/MS
- Run second dust sample in full scan (50 1000u) and high-mass scan range (240 – 980u)



Comp	onent	House Dust #1	House Dust #2
		ng/g(dust)	ng/g(dust)
BDE-17	(Tri)	Not Found	30
BDE-47	(Tetra)	1961	4326
BDE-66	(Tetra)	157	117
BDE-100	(Penta)	616	1318
BDE-153	(Hexa)	315	1052
BDE-183	(Hepta)	Not Found	46
BDE-209	(Deca)	473	206

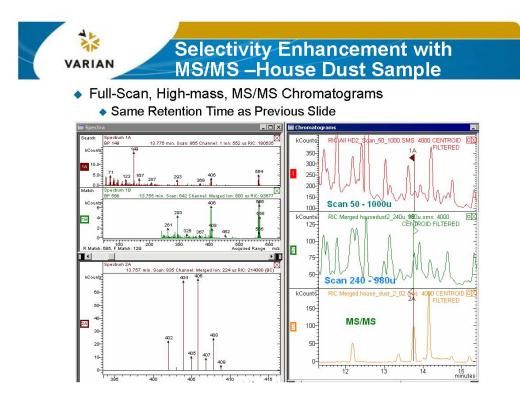
Hardwood Floors

Carpeted



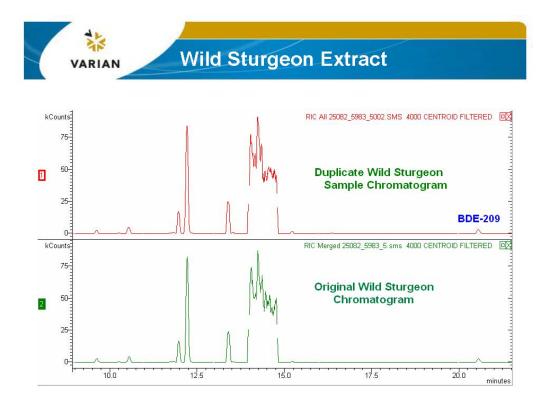
375

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VARIAN Fish Tissue Sample

- Wild sturgeon extract received from nearby lab
- Run earlier on magnetic sector instrument
- Exact volume not available. Estimated that 10.8g concentrated to ~50µl
- Volume too low to estimate correct IS addition, so quantitation based on external standard calibration
- Quality of external standard curves comparable to those for internal standard



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## Wild Sturgeon Results (pg/g Fish Tissue)

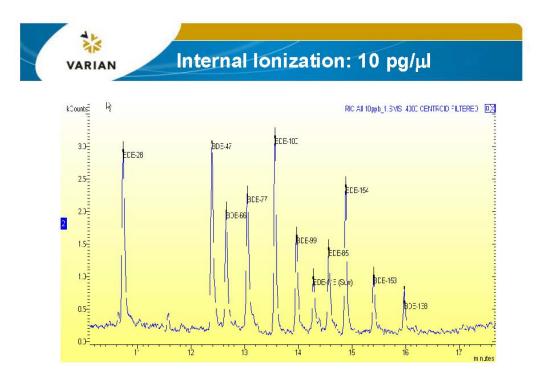
Compound	Magnetic Sector	4000 MS Ion Trap - #1	4000 MS Ion Trap - #2
BDE-17	30	57	36
BDE-47	3950	3919	3893
BDE-66	45	n.d.*	47
BDE-100	651	881	912
BDE-153	75	46	89
BDE-183	22	14	18
BDE-209	0	118	108
Total BDEs	6668**	5035	5103

\* - Interference by neighboring tetra-BDE

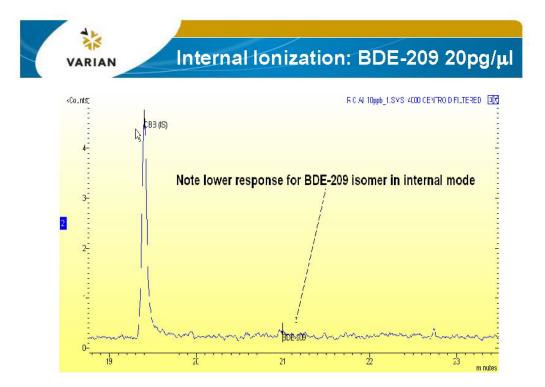
\*\* - More congeners calibrated for sector instrument



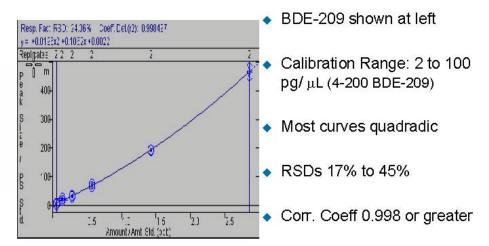
- 2 µL pressure-pulsed, splitless injections (8400 AutoSampler)
- 1177 Injector @ 250C
- Rapid MS Column CP-Sil8 (10m x 0.53mm ID x 0.25df)
  - 80C (hold 1.5min) // 250C @ 12C/min // 300C @ 25C/min // Hold 6.33 min (Total Run Time 24.00 min) Flow Rate = 2 ml/min
- MS/MS
  - Internal Ionization Configuration
  - Temperatures: Transfer Line 280C/Ion Trap 210C/Manifold 60C
  - Multiplier 10<sup>5</sup> gain + 300V
  - ◆ AGC Target 5000
  - Filament Delay 8.00 min
  - MS/MS Time-Programmed for each BDE component
  - I.S. Decabromobiphenyl (DBB)



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## Internal Ionization: 8 reps @ 5pg/ μL BDE-209 @ 10 pg/ μL

13			
Analyte	Ave	RSD (%)	MDL
BDE-28	4.869	4.8	0.703
BDE-47	4.972	3.8	0.560
BDE-66	4.804	5.7	0.816
BDE-77	4.814	4.8	0.692
BDE-100	4.837	5.2	0.749
BDE-99	4.714	9.2	1.305
BDE-85	4.036	12.2	1.479
BDE-154	4.821	6.3	0.915
BDE-153	4.015	9.0	1.079
BDE-138	3.526	12.4	1.309
BDE-209	9.803	17.8	5.218

VARIAN

## Conclusions

- Concepts reduced to practice in PBDE analysis:
  - Vacuum chromatography practical for Deca-BDE
  - ◆ EI/MS/MS with 10<sup>3</sup> practical quantitation range
  - PBDEs can be determined in real samples with minimal cleanup
- Internal vs External Ionization
  - External more sensitive, less noise
  - Better RSDs and Linearity in external for most compounds
  - Future project: Develop MRM for all congeners

# **Session 7**

# Analysis in Perchlorate Analysis

## **DoD Handbook for Perchlorate Sampling and Testing**

#### Mr. Fred McLean\* and Mr. William Ingersoll

U.S Navy Laboratory Quality & Accreditation Office 1661 Redbank Road Goose Creek, SC 29445 Phone: (843) 764-7337 mcleanfs@navsea.navy.mil \* presenting author

## ABSTRACT

The Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) is working towards a unified approach for all Components in the area of perchlorate sampling and testing. This has lead to the development of the *Handbook for Perchlorate Sampling and Testing*.

The Handbook provides instruction on planning and execution for perchlorate data generation. A critical part of the process is proper project scoping and development of Conceptual Site Models. Identification of exposure pathways can determine the extent of sampling and testing required. The handbook gives guidance on development of project quality objectives, sample design, collection, and selection of analytical services. New technologies for the analysis of perchlorate are discussed. The pros and cons of different analytical methods from EPA are explained.

The goal of the Handbook is to inform Components of the available options for sampling and testing for perchlorate. With the proper information, a Component can make decisions that provides the required data quality.

## Method 6850 - Determination of Perchlorate Using High Performance Liquid Chromatography/Mass Spectrometry (LC/MS)

#### Robert P. Di Rienzo\*

DataChem Laboratories, Inc. 960 West LeVoy Drive Salt Lake City, UT 84123 (801) 266-7700 dirienzo@datachem.com

#### Kham Lin

K'(Prime) Technologies, Inc.

### **USEPA OSW**

Inorganics Method Development \* presenting author

## ABSTRACT

A new method for the detection and confirmation of perchlorate utilizes liquid chromatography to separate perchlorate from interferences and mass spectrometry to confirm and quantify. DataChem Laboratories, Inc. in conjunction with K'(Prime) Technologies, Inc. has developed this new liquid chromatography mass spectrometry method for the detection and confirmation of perchlorate in drinking water, ground water, saline water, soil and biota samples. The USEPA is currently evaluating method SW846 Method 6850, with an inter-laboratory validation study.

Method 6850 for the determination of perchlorate uses a newly developed, commercially available, liquid chromatography column developed by K'(Prime) Technologies, Inc. By using this column in an Agilent 1100 LC/MSD system, the technique separates perchlorate from known interferences in difficult matrices and can detect perchlorate in matrices that are unacceptable for analysis by drinking water methods.

Mass spectrometry is used to monitor perchlorate at mass 83, which is achieved by the partial fragmentation of perchlorate to remove an oxygen atom. Confirmation of perchlorate is obtained not only by retention time and mass but by the isotopic ratio of mass 83 and 85 and an internal standard of Oxygen-18 labeled perchlorate.

Method 6850 can easily quantify perchlorate at 0.2 ppb in environmental sample matrices, uses simple determinative techniques available to current LC/MS technologies and does not require systematic pretreatment of samples prior to analysis. The analysis is accomplished in under thirteen minutes and can process up to 20 samples in an eight hour sequence with all appropriate quality control.

Inadequacies of EPA drinking water concerning matrix interference, high dissolved solids and conductivity are eliminated and confirmation of perchlorate is accomplished with method 6850.

# Analysis of Perchlorate by IC/MS/MS and Development of Method 6860

#### **Richard Burrows**

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### ABSTRACT

This paper will describe the development and performance of an IC/MS/MS method for analysis of perchlorate. We will cover instrumental conditions and critical QA parameters to ensure that the method is performing effectively. The current status of SW-846 method 6860 will be presented.

# A Discussion of Separation and MS Detection for the Determination of Perchlorate in Real World Samples

### R. Slingsby<sup>\*1</sup>, C. Saini<sup>1</sup>, C. Pohl<sup>1</sup> and H. El Aribi<sup>2</sup>

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### ABSTRACT

In the last several years, IC-MS and IC-MS/MS technologies have been developed and successfully applied to the determination of perchlorate in drinking water and various food types. Results from the analyses of many real world samples have been the topic of great national and international interest. As these technologies are applied to more complex sample matrices including well water, waste water, various agricultural crops and finished food products, the demands for robust and adaptable methodologies are increasing. In a sense the "toolbox" available to the analyst must become larger and more powerful as interest in more complex sample types increases.

Ion exchange resins have been used successfully for a number of years to selectively retain perchlorate in relation to common matrix ions including chloride, carbonate and sulfate. The IonPac® AS16 resin has an ion exchange latex agglomerated to a highly cross linked, aromatic substrate resin particle. This phase selectively retains polarizable anions including perchlorate while also providing excellent peak shape in aqueous eluents. As the perchlorate analysis using this column was applied to well water, aromatic sulfonate contaminants were found to interfere with the elution of perchlorate on the AS16 ion exchange phase. New synthesis technology was recently developed that provides novel selectivity for perchlorate and the aromatic sulfonates. The IonPac AS20 stationary phase is based on the new Hyperbranched Condensation Graft Technology, which combines the excellent peak shape benefits of Dionex latex technology with the selectivity benefits of graft technology. The AS20 anion exchange polymer is built with an aliphatic backbone and therefore, provides an orthogonal selectivity to the AS16 column. These 2 columns expand the analyst's "toolbox" by providing complementary separation information even for very complex sample types.

Multiple reaction monitoring (MRM) is the standard technique for quantitative LC-MS/MS analysis. In general, the LODs/LOQs, precision, and accuracy achieved with this technique are difficult to match. In this paper, we will discuss the chemistry of the separations, the coupling to MS and MS/MS detection using on-line matrix diversion and an isotope-enriched perchlorate internal standard. The analytical statistics (including the <sup>37</sup>Cl/<sup>35</sup>Cl ratios) obtained for drinking water, waste water, soil extracts and several food samples will be presented.

## Perchlorate in Water – A Comparison of Methods 314.0 and 332.0

Scott McLean\*, James F. Occhialini, Arin Jones and James Todaro

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## ABSTRACT

Perchlorate is a natural and man-made chemical that has been used as an oxidizer in rocket fuel, munitions and fireworks since the 1950s. It is known to disrupt thyroid function by inhibiting iodine uptake, thereby inhibiting the production of key thyroid hormones. It is very soluble in water and therefore highly mobile. Perchlorate has been detected in drinking water supply wells in several MA communities. In 2004, a drinking water MCL of 1 ppb was proposed by MADEP, consistent with the MCL proposed by EPA in 2002. While the CADHS set a notification limit of 6 ppb, NAS recently concluded that a level roughly equivalent to 20 ppb might be more appropriate. The current accepted method for low level analysis, EPA 314.0, utilizes an Ion Chromatograph fitted with a conductivity detector and a suppressor to reduce interference from background contaminants; however this method encounters problems in the presence of elevated sample conductivity. An alternative method, EPA 332.0, has been developed to address these problems. Method 332.0 utilizes an IC fitted with an MS or MS/MS. The MS technology allows for the detection of perchlorate to sub ppb concentrations even in the presence of high concentrations of interferents. In this paper the authors present method qualification data as well as real world sample data from both methods. Comparison of real world data from samples with high conductivity will demonstrate the capability of Method 332.0 to accurately and precisely quantitate perchlorate at or below the current draft MCLs.

## Trace Level Determination of Perchlorate in Soils and Fertilizers by Tandem Suppressed Conductivity and Mass Spectroscopy

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## ABSTRACT

Perchlorate salts are being used as rocket propellants, in fireworks and in electroplating industry. Currently, perchlorate is being monitored under USEPA Unregulated Contaminant Monitoring Rule (UCMR-1) at 4.0 parts per billion (USEPA method 314.0). Recently, it is believed by the scientific community that perchlorate hinders the iodine absorption ability of the thyroid gland posing higher health risk for the public. Perchlorate contaminated fertilizers and naturally occurring perchlorate in "Chilean Fertilizers" can leach perchlorate into the soils, ground water, surface water and irrigational waters, which in turn contaminate crops of vegetables and fruits. It is critical to identify and quantify levels of perchlorate contamination in soils, fertilizers and waters. This presentation demonstrates use of conventional ion chromatography conductivity detection and mass spectrometer in tandem. Benefits of mass spectrometer will be discussed.

## **DoD Handbook for Perchlorate Sampling and Testing**

#### Mr. Fred McLean\* and Mr. William Ingersoll

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## ABSTRACT

The Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) is working towards a unified approach for all Components in the area of perchlorate sampling and testing. This has lead to the development of the *Handbook for Perchlorate Sampling and Testing*.

The Handbook provides instruction on planning and execution for perchlorate data generation. A critical part of the process is proper project scoping and development of Conceptual Site Models. Identification of exposure pathways can determine the extent of sampling and testing required. The handbook gives guidance on development of project quality objectives, sample design, collection, and selection of analytical services. New technologies for the analysis of perchlorate are discussed. The pros and cons of different analytical methods from EPA are explained.

The goal of the Handbook is to inform Components of the available options for sampling and testing for perchlorate. With the proper information, a Component can make decisions that provides the required data quality.

**Session 8** 

# Innovative Techniques for Environmental Measurements and Monitoring

# Case Studies of Innovative Field Technologies Using a Portable GC/MS

## ABSTRACT

The first case study was a project directed by the US Army Corp of Engineers using a manportable GC/MS to expedite site investigation of a contaminated airfield. The GC/MS was used to characterize both the vertical and horizontal extent of the contamination and the data were used to determine the appropriate placement of monitoring wells. The power of using an on-site GCMS was further demonstrated when an unexpected contaminant was discovered in addition the expected contaminants. By using this on-site technique, the project saved 36% of the expended cost.

The second case study was a project to pinpoint the source of chlorinated solvent contamination around a landfill. The solvents were leaching out into a nearby stream and contaminating the water.

The third study used the portable GCMS in a vapor intrusion application. A city health department alerted the state Department of Environmental Protection (DEP) to a home where they suspected the resident was illegally disposing of solvents. This was based on their preliminary analysis of the area surrounding the home using colorimetric indicator tubes. The GCMS was deployed by the DEP to determine what chemicals were actually present in the home and surrounding area.

## Automated Thermal Desorption Methodology Improvements for Environmental Analyses

#### Andrew Tipler and Zoe Grosser

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### ABSTRACT

The uses of thermal desorption–GC systems in environmental applications are well established. A popular application is for industrial hygiene monitoring where volatile organics are trapped on a tube in the breathing range of workers. It is desorbed and chromatographed to characterize the amount and type of potential hazardous material exposure. Ozone precursor analysis to evaluate outdoor air in noncompliance areas for volatile organics is an exacting analysis requiring collection and analysis within an hour. This allows the modeling of changes in organic concentrations over the course of time to better understand the source and impact of troublesome compounds on the production of ground-level ozone. A third environmental application is the measure of a wide variety of volatile compounds in air that may be toxic. Air toxics are more important as air regulations become more comprehensive and the additional chemicals require assessment. There may also be implications for homeland security.

Although systems have been available for these types of analysis for several years they continue to improve. Recent advances have added additional throughput capability and data integrity testing to improve environmental analyses. Sample integrity can be assessed by adding the internal standard prior to collecting the sample. Testing the tube impedance can indicate the quality of data generated in the current desorption and ensure the integrity of the tube for future use. Other improvements in gas flow control, dry purging, and sample recollection improve laboratory productivity. Manifolds are used instead of valves, demonstrating better cleanliness in support of the trend to use mass spectrometry. This talk will discuss some of these improvements and the implications for existing and future environmental analyses.

I prefer to give an oral presentation.

## Tree Coring for Ground-Water Contaminant Tracking and As An Optimization Tool for Monitoring-Well Placement

## Don A. Vroblesky<sup>1</sup>, Clifton C. Casey<sup>2</sup>, and Gregory J. Harvey<sup>3</sup>

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## ABSTRACT

Trichloroethene (TCE) was detected in cores of trees growing above TCE-contaminated ground water in a variety of environments, including the foothills of the Rocky Mountains, a golf course in Texas, pine forests in South Carolina, and a cypress swamp in South Carolina. The data show that tree coring can be used to locate TCE-contaminated ground water and, in some cases, map the lateral extent. In addition, tree coring can be a useful reconnaissance tool for optimizing well placement. For example, Solid Waste Management Unit 17, Naval Weapons Station Charleston, South Carolina, is in a forested area between two tidally influenced surfacewater bodies. The dominant tree species is loblolly pine. Three exploratory monitoring wells at the site showed the presence of chlorinated-solvent contaminated ground water. Tidal influences on the water table made it difficult to predict the probable transport direction of the contamination, and therefore, difficult to place additional wells to map the plume. As a tool to provide a preliminary assessment of the extent of contamination and as an aid in well placement, 63 trees were cored at the site and analyzed using photoionization detection gas chromatography to determine the TCE, PCE, and *cis*-1,2-dichloroethene (cDCE) content of the head space in sealed serum vials that contained the cores. Most of the cores were collected on a single day. The tree coring showed the presence of two apparently separate plumes of subsurface contamination. One plume was predominantly composed of TCE and the other was predominantly composed of PCE. The tree-coring results were used to direct a subsurface investigation using membrane interface probes, which involved collecting ground-water samples. Contaminant concentrations from the ground-water samples showed good correspondence with the TCE, PCE, and cDCE concentrations in the tree cores. This investigation demonstrates that tree coring can be a fast and inexpensive reconnaissance tool to locate and map volatile organic compounds in contaminated ground water and to optimize placement of monitoring wells.

Prefer oral presentation

## Ambient Air Toxics in the Houston-Galveston Area with High and Low TRI Emissions – Monitoring in Three Areas Using Passive Sampling Devices (PSDs)

#### Thomas H. Stock, Maria T. Morandi, and Masoud Afshar

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#### Kuenja C. Chung

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## ABSTRACT

In order to evaluate and compare the spatial variation of ambient air toxics concentrations in urban areas with high or low density of Toxics Release Inventory (TRI) facilities, a series of passive air monitoring measurements were conducted in each of the selected residential areas in the Houston-Galveston area. The purpose of this task was to perform intensive spatial monitoring in the areas surrounding three existing air monitoring stations, i.e., in a high-density TRI area with high mobile source influence, a high-density TRI area with low mobile influence, and a low-density TRI area.

As the high-density TRI area with high mobile source influence, an area including a sourceoriented ambient air-monitoring site in the Houston Ship Channel area (Clinton site) was selected. As the high-density TRI area with less mobile source influence, an area including a source-oriented residential ambient air-monitoring site in Deer Park, (Deer Park site) was selected. And, as the low-density TRI area, an area including a receptor-oriented (residential) ambient air monitoring site in north Houston, (Aldine site) was selected.

72-hour samples of volatile organic compounds (VOCs) were collected using the 3M 3500 Organic Vapor Monitors (OVMs). Samples were collected six times with the planned sampling frequency of every 24 days, over approximately 4 month. During each of six sampling events, ambient samples were collected outside ten residences in each of the three areas, within a 2mile radius of the central site, and simultaneously at the state-operated air monitoring site and at the centroid of the census tract in which the monitoring site was located, which totals 36 sampling sites. Field blank and field duplicate samples were also collected for quality assurance and quality control. All OVM samples were extracted and analyzed for 31 target compounds by gas chromatography/mass spectrometry (GC/MS).

For the major target VOCs, which include BTEX, MTBE, and chlorinated VOCs, the results are compared for the concentration distributions at all houses, the centroid and the central monitoring station for each of the three study areas. Concentrations distributions are also compared among the three study areas.

## **Current Passive Diffusion Sampling Devices and Their Performance** with Selected Target Analytes

### Dee O'Neill

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## ABSTRACT

For several years now Passive Diffusion Sampler (PDS) technology has been applied to many sites to improve the information gained during monitoring and to reduce the cost of sampling. Wide acceptance of these samplers has been gained for hydrophobic Volatile Organic Compounds (VOCs) using the polyethylene bag technique with hundreds of studies demonstrating their effectiveness since 1998. While this material is extremely useful for these specific target analytes, there is a need to identify additional materials that would enable similar performance for hydrophilic organics, semivolatile organics and inorganic target analytes as well. Additional studies using new materials have been underway and progress can be reported on several additional options.

#### These include:

- Polyethylene Diffusion Bag Sampler (PDBS)
- Nylon-Screen Diffusion Sampler (NSDS)
- Rigid Porous Polyethylene Samplers (RPPS)
- Dialysis Membrane Diffusion Sampler (DMDS)
- Polyethylene Vapor Diffusion Sampler (PVDS)
- Semi-Permeable Membrane Device (SPMD)

Along with USGS, efforts to develop passive diffusion samplers for common long term monitoring inorganic and organic parameters include both laboratory and field demonstration studies. Recent data will be presented to demonstrate the performance and feasibility of these materials in prototype sampler designs.

#### EPA SITE PROGRAM DEMONSTRATION PROJECT RESULTS: TEQ SCREENING IN THE FIELD USING INTEGRATED PARALLEL IMMUNOASSAYS FOR DIOXIN/FURAN TEQ AND DIOXIN-LIKE PCB TEQ

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The realtime analytical component of EPA's Triad approach to site assessment and remediation is supported on the organic side by commercial immunoassay kits accepted within the SW-846 Compendium of Solid Waste Methods. Most solid waste immunoassay work to date has involved analytes such as total PCB, total petroleum hydrocarbons, and PAH. Kits for these analytes were approved during the early 1990s in the newly created 4000 series of methods and have seen routine field use since then. More recently, Method 4025 for dioxin/furan TEQ, based on a commercially available kit, was approved in 2001 by EPA's Office of Solid Waste and Emergency Response (OSWER). Because of this acceptance, Method 4025 is now often considered an important first step in site assessment or an essential time saving tool during remediation.

Because of the history of other 4000 series immunoassays and the obvious potential benefit of such a method for field dioxin/furan screening, the EPA Superfund Innovative Technology Evaluation (SITE) Program conducted a field demonstration project in 2004 for Method 4025 and related technologies. In addition to demonstrating the kit on which Method 4025 is based, CAPE Technologies added a second immunoassay kit for measurement of TEQ from dioxin-like PCBs. This PCB TEQ kit is expected to be validated in the near future for use in SW-846 Method 4026 for TEQ from dioxin-like PCBs.

Due to the extremely wide range of source materials, sample types, and TEQ levels, the sample processing of the original Method 4025 was not used. Instead, an adaptation of the Smith-Stalling cleanup method was used for the rapid batchwise cleanup of extracts made by shaking solid samples in acetone:hexane. This method, described before at this conference as Method 4025m (modified Method 4025), allows for easy capture of two discrete fractions, one for dioxins and furans, and the other for dioxin-like PCBs. In the SITE demonstration project, these fractions were analyzed separately using their respective immunoassay kits. The resulting TEQ<sub>D/F</sub> and TEQ<sub>PCB</sub> values were first evaluated separately, then again after adding the two component TEQ values together to get a total TEQ value.

During the demonstration project, 209 soil and sediment samples were analyzed by both CAPE Technologies immunoassay methods as well as by Methods 1613B and 1668A. Comparisons were made in sample throughput, cost, ease of use, and waste generation, as well as in various analytical performance measurements, such as decision making at pre-selected target levels. The summary of the final report stated that, "These data suggest the CAPE Technologies kits could be an effective screening tool for determining sample results above and below 20 pg/g TEQ and even more effective as a screen for samples above and below 50 pg/g TEQ, particularly considering that both the cost (\$59,234 vs. \$398,029) and the time (3 weeks vs. 8 months) to analyze the 209 demonstration samples were significantly less than those of the reference laboratory." Specifics of the report will be described in more detail. Suggestions about implementation of this technology for routine use will also be presented.

## **Session 9**

# Homeland Security – Triage Response

# A UPLC/MS Multi-Analyte Screening Method for Deleterious Organics in Water

#### Jim Krol<sup>1</sup>, and Lawrence Zintek<sup>2</sup>

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#### ABSTRACT

The determination of deleterious organics in drinking water, or soil extract, is one of the particular areas of the Homeland Security Presidential Directive (HSPD-9) that will impact the EPA. It mandates that the EPA Office of Water expand monitoring and surveillance systems for recognizing a terrorist attack, or a significant change in water quality. This is a daunting task because of the breadth of organics, coupled with the numerous water sources required to be monitored.

The question is raised...what organics are present in this water? Whether it is drinking water, surface water, soil leachates, or wastewater, where does a chemist begin to answer this question? Time is critical.

The ability to perform a multi-analyte "screen" for numerous organics simultaneously would help maximize efforts to note the presence and significance of poisonous agents. This requires a broad analytical approach strategy utilizing the specificity of Liquid Chromatography / Mass Spectrometry (LC/MS and LC/MS/MS). Recently, UPLC technology, Ultra Performance Liquid Chromatography, a revolutionary advance in chromatographic science, became available offering enhanced resolution and faster analysis times.

Thus, a "universal" reversed phase gradient providing high resolution analyte separation coupled with the specificity of mass spectrometry allows for the "screening" for multi-analytes in less than 15 minutes.

This method incorporates the use of ESP libraries that can be used with single quadrupole MS instruments in the field, or the same method with MS/MS to quantification and confirmation in the laboratory. Time of Flight MS is becoming practical and offers exact mass determination to the 4<sup>th</sup> decimal place. This high specificity is the future of identifying unknown analytes.

This presentation will discuss the development of a single, multi-analyte screening strategy for several deleterious pesticides and herbicides in drinking water using UPLC/ Electrospray Mass Spectrometry. This work is being conducted in collaboration with USEPA Central Region Laboratory Region 5. Several analytical issues will be raised to stimulate audience discussion and to solicit input to evolve this UPLC/MS strategy into a validated screening method template.

# Using Common Parameter Monitoring as a Surveillance Tool for Water Security. Intelligent On-Line Monitoring to Detect Water Terrorism.

#### Dan Kroll, Karl King

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#### ABSTRACT

Drinking water is one of the nation's key infrastructure assets that have been deemed vulnerable to deliberate terrorist attacks. While the threat to reservoir systems and water sources is deemed to be minimal, the vulnerability of the drinking water distribution systems to accidental or deliberate contamination due to a backflow event is becoming a well-recognized possibility. The myriad possible points of incursion into a distribution system and the ease of mounting a backflow event, combined with the fact that little or no quality monitoring occurs after water has left the treatment plant, makes the danger of such an attack acute. This was clearly stated in a GAO report to Congress that listed the vulnerability of the distribution system to attack as the largest security risk to water supplies.

Prior to this there has not been a system capable of detecting such an event and alerting the system's managers so that effects of an attack or accident can be contained. The general scientific consensus is that no practical, available, or cost-effective real-time technology exists to detect and mitigate intentional attacks or accidental incursions in drinking water distribution systems.

A system designed to address the problem of distribution system monitoring is described here. The developed system employs an array of common analytical instrumentation, such as pH and chlorine monitors, coupled with advanced interpretive algorithms to provide detection/identification-response networks that are capable of enhancing system security, as has been advocated by several Federal research initiatives. Through the use of laboratory testing, pilot scale testing on pipe loops, and real world beta site deployment the system has been shown to be effective in detecting a wide diversity of possible threats. The system has been challenged with, and found effective against, a variety of agents including TIMs (toxic industrial materials), TICs (toxic industrial compounds), chemical warfare agents, and biological warfare materials. Other possible more obscure classes of threat agents such as street drugs, homemade toxins and commercial preparations have also been tested. In addition, the system has been shown to recognize common accidental intrusions such as antifreeze and sewage.

The response of these various agents is not only adequate to detect the presence of a contaminant, but the unique profile of the responses allow for some degree of identification. Through the use of a searchable library of agent profiles the system is capable of providing not only an alarm but also an identification of the likely cause. The profiles of over 80 of the most likely threat agents and many common contaminants have been compiled.

A proprietary baseline estimator dramatically and immediately reduces false warnings from regular fluctuations in operational parameters upon start-up. As time since deployment

increases the number of false positives is rapidly reduced to near zero by the system's programmed ability to learn what is normal for a given operation.

The rapid detection and identification of breaches of security in the water distribution system is crucial in initiating appropriate corrective action. The ability of the described system to detect incursion on a real time basis and give indications as to the cause could dramatically reduce the impact of any such scenario. As the vulnerability of the distribution system becomes more widely recognized, the deployment of a system such as the one described will be an invaluable tool in maintaining the integrity of the nation's drinking water supply.

#### Near Real-Time VOC Monitoring of Public Drinking Water Systems in Response to Water Security Concerns

#### **Carol Thielen**

#### ABSTRACT

Because of the growing concern of terrorist activity directed at this country's infrastructure, the USEPA, the American Water Works Association (AWWA) and numerous municipal water systems have recognized the need to monitor public drinking water distribution systems to detect unknown chemicals which may be intentionally or unintentionally introduced into the water. Within this monitoring system, the detection and identification of VOCs are of the highest concern.

This presentation describes a VOC monitoring system currently installed in a public drinking water system that has been operational for the last 2 years. This monitoring system uses an unattended in-situ, purge and trap GC to monitor VOCs in the influent water to the water treatment system. The GC makes measurements every 30 minutes producing detection limits in the parts per trillion range. This equipment provided the city with a means of early notification when a surge of MTBE and other unknown contaminants were detected in the influent water. The system was initially installed for water security purposes and has demonstrated it's efficacy within the first year of installation.

In addition, this presentation also describes a more sophisticated unattended monitoring system which incorporates many sensors and probes for a variety of water quality indictors in addition to VOCs. This system will automatically compile the real time data, use an inference engine to make adjustments for seasonal variations and alert operators when any of the monitored parameters are outside of the predetermined range.

#### An Automation of Analytical Data Perspective for Homeland Security

#### **Paul Banfer**

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#### ABSTRACT

The automation of analytical data for Homeland Security requires a dynamic, flexible, and manageable process that can be quickly distributed to multiple locations and provide rapid, efficient, and consistent analytical data of the highest quality and integrity from many sources to the expert decision makers.

To establish these processes we must first define the scope:

- A) Potential Threats
- B) Emergency Response Immediate, 8 hours, 24 hours, 48 hours, 72 hours
- C) Contamination Sources, Hazards, Transfer Mechanisms, and Solutions
- D) Analytical Laboratory capabilities
- E) Analytical Methods to perform
- F) Preparation for occurrence

The potential problems:

- A) Training Issues
- B) Laboratory Capability
- C) Logistics
- D) Mobility
- E) Distribution
- F) Consistency of data deliverables

Potential Solutions:

- A) Training
- B) Distribution of software for data deliverables
- C) Software that is mobile, flexible, manageable, and fast
- D) Automation of Quality Assurance
- E) Consistency of deliverables
- F) 100% automation of Method and Standard Operating Procedure verification, Data Validation, and Deliverable Review
- G) Communication of Results from Business to Business, IMS to IMS
- H) Combining information to be represented as one
- I) Communication of Results to decision makers
- J) Potential Models Chemical, Biological, Water resources

#### The State Laboratory: Emergency Response and Data Integrity

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#### SESSION DESCRIPTION

This session will describe state laboratory infrastructure and the essential role state laboratories play in responding to emergencies. The discussion will emphasize the respective roles of CDC, EPA, and NELAC in ensuring integrity of state laboratory data generated during emergency responses. The discussion will show how state laboratory certification/accreditation programs view EPA's oversight role as key to ensuring data integrity.

#### OBJECTIVES

- To describe how state laboratories serve as a unifying element for many state programs, including those directly related to preparedness for emergency actions requiring analytical support
- To stress the importance of including state laboratories as a key element in development of an integrated all-hazards emergency response plan.
- To highlight the role of state laboratories in terrorism preparedness and response.

#### ABSTRACT

This session will examine/describe how state laboratories serve as a major underpinning of an integrated, all-hazards approach for an effective emergency response. This includes responding not only to terrorist events, but also to other events that threaten public health, such as natural disasters (e.g., earthquakes and hurricanes), environmental exposures (e.g., spills and industrial accidents), accidental leaks, discharges, or explosions.

In emergencies, state laboratories must provide data that require the highest level of integration between the laboratory and one or more essential partners. Partners can include state/local health departments, local/state/federal law enforcement, local HazMat and Civil Support Teams, EPA, CDC, and other private or governmental laboratories. Reliable data required by these partners, to make critical decisions regarding public health and safety, can only be obtained through high quality laboratory analyses. It is critical that the quality/reliability of state laboratory data be documented by an established accreditation/certification process.

State laboratories involved with analysis of drinking water and other environmental matrices are often accredited according to National Environmental Laboratory Accreditation Conference (NELAC) standards, certified by the EPA Drinking Water program, or accredited by state-specific accreditation programs. State laboratories involved in analysis of clinical specimens are

required to meet regulatory standards of the Clinical Laboratory Improvement Act (CLIA) and are under CDC oversight as part of the national Laboratory Response Network (LRN). Many state laboratories are involved in more than one of these programs. There continues to be a pressing need for EPA to expand its accreditation/certification role to include *all* of its environmental programs, not just drinking water, so that laboratory data integrity can be ensured in all areas of importance to emergency preparedness and response.

Making critical, rapid decisions necessary in the face of credible terrorist threats, or a multitude of other events that may threaten public health, requires accurate, timely laboratory data that are carefully controlled for quality. While tremendous progress has been made in this regard for the analysis of clinical specimens for both biological and chemical terrorist agents, there remains much to be accomplished to assure quality laboratory data in the analysis of environmental samples and to achieve full integration of state laboratory capability and emergency response. Remaining unaddressed gaps include issues regarding environmental sampling/analysis, food sampling in chemical terrorism events, and appropriate training of first responders and law enforcement in the uses/limitations of field-testing devices. This session will describe the need for credible state laboratory data in response to all-hazards. For this purpose, credible laboratory data will be defined as those based on defensible laboratory procedures with appropriate QA and QC compliant with national standards such as NELAC, or federal standardized methods such as those developed by CDC or EPA as part of a national laboratory infrastructure that includes the LRN and the Food Emergency Response Network (FERN).

#### Unknown Sample Triage Using a Class III Glove Box

#### Phillip Adams

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#### ABSTRACT

Over recent years, there has been an increased need at Public Health laboratories and other testing facilities to have a system in place for handling and analyzing unknown samples in a safe and effective manner. These unknown samples often fall into the FBI's 'suspicious package' category, especially if any evidence of a threat is present. This presentation will outline the necessary steps for unknown sample triage of potentially hazardous materials using a class III glove box. This containment system offers possibly the best available protection to the analyst for both chemical and biological hazards, and ensures sample integrity during analysis.

The main focus of this presentation will be on the screening for hazardous chemicals inside the glove box. The hazards covered will include radiologicals, explosives, corrosives, volatiles, and chemical terrorism agents. Subsequent testing for biological agents will be done outside the glove box in a Biosafety Level III laboratory. The tests will follow the EPA's triage screening protocols that are currently in draft format, and which should be finalized later this year.

The intention of the presentation will be to give the audience an idea of how the EPA's draft triage screening protocols may be implemented in practice, using a class III glove box as the all-hazard containment equipment. The presentation will also address peripheral issues such as correct personal protective equipment, training, chain of custody forms, secure sample storage, and elements that should be included when writing work instructions for people conducting the screening.

#### Building Environmental Laboratory Capability in Support of Emergency Response

Dana Tulis (<u>tulis.dana@epa.gov</u> 703-603-8722) Allan Antley (<u>antley.allan@epa.gov</u> 706 355-8506) US Environmental Protection Agency

#### ABSTRACT

Last year, Agency representatives presented a session regarding plans to address laboratory needs in the event of a terrorist incident. In the event of an actual or suspected terrorist incident, comprehensive laboratory resources will need to be called upon to allow the nation to deal with any situation. Over the past year, the Department of Homeland Security, the Centers for Disease Control, and the Environmental Protection Agency led an effort to draft a Memorandum of Understanding to formalize a federal Integrated Consortium of Laboratory Networks (ICLN) capable of sample analyses for chemical, biological and radiological contaminants of concern in clinical, food, plant, and environmental media.

The President's National Homeland Security Strategy calls upon EPA to be the primary agency responsible for environmental sampling and analyses in response to a terrorist incident. In response to this strategy, and in concert with the Agency's role in the ICLN, an environmental laboratory response network program (eLRN) is in the formative stages in the Office of Emergency Management.

EPA possesses limited capabilities and capacities to analyze environmental samples for chemical, biological, and nuclear materials associated with Weapons of Mass Destruction (WMD). The eLRN is exploring approaches to address this limitation. The Agency's primary analytical capability is oriented toward routine analysis of industrial chemicals, pesticides and conventional pollutants. The first phase of the eLRN will be to formalize network relationships using these pollutants as the model. EPA intends to fully integrate state environmental laboratory counterparts into the eLRN similar to the integration utilized in the other networks.

The structure of the ICLN and the associated networks will be discussed as well as the structure, approach, and status of the eLRN.

The eLRN approach will continue the follow the precepts below:

- to the extent possible make use of the nation's current laboratory resources
- address the problem in the most cost-effective manner
- develop a solution as quickly as possible

#### Low-Cost, High-Volume Air Monitoring for Homeland Security

#### Adam L. Hamilton, P.E.

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#### ABSTRACT

Regional sampling for chemical and biological threats often depends on stationary point samplers that are expensive and provide limited spatial coverage. Augmenting existing point sampling networks with lightweight, inexpensive mobile samplers provides a cost-effective option that enhances the spatial coverage. Suitable mobile platforms include public vehicles (such as buses and trains that operate on predefined routes), public service fleets (police, sheriff, EMS, etc.), and perhaps private fleets (utility, service, etc.). In some cases, even small unmanned aerial vehicles (UAVs) may be used.

A low-cost, high-volume chemical/biological sampler has been developed and tested. This new sampler is:

- Inexpensive;
- Expendable;
- Lightweight (<100 grams);
- Simple to use;
- Capable of collecting trace amounts of material; and
- Operates without electrical power.

The design was a collaborative effort between environmental engineers, aerospace engineers, and manufacturing experts. The result is the Aerotret®. The Aerotret contains filter media for capturing biological materials, as well as a carbon-based sorbent for capturing chemical threat agents (and/or their signatures) present in the form of trace organic chemical vapors. The sampler uses an airfoil shape to enhance flow through the system and to provide the differential pressure to drive air across the filter (for aerosol collection) and the sorbent bed (for vapor collection). The electret filter material has a permanent electrostatic charge and high filtration efficiency for the capture of particulate and aerosols with particle diameters between 0.3 and 10 µm. The inner portion of the filter has a "W" configuration, which provides a means of separating particles by size, driving the larger particles into the center of the W shape and capturing the smaller particles in the airfoil portion of the filter. The outer portion of the filter forms two airfoils. Computational Fluid Dynamics (CFD) modeling indicates the collection rate is equivalent to about 200 liters per minute when the sampler is moving at 35 knots.

The sampler was tested in wind tunnel challenges with various air loadings of fine and ultra fine particle standard dusts with traceability to NIST. Collection efficiencies of the 0.7-10 µm particle size dust standard were greater than or equal to 80%. Simple and efficient installation, handling, and analytical finishes have also been developed to support the sampler.

#### Validation of Sampling and Analysis Methods for Homeland Security Measurements

Larry D. Ogle, David L. Lewis, Molly Isbell, and Kennedy Gauger

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#### ABSTRACT

As new samplers and sensors are developed for Homeland Security monitoring for CBRN, explosives, and toxic industrial chemicals, it is of vital importance that these devices be validated under realistic scenarios. Validation should include tests to verify response according to the manufacturer's specifications and to show that performance satisfies the needs of the end-user. Ideally, the response of these units will be demonstrated against the actual target agents, compounds, or biological threats. Complicating factors in validating the devices are the facilities and permits required to handle and test extremely toxic CBRN materials. In many cases, less toxic surrogates are used to validate device performance.

Device validation should be designed to show that they will display advertised sensitivity and will exhibit a minimum of interferences or false positives. For most devices, operation has been "validated" in a laboratory environment, but there may be a significant "gap" between performance demonstrated during product testing and performance when operated by the end-user in a field detection situation. This paper addresses these performance gaps and describes details for design and implementation of a field validation/verification program for new and improved samplers and sensors. In addition, traditional EPA validation steps have focused on determining precision, accuracy, and sensitivity of the measurement process. For CBRN field instrumentation, rather than obtaining accurate quantitative measurements, the focus of the validation process may be directed toward answering the question of presence or absence at a specific concentration with changing environmental interferences.

A successful field validation program begins with a comprehensive and well-designed test plan. The test plan should include a statistically valid test design; specify the range of test materials (e.g., target compounds) and conditions; detail release conditions and test materials, including the potential for interferents; describe the Ground Truth method to be used for comparing performance; and include a Health and Safety plan. The Health and Safety Plan must address test material toxicity, personal protective equipment (PPE), and potential ambient concentrations. As appropriate, release levels should be modeled and exclusion zones established to restrict access to hazardous areas during testing. Following the test, the data should be complied and a final report developed that incorporates a statistical evaluation of all data with conclusions based on measurement data.

# The TIGER Biosensor: Applications in Biodefense, Epidemiology and Infectious Disease Surveillance

#### Steven A. Hofstadler, Kumar L. Hari and David J. Ecker

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#### ABSTRACT

The TIGER (Triangulation Identification for Genetic Evaluation of Risk) biosensor provides a novel and universal strategy for the detection and characterization of microorganisms associated with a potential biological warfare attack or a natural outbreak of an emerging infectious disease. The process uses mass spectrometry, signal processing, and base composition analysis of PCR amplification products from biologically conserved regions of microbial genomes to simultaneously identify the organisms present in a sample without the need for culture. The sample can be derived from air filtration devices, clinical samples, or other sources. Core to this approach are "intelligent PCR primers" that target broadly conserved regions of microbial genomes that flank variable regions. This strategy distinguishes TIGER from other detection/identification strategies in that TIGER requires no prior knowledge about an organism in order to identify it in a sample. The approach requires that high-performance mass measurements be made on PCR products in the 80 – 140 bp size range in a high-throughput. robust modality. The base compositions from multiple primer pairs are used to "triangulate" the identity of the organisms present in the sample. Use of species-specific primers allows rapid strain-typing of the organism. The concept is equally applicable to bacteria and viruses and could be further applied to fungi and protozoa. Moreover, the use of biologically essential gene targets to obtain microbe signatures enables the high-probability detection of both natural and bioengineered agents.

The TIGER system has been rigorously validated for use in biodefense applications, including surveillance for biological weapons agents in environmental samples, and tested against a broad range of biological samples in military troop health settings. For applications in air surveillance, the detection of numerous biothreats, surrogates, and near neighbors is demonstrated by spiking air filtrate with spores, vegetative cells, virion, or gDNA from threat organisms. Excellent performance is demonstrated even in the presence of a significant "clutter" of background organisms. Preliminary results will be presented on testing normal drinking water for the presence of biological threat agents. In an emerging infectious disease surveillance modality, an example will be shown in which the SARS virus was characterized and readily distinguished from other closely related human corona viruses using primer pairs that were designed prior to the availability of the SARS sequence. Further, in a collaborative effort with Naval health officers, we have examined cultures and direct throat swabs obtained from military personnel suspected to be suffering from Group A Streptococcus (GAS) infections. Samples were first analyzed using a panel of survey primers that readily identified the infectious agent as Streptococcus pyogenes, clearly distinguishable from all other organisms, including other streptococci and staphylococci. Subsequent TIGER analysis with Streptococcus-specific primers rapidly yielded emm-type strain resolution for each sample, which was later

corroborated with conventional MLST analyses. This study demonstrated that TIGER can be used to detect and identify infectious agents directly from throat swabs. In the present configuration, hundreds of samples can be analyzed within 12 hours allowing near real-time evaluation of patient samples and will make possible more rapid and appropriate treatment of patients in an ongoing epidemic. The use of "drill down" primers allows closely related strain variants to be distinguished and accurately identified. This is of particular importance when distinguishing biological weapons agents from near-neighbor surrogates, or when tracking the spread of particularly virulent strains of disease-causing organisms.

#### **Quality Control Challenges for Extremely Toxic Compounds**

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#### ABSTRACT

Continuous monitoring for chemical and biological agent attacks has become of vital interest to our national Homeland Security initiatives. Monitoring systems (such as those used in the Biowatch program) placed in large urban areas across the country will generate a huge amount of data, with most if not all, being negative for the CW or BW agents of interest. However, a positive result, and the decisions that must be made in response to this result, can have an enormous impact on the affected location and on our nation. Therefore, the Quality Assurance and Quality Control program associated with these monitoring programs must vigorously assess the performance of the continuous monitoring systems to fully characterize and understand that performance.

Minimizing the number of false positive results from these measurement programs is of great interest, since the decision to evacuate or quarantine a large population area based on a faulty measurement could have a grave negative impact on the population and on the monitoring program. Of equal or greater importance is the determination of the program false negative rate (e.g., how often would we NOT see the target when it was actually present). Common QA practices such as field spikes and blind media spikes are impractical since the targets of interest are extremely toxic and their handling and shipping are restricted. However, false negative rates can be assessed through the use of surrogates provided the laboratory routinely monitors for the surrogate compounds. In addition, the performance of the monitoring systems with the actual targets can be routinely assessed in controlled laboratory experiments at surety facilities designed to handle the agents.

This paper will discuss the approaches to assessing the continuous monitoring systems performance via QA/QC samples employing control samples spiked with surrogate compounds. In addition, the numbers of blanks, spikes, and other controls necessary to establish high confidence levels will be discussed. Challenges associated with monitoring system evaluation using the actual targets will be discussed as it is considered to be very important to assess collection efficiencies, desorption efficiencies, analytical variability, etc., with these agents.

## Session 10

## **Managing Decision Uncertainty**

#### How to Fully Integrate Available Information Resources: Maximizing Planning for Environmental Monitoring and the Real Benefits to the Planner

#### Ruby N. White

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#### ABSTRACT

Scientists, analysts, engineers, and managers know that planning environmental monitoring or increasing understanding of current environment status requires ready access to available information resources. Public sector planners, including those conducting technical monitoring as well as communities, use Web tools offered by EPA and others to construct targeted information searches. Understanding how these tools work as well as the information provided is critical to their efficient use in planning, and ultimately, the success of associated technical activities. Availability of all these tools, along with associated tutorials that offer varying degrees of assistance, may not be known to planners and the public.

This technical presentation provides:

- a detailed analysis of information resource tools available to planners,
- a standard approach for structuring access to best benefit users,
- a method for users to compare information available to their planning objectives, and
- a feedback mechanism to ensure continued improvement in integrating the tools into environmental planning.

#### Automation of Analytical Results for the Triad Approach

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#### ABSTRACT

The analytical industry now provides an array of Scientific Data Management Software products that can work with or without a Laboratory Information Management System (LIMS). These software products focus on the analytical quality assurance, quality control, and data deliverable production within an analytical department (Volatiles, Semi-Volatiles, Pesticides/PCBs, Metals, and General Chemistry). Therefore, these systems can be used quite extensively with the Triad Approach because they can be used in a Mobile Lab, an ASP, or contracted along with a laboratory analyst and instrument to meet specific needs of the project, quality assurance, and regulatory requirements.

One of the key elements of the Triad Approach is the real-time measurement for real-time decision making. To facilitate real-time decision making, analytical data need to be of sound quality and Data Management tools used in the field should be able support rapid transfer of data to all the interested parties.

These systems can also work with Laboratory Information Management Systems or other Data Management Systems to pass information back and forth as if the process was one streamlined system.

This presentation will focus on the automation of Analytical Results to the Decision Makers through analytical software:

- 1) Mobility
- 2) Flexibility
- 3) Manageability
- 4) Capability
- 5) Communication of analytical data
- 6) Monitoring and analytical process, procedures, and data
- 7) Defensibility

#### The South Dakota Triad Challenge

#### **Dennis Rounds** South Dakota Petroleum Release Compensation Fund 445 East Capitol Avenue, Suite 200 Pierre, SD 57501 E-Mail: <u>dennis.rounds@state.sd.us</u> Phone: 605-773-3769

#### ABSTRACT

In the fall of 2004, the South Dakota Petroleum Release Compensation Fund (PRCF) initiated a study to determine the cost and effectiveness of using the Triad approach at relatively small petroleum release sites. The results of the study suggest that the Triad approach will work well managing data uncertainty at small sites and may be preferential to other more conventional methods of site characterization.

The South Dakota PRCF is a state agency and is the financial assurance mechanism for regulated UST owners. Five sites were chosen for the study which included three active gas stations, one closed gas station and a railroad fueling site. The EPA provided the PRCF with a \$50,000 grant to assist with the study. All locations were considered "legacy" sites because the petroleum releases had been discovered some time ago, yet none of the sites were effectively moving toward regulatory closure. Some of the sites had been in the assessment process for over a decade with no remediation to date. The known tanks at the closed gas station had been removed over 10 years ago, but no assessment had been conducted. The goal of the study was to apply the principals of the Triad in order to rapidly characterize the sites, develop accurate conceptual site models, establish clear cleanup goals and move the languishing sites toward regulatory closure as rapidly as possible. The principals of the Triad were used in the management of the project sites. In accord with the graded approach endorsed by Triad, planning and site work were tailored to fit the relatively small nature of the petroleum release sites. The PRCF contracted with Columbia Technologies to conduct the field analyses and Midcontinent laboratories to perform collaborative analyses as necessary using quality assured laboratory methods. A team was assembled for each site which included personnel from the PRCF, the SD Dept. of Environment and Natural Resources, the owner or his agent, the environmental consultant and personnel from Columbia Technologies. Systematic planning was conducted with the use of an experienced Triad mentor to establish clear objectives for each site. Direct push and direct sensing technologies were used in the field to gather site data and conduct the rapid, real-time measurement aspect of the Triad approach. All team members remained on site until uncertainty was minimized and data gaps were filled. Decisions regarding the depth and location of borings and the type and number of collaborative lab samples were made by the team on site, relying on the real-time measurements. The team did not move to the next site until all members were satisfied that data uncertainty had been minimized to an acceptable level. Columbia Technology's "Smart Data Solutions" was used to convey field collected data to a secure internet website where it was posted as it was collected. The site model was updated several times per day using 3-D graphic images to aid the team in reducing uncertainty and filling data gaps. All 5 sites were successfully characterized within a single three-week period. All sites now have clear objectives for remediation and site closure.

#### Managing Decision Uncertainty on Navy Cleanup Projects

Kimberly Gates, P.E.

#### ABSTRACT

Almost all (important) environmental decision problems involve some level of uncertainty either in its data or measurements, the values assigned to parameters describing future work on the site or even about the environment in which we operate. 'Uncertainty' refers to our imperfect and inexact knowledge of the world. We can use certain management approaches to quantify and "tame" uncertainty on cleanup projects.

The Triad approach seeks to manage uncertainty throughout the cleanup process. The three key elements of the Triad approach that achieve successful uncertainty management are: systematic planning, dynamic work strategies, and real-time measurements. The first stages of the Triad approach determine whether the type, quantity, and quality of environmental data needed to support a decision has been achieved. This presentation will introduce environmental professionals to this approach for managing decision uncertainty using case studies from two Navy sites to demonstrate the success of the Triad approach. The Navy will continue support the utilization of Triad approach for managing decision uncertainty for cleanup projects from initiation of the Triad team until the land is available for reuse.



## Quality Assurance and Quality Control for Triad Projects

21<sup>st</sup> Annual National Environmental Monitoring Conference Washington DC July 2005

> TODD A. KIMMELL (ANL) DEANA M. CRUMBLING (EPA)

#### Argonne National Laboratory



A U.S. Department of Energy Office of Science Laboratory Operated by The University of Chicago



## Acknowledgments

The Triad is an EPA initiative, with active federal involvement by representatives of:

- The U.S. Army Corps of Engineers
- The U.S. Navy
- The U.S. Air Force
- The ITRC and state representatives
- DOE's Argonne National Laboratory

My Co-author, Deana Crumbling, represents EPA's Technology Innovation and Field Services Division, part of the Superfund Program (old TIO)



Office of Science U.S. Department of Energy

## Introduction

- Our presentation is intended to present Quality Assurance and Quality Control (QA/QC) needs for Triad-based programs
- First, I'll present a brief summary of what Triad is all about
- Then I'll discuss QA/QC concepts and show how they relate to Triad
- I'll then discuss specific Triad QC components
- And conclude with the main Triad-QA/QC message we want you to go away with, which can be characterized by one word:

#### "FOCUSED"





## EPA's Triad Initiative

- Systematic Planning (facilitated by Conceptual Site Models - CSMs)
- Dynamic Work Strategies (leading to more and more accurate CSMs)
- Real-Time Measurement Systems



For site characterization and remediation projects



Office of Science U.S. Department of Energy

## What is the Goal of the Triad Initiative?

- Encourage the environmental community into understanding that there is a critical need to adopt 2<sup>nd</sup>-generation practices
- Adopt modern characterization, remediation and monitoring technologies and strategies to improve CSM accuracy and cleanup efficiency
- Implement change in related areas, such as procurement, project planning, regulation development/implementation, and QA/QC



## The Triad IS about...

- Facilitating communications with stakeholders initially and throughout the project
- Cultivating professional competence and multidisciplinary teams ("allied environmental professionals")
- Constructing accurate CSMs (as a primary Triad product) to support cost-effective decisions
  - Done in real-time to cut lifecycle costs
  - Controlling for sampling variables and focusing QA/QC to manage data uncertainties specific to project decisions
- Actively managing decision uncertainty using efficient and cost-effective tools and strategies





## What is the "Keystone" Concept for Triad?

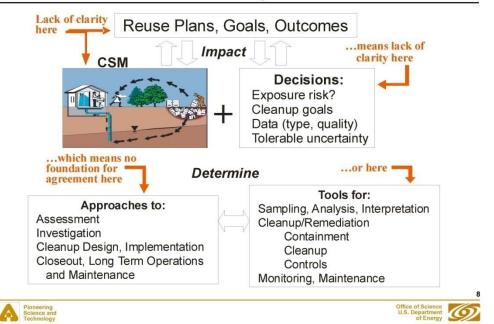
• The "Keystone" principle that links all other Triad concepts is

#### Management of decision uncertainty

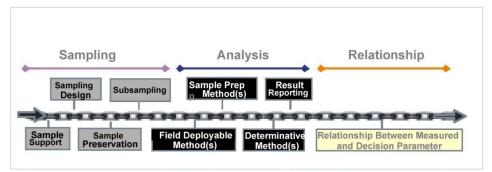
- Is gathering new data necessary to manage decision uncertainty? Ask yourself: Will new data or information change the decision?
- When data relevant to the decision-making process needs to be gathered then:
  - Need to target specific data needed
  - Collect and manage that data with the goal of minimizing decision uncertainty



### Where Does Uncertainty Lie?



## The Data Quality Chain

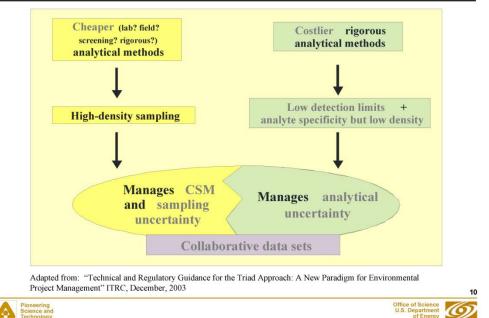


All links in the Data Quality Chain must be intact for Decision Quality to be supported!

Adapted from: "Technical and Regulatory Guidance for the Triad Approach: A New Paradigm for Environmental Project Management" ITRC, December, 2003

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# Collaborative Data Sets Play an Important Role in the Triad



# How Do We Combine These Concepts Into a Cost-Effective QA/QC Program for Triad?

- Systematic Project Planning provides the foundation for
  - Ensuring quality at all levels
  - Fostering effective management of decision uncertainty
- Dynamic Work Strategies means
  - Changing or modifying measurement systems as conditions warrant AND
  - Changing or modifying QC protocols as conditions warrant
- So Triad projects should include
  - Initial planning sessions geared towards management of decision uncertainty
  - Subsequent planning sessions that consider changing or modifying measurement systems and QC protocols as warranted





## QA/QC is Focused Under Triad...

- QC in the Triad sense involves controlling factors that could introduce uncertainty into the data quality chain
- QC under Triad includes control of field and lab methods:
  - Equipment is working properly
  - Field and laboratory operators are performing appropriately
  - Geotechnical techniques are properly preformed
  - Samples are collected and processed using the proper procedures and with the proper sample support
  - Software and computer programs are appropriate and properly applied





# What Specifically Do We Mean By Use of the Term "Focused?"

- Initially focused on QC NEEDED to determine method performance, and to document data quality
- Then refocused as the CSM is modified with new data to target specific data elements that are relevant to the decision-making process
- BECAUSE the level, frequency or type of data needed may change over time, the level of QC activity can also be changed over time – to focus on specific elements that impact decision uncertainty
- Ultimately, QC under Triad is focused on parameters that are relevant to the decision-making process



# Now Lets Look at Some Examples of Focused QC...

- As objectives move from producing data for risk assessment to hot spot identification, QC may be relaxed
- If data collection switches from supporting a remedial action to site closure documentation, QC may become more stringent
- If the real-time method is producing non-detect results, more frequent use of laboratory methods may also be considered as part of a collaborative data set. Additional options can include steps to confirm that that field non-detects are not false negatives and include:
  - Increase the frequency of low-level spikes
  - Perform more frequent low-level calibrations
  - Increase field and laboratory quality control samples



Pioneering Science and



## And Some More Examples of Focused QC...

- After initial determination that real-time methods are producing accurate results, less frequent use of lab methods may be considered as part of a collaborative data set
- Sample matrix characteristics might change unexpectedly (e.g., due to higher moisture content, increased organic carbon content) and warrant closer monitoring of method performance
- Focus back-end data review (verification/validation) on data that will drive decision making
- After initial data verification/validation shows that systems are in control, reduce the frequency of these activities



### *Triad QC Includes the Need For Some Traditional QA/QC Elements*

- A "culture of quality" among all team members, especially the field team because especially with real-time measurement systems, the first line of defense is in the field
- A QAPP that documents methods to be employed and QA/QC to be performed
- Well-defined performance goals and metrics such as criteria for data precision, accuracy, representativeness, comparability and other traditional data quality indicators (articulated in the QAPP)
- Use of and adherence to Standard Operating Procedures for ensuring consistency and data reliability (also articulated in the QAPP)
- A knowledgeable technical team member with responsibility for project QA/QC (often called a Quality Assurance Officer)



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### But Triad QC Includes Some Elements That Are Unique to Triad or Different in Scope...

- A QAPP that that is dynamic and that can be modified and focused (often in the field) as the project proceeds
- Methods Evaluation Studies (Demonstration of Method Applicability)
  - To establish a quantitative relationship between field-deployed methods vs. laboratory methods with site-specific media
  - To identify specific parameters that must be carefully monitored, such as moisture content of soils and sediments
- Provisions for "Customized QC" for the performance of laboratory methods in the spirit of PBMS
  - Is the type of stringent QA/QC called for by standards setting organizations always necessary?
  - A cold hard look, for example, at calibration regimes, internal standards, surrogate analyses, and laboratory QC samples





# And Other Elements That Are Unique to Triad or Different in Scope...

- Data Evaluation Data verification and validation is a critical component of QA/QC programs. Key questions are:
  - How can data evaluation be performed real-time?
  - What level of data evaluation is really necessary?
  - Can evaluation steps be done in a less time-critical fashion?
- Logistical Considerations Time is often critical when field activities are underway and decisions need to be made in realtime. For example:
  - If laboratory data are needed as a point of comparison, there
    may be a lag between availability of real-time and lab results
  - Establishing communication protocols between field teams, database managers and decision makers can also be critical
  - Readiness reviews and "dry-runs" can be an important QA component of a Triad program to ensure that logistical considerations have been sufficiently addressed





## Conclusions

- Triad is a coordinated effort to integrate proven strategies into a framework that improves the cost-effectiveness of and confidence in project outcomes
- Triad is all about managing decision uncertainty
- QA/QC for Triad projects is different from conventional cleanup programs
- While the goal is the same To generate data of known quality whose quality characteristics are documented, verifiable and technically defensible
- QA/QC for Triad is focused then adapted and refocused as a project proceeds in response to changing project needs or site conditions.
- Focused QA/QC inherently means using limited QA/QC resources so as to maximize decision certainty
- Put your QA/QC money where it will do the most good data sets important for decision making, and QC necessary to document data quality





## For More Information

- EPA's Triad resource center may be accessed at <u>http://www.triadcentral.org/</u>
- Dan Powell
  - U.S. EPA

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### A Bit About Myself and Argonne's Triad Experiences

- Todd Kimmell Former EPA employee and an "Argonnite" since 1993
  - Environmental Scientist and Policy Analyst
  - Worked for EPA in OSW's Methods Program in "the early days" (TCLP)
  - A RCRA "weenie" specializing in the DOE and DOD "special waste" issues, including conventional munitions and chemical warfare agents
  - Heavily involved post-HSWA in EPA's RCRA Corrective Action Program
  - QAO for two large Army RIs with conventional and CWA munitions issues
  - Input to EPA's Triad initiative, specifically with respect to QA/QC
- Argonne's Environmental Assessment Division (Chicago and DC)
  - Involved in many site characterization/remediation programs over the years
  - Developed a precursor to Triad called Adaptive Sampling and Analysis Program (ASAP)
  - Involved in many facets of DOD's munitions and range management initiatives
  - Assisting EPA in developing the web-based Triad Resource Center
  - Assisting several federal and private organizations in implementing Triad





#### Addressing the Misconceptions About QA/QC in Triad Projects

#### William M. Davis

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#### ABSTRACT

The Triad approach addresses the uncertainty associated with site heterogeneity by using field based measurements to assess, generally in near real time, the representativeness of samples and sampling strategies. By using predetermined yet flexible sampling protocols (proscribed in the dynamic work strategy), the site investigation converges on the sampling density necessary to reduce the conceptual site model (CSM) uncertainty to acceptable levels that support the site decisions the project was designed to address. Analytical uncertainty is also assessed and controlled to the level necessary to support site specific decisions. All Triad projects have Quality Assurance Project Plans.

Precision and accuracy are very important DQOs with any project. All data used to support site decisions must be of defined quality. There has long been a perception that field measurements can not meet precision and accuracy requirements to provide data of adequate quality for various site decisions (i.e. remedial design/selection, risk assessment). The Triad approach addresses precision and accuracy in terms of whether a technique or system provides data of defined quality which is adequate to support decisions, often in real-time or near real-time. There are many different field measurement technologies available today, all of which can provide data of known quality as long as the analyst performs proper QC procedures. Even relatively qualitative data such as the GeoProbe membrane interface probe, immuno-assay and colorimetric measurements require calibration and periodic calibration check samples to insure they are operating within acceptable precision and accuracy criteria.

In Triad projects, data are generally used in real-time, as often as immediately after the completion of the analysis, to make decisions about the site investigation. It is critical that the field analyst use the QC data as it is collected to access data quality in real-time and to alert the on-site project manager to any QC problems are encountered. It is true that data are used in Triad projects to make decisions before third party validation occurs. This is due to the immediate use of the data by the core technical and decision teams. However, this does not mean that the proper QC procedures are not followed or that third party validation is not possible. Field measurement technologies produce data sets that are often validated after the completion of the field portion of the project. The level of QC and data validation required are project specific and are key elements in the systematic planning portion of Triad projects.

A hallmark of Triad project uncertainty management is the use of multiple data streams to evolve the CSM. For example, collecting high resolution geologic data and high resolution contaminant distribution data often allow a very detailed understanding of the contaminant distribution relative to groundwater flow. The convergence of the collaborative data sets adds to their ability to manage project decision uncertainty. It should be remembered that the objective of QA/QC procedures for any project is to insure that data quality is adequate to support project specific decisions. Although not a traditional form of project QC, the convergence of

collaborative data sets is a strong component of all Triad projects that is used to manage decision uncertainty.

This paper will discuss the implementation of QA/QC procedures during a recent Triad investigation that used semi-quantitative data (MIP) and both on site quantitative (EPA Method 8265) and off-site quantitative (EPA Method 8260b) data to address uncertainty in the CSM.

# Laboratory Certification for Field Analytical Methods and Triad in New Jersey: Perfect Together

#### Stuart Nagourney and Brian Sogorka

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#### ABSTRACT

New Jersey has more than 10,000 contaminated sites, many of them brownsfields areas where timely remediation is critical to commercial viability. The Triad approach promoted by the United States Environmental Protection Agency and the Interstate Technology Regulatory Council, has been adopted by the New Jersey Department of Environmental Protection (NJDEP) as a way to expedite the cleanup of such contaminated sites.

NJDEP is exploring mechanisms to convince its staff and management that field analytical measurements can be relied upon to build accurate conceptual site models and reduce project decision uncertainty while saving time and money. NJDEP is exploring ways to break traditional beliefs that data generated by certified permanently-sited laboratories are definitive, despite the very low density of such data points. Two NJDEP units, one responsible for laboratory certification, and the other for management of site cleanups, are collaborating to improve the confidence in and acceptability of field analytical data by management and staff within the NJDEP. This talk will provide an update on NJDEP's experiences and lessons-learned developing an accreditation program for service providers of field-generated data, as well as technical and institutional barriers.

## Session 11

## Inorganic Methods – Advances in Elemental Speciation

#### Chromium(III) Oxidation in Chromite Ore Processing Residue-Enriched Soils: Theoretical Predictions and Experimental Observations

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#### ABSTRACT

The oxidation of Cr(III) to Cr(VI) in field-moist soils can theoretically occur using Mn(III,IV) (hydr)oxides as the oxidant. Uncertainties surrounding whether or not this redox reaction may occur in chromite ore processing residue (COPR)-enriched soils have complicated decision-making on the analysis, remediation, and regulation of these alkaline soils containing both Cr(III) and Cr(VI). Thermodynamic predictions show that pH and Eh are soil master variables affecting the speciation, solubility, and reactivity of Cr(III) and Mn(III,IV)(hydr)oxides. The hexaquo Cr<sup>3+</sup> cation is the most reactive form of Cr(III) with negatively-charged Mn(III,IV)(hydr)oxides; and insoluble forms of Cr(III) in paracrystalline hydroxides, crystalline oxides, and organic complexes are much less reactive or are not oxidizable at all. In alkaline (pH 8-13), aerobic COPR-enriched soils; Cr(III) has been shown to be inert toward oxidation to Cr(VI), and Cr(VI) has also been shown to be inert toward reduction under unremediated field conditions.

During the hot, alkaline extraction used to dissolve sparingly-soluble and soluble forms of Cr(VI) from COPR-enriched soils (USEPA SW-846 Method 3060A); residual chromite,  $Cr_2O_3$ , and other COPR-borne forms of Cr(III) are not prone to oxidation, especially with  $Mg^{2+}$  added as a suppressant. In COPR soils in which remediation-by-reduction methods have been used to convert Cr(VI) to Cr(III), questions have been raised as to whether such newly-reduced, precipitated Cr(III) may re-oxidize in the future by  $O_2$  or Mn(III,IV). Laboratory and field studies have failed to show that such a re-oxidation reaction occurs, presumably due to the recalcitrant nature of insoluble complexes of Cr(III) with OH<sup>-</sup> or the oxidized forms of the reducing agents used for remediation.

A knowledge of the redox soil chemistry of Cr is essential for predicting accurately the extent to which a given form and concentration of Cr(III) might oxidize in a COPR-enriched soil. Thermodynamic predictions, kinetic experiments, and site-specific observations and field trials provide evidence that COPR-borne forms of Cr(III) are not expected to oxidize to Cr(VI) under alkaline, aerobic field conditions.

# Application of Chromium (VI) Speciation Results for Remedial Alternatives Evaluation

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#### ABSTRACT

Although speciating quantitatively hexavalent chromium [Cr(VI)] in industrial wastes has been routine practice in the electroplating industry since the late 1960s, reliable quantification of Cr(VI) in solid matrices (e.g., soils, hazardous wastes) has been achieved only since the early 1990s. This was accomplished through research and development focused on the characterization and treatment of Cr(VI)-bearing soils, particularly those enriched with chromite ore processing residue (COPR). A major landmark in quantifying Cr(VI) in solid matrices was promulgation of SW-846 Methods 3060A/7196A (Alkaline Digestion Followed by Colorimetric Determination for Analysis of Cr(VI) in Solid Matrices) in 1997. Acceptance of these methods essentially "superceded" prior conventional practice for quantitative speciation of Cr(VI) in solid matrices. The approach to speciation embodied in these commercial laboratory methods requires the use of ancillary parameters to determine the oxidizing or reducing state of a sample. Comparative analysis of samples containing COPR using SW-846 Method 6800 (Elemental and Speciated Isotope Dilution Mass Spectrometry) yielded essentially identical results to those obtained by Methods 3060A/7196A. However, Method 6800 has been used by only a limited number of researchers and is not yet available commercially. Notwithstanding, a regulatory agency has proposed Method 6800 for Cr(VI) analysis as part of a tiered decision hierarchy related to Cr(VI) data use that would reject Cr(VI) data obtained using Methods 3060A/7196A for site characterization and remedial actions.

The data and knowledge gained comparing various extraction and analysis methodologies for Cr(VI) quantitation have been applied both to characterize the extent of Cr(VI) contamination at multiple sites suspected to have COPR, and were subsequently applied to determine whether Cr(VI)-bearing soils, particularly those enriched with COPR, could be treated to meet soil Cr(VI) cleanup levels under regulatory consideration.

Applying the significant differences in mobility and toxicology between soluble Cr(VI) and stable insoluble Cr(III), the results from characterizing thousands of solid matrix (e.g., COPR, soils, debris, various other fill materials) samples and extensive treatability efforts have been derived from basic laboratory research, field pilot studies of remediation technologies, and full-scale *ex situ* treatment system implementation. A major focus of treatability testing was to determine if elevated Cr(VI) could be irreversibly reduced to contemplated soil cleanup criteria levels {~20 mg/kg to ~200 mg/kg), depending upon site-specific risk assessment results obtained from scenarios ranging from residential to industrial/commercial property use.

This paper addresses the application of Cr(VI) speciation data collected for site characterization and delineation, and evaluation of treatment alternatives and achievement of Cr(VI) soil cleanup

criteria via *ex situ* or *in situ* site remediation activities. The data and results are examined in terms of scientific validity, process performance achievement, and treatment methodology limitations/obstacles to successful transformation of Cr(VI) to an immobile and essentially non-toxic Cr(III)-bearing solid matrix that allows sites containing COPR-enriched soils to be remediated and restored to beneficial community use. The proposed use SW-846 Method 6800 and its implications on (a) site characterization and delineation, (b) "clean closure" where remediation activities have met the designated Cr(VI) soil cleanup criteria, and (c) these same issues associated with other metals-contaminated sites nationwide, will also be addressed.

# An Evaluation of Analyte Isolation and Analytical Finish Methods for Cr(VI) in Solids

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### ABSTRACT

Chromium exists predominantly in two stable forms (valence states): Cr(VI) and Cr(III). In environmental samples, there is a fundamental need to differentiate between the two valence states because Cr(III) is not considered to be toxic to humans, whereas Cr(VI) is considered an inhalation carcinogen. The analysis for total chromium is relatively straightforward; however, the successful analysis for Cr(VI) in complex soils/solid matrices can be more complex.

In solid materials, there are always two steps to the quantification of Cr(VI): The extraction or isolation of the hexavalent chromium species and the analysis of the digestate for Cr(VI). The development and ultimate promulgation of SW-846 alkaline digestion procedure (Method 3060A) provided the public with the critical means to reliably extract Cr(VI) in a solid matrix sample while preserving the native valence state of chromium in its environmental setting. A variety of analytical finish methods are available to guantify the Cr(VI) in solution. The colorimetric procedure, SW-846 Method 7196A, is the most common for Method 3060A digestates because of its wide availability and low cost. Somewhat less widely used is SW-846 Method 7199 which utilizes an ion chromatograph (IC) with separation of Cr(VI) on an anion exchange separator column. SW-846 Method 6800 - Elemental and Speciated Isotope Dilution Mass Spectrometry- was promulgated in 1998 and can be used to speciate chromium as well as other metals of environmental concern (e.g. As, Hg) in solid matrices. Method 6800, however, is not yet available at commercial laboratories and the unit cost [currently much higher than 7196A or 7199] for commercial utilization remains to be determined. In New Jersey, regulators previously mandated the use of an alkaline digestion procedure (NJDEP-modified Method 3060) coupled with a colorimetric analytical finish (NJDEP-Modified Method 7196A) on New Jersey chromite ore processing residue (COPR) Sites. NJDEP-modified Methods 3060/7196A specify essentially identical chemistry and laboratory procedures as SW-846 Methods 3060A/7196A.

Regardless of the analytical finish method performed, extensive research has conclusively demonstrated that in the analysis for Cr(VI), soils or waste samples rich in reducing agents (*e.g.*, organic matter, sulfides, ferrous iron) cannot support chromium in the hexavalent state. Low matrix spike recoveries (<75%) are actually expected in these matrices, not because of bias, but because Cr(VI) cannot exist. Thus, the evaluation of Cr(VI) matrix spike recoveries when assessing method performance or potential bias requires a non-traditional approach. Specifically, it is inappropriate to automatically deduce poor method performance or result bias for Cr(VI) matrix spike recoveries outside the traditional acceptance range (e.g. 75%-125% recovery).

Despite definitive research results and peer-reviewed publications, a regulatory agency has recently suggested policy changes that would render Cr(VI) results via Methods 3060A/7196A (or

NJDEP-modified Method 3060/7196A) unacceptable for samples associated with matrix spike recoveries <75% or >125%. The proposed change would reject data associated with matrix spikes <75% or >125%, essentially mandating the use of Methods 3060A/6800 for the analysis of Cr(VI)

A sizeable database of Cr(VI) analytical results for soils and sediment samples by the various analytical finish methods has been developed through remedial investigation of more than 35 COPR sites in New Jersey. The database includes an abundance of matrix spike recovery data by the various methods. This paper provides a comparison of the results generated by the various methods and a discussion of matrix spike recovery as it relates to the efficacy of these methods.

# When It Comes to Speciation, "To Label or Not To Label? That Is the Question"

### Brian Buckley, Willie Johnson, Qiang Tu, Eric Fisher and Riumin Xie

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### ABSTRACT

EPA method 6800 allows the analyst to monitor changes in the chemical species during sample; transport, storage, processing and even analysis. Using stable isotopes allows multiple spiking solutions to be added to the sample, each labeling a different chemical species. It would appear that this method should allow everyone to perform speciation analysis without fear of introducing bias to the analysis. With this capability, one might wonder what are the limitations to the method? The first is that it requires mass spectrometric measurement of each of the chemical species. Second is that it is not applicable to analytes with only one isotope (monoisotopic) and third is that it can be very expensive to perform. There is an alternative to method 6800, using recovery percentages. For those who use optically based detection techniques this alternative is a viable option. For the more frugal analyst it may present an alternative worth exploring and for someone looking at either As or Mn, it is the only way to check for interconversion. Its drawback is that it does not allow the analyst to observe species cycling if it occurs. This presentation will focus on the use of recovery percentages to monitor for species interconversion and its application to As and Hg speciation.

### Bromate/Bromide Speciation by HPLC-ICP-MS

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### ABSTRACT

Water for public consumption must be purified prior to distribution. A number of processes are used for water purification, including treatment with ozone to kill bacteria. While this method is effective, ozonolysis can also convert bromide (a natural component of many waters) into bromate ( $BrO_3^{-}$ ), a carcinogen. Therefore, the need exists to measure bromate in drinking waters, which means that it must be measured separately from other forms of bromine. Current methods for measuring bromate and bromide involve separating the bromine-containing components by ion chromatography and using ICP-MS as a detector monitoring bromine at m/z 79; this is the protocol stated in EPA method 321.8.

This work focuses on bromide/bromate speciation by ion chromatography using Dynamic Reaction Cell (DRC) ICP-MS as the detector. A faster chromatographic method is developed than presented in the current version of method 321.8, more closely matching the capability of the detector. Stability and repeatability of the bromate signals over several days are demonstrated. Possible interferences and the detection limit achievable will be discussed.

# Dynamic Metal Speciated Analysis such as Cr(VI) and Alkylmercury Examined and Applied

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### ABSTRACT

Elemental speciation is one of the most challenging analytical measurements. To make matters worse, there is a devastating lack of both standards and diagnostic tools inhibiting the progress of the field. Some elemental species undergo conversion or degradation of the species of interest during sampling, storage and the measurement steps. Until recently there have been no diagnostic tools to trace the fate of species since conventional speciation methods can only measure the species' concentrations in the final solutions at the time of measurement. Knowing the transformation of the species is critical in the preparation and certification of standard reference materials and for accurate speciated measurements. Other countries such as the European Community through EVISA have reached these conclusions and have supported only isotopically traceable solutions are effective diagnostic tools. Speciated isotope dilution mass spectrometry (SIDMS), which addresses the correction for such degradations or conversions and its use in validation will be demonstrated and discussed (1, 2). It has been demonstrated to accurately determine the species concentrations at both the time of spiking and measurement. The method also has the ability to perform diagnostic analysis isolating specific procedural protocol steps in specific matrices enable their evaluation for species shifting potential. SIDMS has the potential to be used as a diagnostic tool to validate other methods and to evaluate speciated standards. By spiking the sample at each step with enriched stable isotopes of the same species, SIDMS can be used as a diagnostic tool to identify the steps at which the species are altered (3).

As examples, SIDMS has been applied to monitoring the fate of Cr(III) and Cr(VI) in processing samples using EPA method pair 3060A/7196A, which has been used for the quantification of Cr(VI) in solid samples. Method 3060A includes extracting Cr(VI) from samples in an alkaline solution, filtering the extracts and neutralizing the filtrates. Method 7196A is a colorimetric detection method, using diphenylcarbazide to complex with Cr(VI) which forms a purple product that is usually measured at pH 2. IC and HPLC ICP-MS were used as detection methods in this study (4).

The results of this study showed that classical methods may not be able to detect alteration of the species from difficult matrix samples, and that neutralization and measurement steps can contribute to transformation of species. In specific and difficult matrices, Cr(III) could be oxidized to Cr(VI); and, during neutralization, Cr(VI) could be reduced to Cr(III). The degree of the species' conversions is highly dependent on the sample matrix and the instrument's operating condition. When using EPA method 7196A to quantify Cr(VI) in some soil extracts, low recoveries were observed. SIDMS, however, obtained 100% recoveries despite the reducing matrices. SIDMS has successfully identified and corrected these conversions,

demonstrating that SIDMS is a more appropriate method in speciation analysis and that it is also a diagnostic tool for other speciation methods. Applications in coal fly ash have been studied extensively and are widely applicable across the nation. The study in this matrix will show that Cr(III) is oxidized to Cr(VI) in coal combustion processes and that it then is stable in the environment and is released as run-off from fly ash sites (5). Road aggregate materials that leach Cr(VI) and sediments where the Cr(VI) is deposited are examples of methods specific matrix conversions (11).

Mercury has recently become the focus of debate on transformations between methylmercury and inorganic mercury during the measurement process (6). SIDMS is a method that can be generalized for many poly isotopic species that have the potential to be transformed from species to species during the evaluation process such as Cr, Hg, Se, Sn and other species where multiple isotopes (in species relevant forms) provide the necessary resources to make these measurements. Standards may also be produced with isotopically enriched species that may be evaluated prior to use if processes alter the species forms (7, 8).

Field environmental examples are presented to demonstrate the effectiveness of SIDMS for Cr in the coal fired power industry. SIDMS has been standardized and approved as a new EPA method "6800" (9, 10). This new EPA method is intended to assist with some of the uncertainty in speciated environmental measurements. The general method is described and applications to both Cr species and Hg species will be discussed.

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# Session 12

# **Managing Uncertainty**

### Managing Decision Uncertainty Resulting from Hydrogeologic Heterogeneity in Groundwater Contamination Investigations

### Seth Pitkin

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### ABSTRACT

The Triad Approach focuses on managing uncertainty in decision-making relative to management of the site. Decision uncertainty stems primarily from two types of uncertainty associated with the data: sampling uncertainty and analytical (or measurement uncertainty). Analytical uncertainty is well understood and is controlled at low levels. Sampling uncertainty arises from the heterogeneity inherent in natural hydrogeological systems. Examples include the spatial structure of: hydraulic conductivity controlling groundwater flow; capillary pressure controlling non aqueous phase liquid movement; and the very weak nature of hydrodynamic dispersion in directions normal to the primary groundwater flow direction, resulting in very steep concentration gradients.

Conventional techniques used to investigate groundwater contamination, such as monitoring wells result in depth-integrated, flow weighted average concentrations and large spacings between samples that result in a high level of uncertainty in the conceptual site model and hence in decision making. A primary means of reducing sampling uncertainty is through the use of tools and techniques that provide many more data points at a more appropriate scale than conventional methods.

The Waterloo Profiler is a direct push groundwater sampling tool that has been modified to allow for the collection multiple data sets that are used collaboratively to test and revise the conceptual site model. The modified Waterloo Profiler provides discrete groundwater samples at virtually any vertical spacing while developing a continuous log of the Index of Hydraulic Conductivity as well as hydraulic head distributions and specific conductance, pH, oxidation/reduction potential and dissolved oxygen of groundwater.

A Triad investigation team incorporated source zone data collection in the vadose zone using a passive soil gas survey, membrane interface probe explorations and conventional soil coring and onsite analyses along with the integrated data sets provided by the Waterloo Profiler below the water table to revise a conceptual site model through a three week investigation. The uncertainty that had been hindering use of the site for over a decade was reduced as a result of the investigation and the stakeholders were able to move forward.

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**Seth Pitkin** is a Senior Hydrogeologist and Leader of the Investigation and Remediation Group at Stone Environmental in Montpelier, Vermont. He received a B.S. in Geology from the Evergreen State College in 1984 and a M.Sc. from the Department of Earth Sciences at the University of Waterloo in 1994. Mr. Pitkin was involved with the development of the Waterloo Profiler at the University of Waterloo and over the ensuing years has adapted, expanded and improved the profiler. He has over 19 years of experience in the investigation of groundwater contamination, predominantly chlorinated solvent plumes in porous media and twelve years of intensive experience in Triad-type site investigations.

### Chemical Measurements Traceability, Validation and Uncertainty

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### ABSTRACT

In environmental testing one of the data quality indicators required is comparability. The predominant process to ensure comparability has been based on performing the same method and demonstrating the same quality control performance limits. The international community is establishing a uniform process for ensuring chemical measurements achieve comparability by a defined process for traceability. This process involves the need for method validation and the expression of the estimated uncertainty.

To achieve comparability of results, a link is needed for all the individual measurement results to some common, stable reference or measurement standard. Results are compared through their relationship to that reference. The linking of results to a reference is termed "traceability." The definition of traceability as found in the International Vocabulary of Basic and General terms in Metrology (VIM) is the: "property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

In an increasingly regulated environment attempting to move to a performance approach rather than a method based process, laboratories are under pressure to demonstrate that their use of measurement method is indeed both appropriate and sufficient. This can be achieved through the use of the appropriate reference standards. Many of the physical quantities used in routine chemical measurement are supported by extensive and effective calibration and traceability systems, making the traceability for these quantities straightforward. The values of chemical quantities involved are typically drawn from a wide range of reference materials and data with varying sources and unknown origins. The selection is often based on the statement on the certificate that it is "NIST traceable" with little regard or knowledge how this is established and without any stated uncertainty.

Chemical measurements require confirmation of identity and amount of the element, compound or mixture. Further, it is not uncommon for the measurement of the element, compound or mixture in complex matrices, to include chemical results that arise from the measurement of operationally defined parameters, for example "Toxicity Characteristic Leaching Procedure [TCLP] or Oil and Grease) (sometimes called "empirical" measurements or method defined parameters). In such circumstances, it is not always so straightforward to identify the requirements for traceability, or to demonstrate that the traceability in place is adequate, since the current process is to perform the method without modification. The problem is when the matrix being measured or options are found in these methods require method modification to

achieve performance. The result is confusion as to what is allowed and not allowed when method modifications are made by the testing organization.

This talk presents the key elements in achieving traceability including the process for method validation and estimating the uncertainty in order to ensure the comparability, identity and amount of the environmental chemical measurements used for making the decision.

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#### A Harmonized National Accreditation Standard: The Next Step for INELA Field Activities By Dawn D. Thomas, ASO COM

The original charter of the National Environmental Laboratory Accreditation Conference (NELAC), when established in the early 1990's, was to "foster the generation of environmental laboratory data of known and documented quality through the development of national performance standards for environmental laboratories". However, it has been generally recognized within the environmental community, over the years, that the quality of environmental laboratory data can only be assured if minimum performance standards exist for field sampling and measurement activities - the "front-end" of the environmental data generation process. To assure the production of environmental data that are scientifically valid and can be used with a high degree of confidence by the end-user, control of environmental laboratory analytical processes and field sampling and measurement processes are of equal and significant importance. Accordingly, in July 1998, the Constitution of NELAC was amended to reflect the growing interest of many stakeholders to expand its scope to include both field sampling and measurement activities. Subsequent to this Constitutional amendment, the Field Activities Committee was officially established in 1999 as a NELAC standing committee responsible for the development of performance standards applicable to those organizations performing field sampling and measurement activities.

In July 2002, Chapter 7, *Field Activities Standard*, was added to the NELAC Standard to address minimum quality and technical requirements for field sampling and measurement activities. The initial draft of this chapter excerpted selected verbiage from Chapter 5, *Quality Systems*, of the NELAC laboratory standard and did not specifically address other accreditation components (e.g., proficiency testing (PT), on-site assessment, and accreditation process) or requirements for sampling specific environmental matrices. In 2003, NELAC divested itself of the environmental standards development process and the Institute for National Environmental Laboratory Accreditation (INELA), a consensus based standards development organization, was formed. Within this organization, the INELA Field Activities Committee (FAC) was established to continue the standards development work for an accreditation program designed specifically for field sampling and measurement organizations (FSMO).

#### **Objective and Goals**

The primary objective of the INELA FAC is "to develop and maintain consensus accreditation standards and guidance materials for organizations engaged in environmentally related field sampling and measurement activities, consistent with regulatory and industry-specific requirements". Its long-range focus is to replace the 2002 NELAC *Field Activities Standard* (Chapter 7) with an INELA stand-alone, FSMO-specific accreditation standard(s) that meets the following goals:

- · Encompasses broad scope and wide ranging applicability;
- Based on internationally recognized standards for competency (ISO/IEC 17025) and conformity assessment (ISO/IEC 17011);
- NOT prescriptive in nature, allowing for the development of FSMO-specific policies and procedures; and
- Effectively supported by sound guidance.

#### A Harmonized National Accreditation Standard: The Next Step for INELA Field Activities By Dawn D. Thomas, ASQ CQM

#### Broad Scope and Applicability

If the INELA FAC is to meet its objective of establishing performance standards for those collecting samples and conducting on-site measurements for improved environmental data quality, then the standard must be wide-ranging in scope and applicability to support existing and future state/federal environmental regulations governing field sampling and measurement activities. To this end, a primary goal of the INELA Field Activities Committee is to develop an accreditation standard (or series of standards) that will apply to organizations performing field activities for a wide variety of sampling and measurement media such as air, biological, water, soil, waste, and radiological. Due to the nuances, specific to each media, a "one size fits all" approach to standards development is not appropriate. Accordingly, the FAC has engaged field sampling and measurement "media experts" to collaborate on the development of customized, media-specific FSMO accreditation standards. The development of custom field standards for water and air are the current focus of the committee.

#### **ISO Foundation**

It is the consensus viewpoint of the Field Activities Committee that the common denominator, or foundation, for the custom, media-specific INELA FSMO accreditation standard(s) must be ISO/IEC 17025, <u>General Requirements for the Competence of Testing and Calibration</u> <u>Laboratories</u> and ISO/IEC 17011 (soon to replace ISO/IEC Guide 58), <u>Conformity Assessment – General Requirements for Accreditation Bodies Accrediting Conformity Assessment Bodies</u>. Using this approach to standards development, the role of the INELA FAC will be to utilize its "media experts" to determine **how** to best apply these generic International Standards for a particular area of accreditation (e.g., field activities – water). The INELA FAC "application" of these International Standards, for each sampling and measurement media, will include, but will not be limited to, provisions for additional requirements, exclusion of specified ISO requirements. Using ISO as the foundation for custom-built FSMO accreditation standards facilitates harmonization of individual field standards specific to each sampling and measurement media.

#### Non-Prescriptive Standards Development

Although sampling has, historically, been recognized as a major contributor to the overall measurement error, many organizations performing field sampling and measurement activities today are not currently subject to rigorous and prescriptive quality system requirements, accreditation, or routine oversight. Accordingly, the committee consensus was to take a practical and realistic first step towards improved environmental data quality by establishing an accreditation standard, based on internationally recognized standards, which are minimally prescriptive to provide a high degree of flexibility for the FSMO when implementing the standard requirements. Simply stated, applying this "less is better" approach, the FSMO will be able to craft policies and procedures, which meet the intent of the INELA standard, but are practical, functional and, most importantly, implement-able. The INELA FAC believes that if the resulting field accreditation standards cannot be effectively implemented by all parties

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affected, large and small, public and private, due to overly prescriptive requirements, then we, as a committee, have not successfully completed our mission for improving data quality for better decisions.

#### Sound Guidance

To support the "less is better" approach to standards development and to facilitate successful implementation by all FSMO impacted by the standard, the development of appropriate implementation guidance tools is a key component for realizing an improved outcome – sound and defensible data quality for better decisions. This is the long-term focus of the INELA Field Activities Committee - to "show the way" by providing the necessary guidance and support for standards implementation. Several of the many benefits associated with this INELA service to the environmental community include:

- Acceleration of the FSMO "learning curve" associated with "something new", keeping in mind that many FSMO have not been subject to quality system/accreditation program requirements, historically;
- Improved "buy-in" by minimizing the costs associated with implementation of a new and comprehensive accreditation standard; and
- Consistency of standards interpretation and implementation.

#### Accomplishments

These goals for standards development, as discussed in the previous sections, have evolved over a period of two (2) years as a result of the diligent work and "outside the box" thinking of the INELA FAC. The accomplishments, which follow in this section, have contributed greatly to the refocusing of the laboratory community (regulators and those regulated) on the importance of field sampling and measurement and its role, as the "front-end" portion of the environmental data generation process.

To facilitate the development of media-specific field standards, the committee has been very active in outreach activities to engage more stakeholders – the "media experts" - in the standards development process. The INELA FAC has grown from less then ten (10) members in 2003 to more than thirty (30) participating members today. The committee has also worked to achieve balance of membership, necessary for a consensus standards development organization, with representation from government and municipal agencies; engineering and environmental consulting firms, analytical laboratories and industry. Participation in national/regional conferences and collaboration with other organizations representing specific stakeholder groups will continue to be a focus for the INELA FAC. The committee's success in developing sound field accreditation standards depends on the continuation of these outreach activities.

Consistent with committee direction to develop "applications" of the ISO/IEC 17025 and 17011 standards, a generic (not specific to any one media) application of the ISO/IEC 17025 standard has been completed and will be utilized by the "media experts" to guide the development of media-specific field accreditation standards. This generic application of ISO/IEC 17025 was

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affirmed by the INELA membership in late 2004. Additionally, the groundwork, in the form of a consensus-based conceptual model, for the application of the ISO/IEC 17011 standard was completed and presented at the INELA Accreditation Forum in Charleston, South Carolina last summer. Building on these endeavors, workgroups have been established and are tasked with producing the first Working Draft Standards for a generic application of 17011 and a media-specific (water) application of 17025 by the summer of 2005.

A great deal has been accomplished but there is more work to do.

#### Next Steps

To achieve its on-going objective "to develop and maintain consensus accreditation standards and guidance materials for organizations engaged in environmentally related field sampling and measurement activities, consistent with regulatory and industry specific requirements", the INELA Field Activities Committee must effectively meet certain challenges. They are:

- To know, engage and understand the needs of all stakeholders who will be, ultimately, impacted by the standard(s).
- To know, engage and understand the needs of all potential clients, those who will adopt and implement such a standard(s).
- Finding a consensus viewpoint to the question of *what makes for good quality* to achieve consistent application of the ISO/IEC 17025 and 17011 standards for harmonized individual media-specific field accreditation standards.

With its new approach to standards development, the INELA FAC also has an opportunity to help chart the future path of INELA, as a standards development organization. At the 2004 INELA Summer Forum in Charleston, South Carolina, the INELA Board of Directors expressed their desire for INELA membership to seriously consider a restructuring of the NELAC laboratory standard to better meet the needs of stakeholders, existing and potential clients, and to achieve the desire growth into other areas of accreditation. There are a number of proposals for this restructuring initiative currently being considered by the INELA Board.

One of the proposals being considered has been developed by the INELA FAC, which details an approach to standard restructuring, consistent with the approach being taken for the development of media-specific field accreditation standards. This proposal has been designed to:

- Align with the INELA Strategic Plan.
- Provide a flexible framework for the development of harmonized accreditation standards in new areas such as Homeland Security.
- Positively impact a wide range of stakeholders.
- Appeal to accrediting authorities, regulators, private sector groups interested in adopting and implementing uniform standards of accreditation.
- Assure the production of scientifically valid data that can be used with a high degree of confidence by the end user.

#### A Harmonized National Accreditation Standard: The Next Step for INELA Field Activities By Dawn D. Thomas, ASQ COM

The INELA Field Activities Committee is committed to the development of field accreditation standards using the approach detailed in this paper and strongly believes that this approach can be effectively used for the development of new INELA standards in other areas of accreditation as well. To meet the current challenges and to adequately address the complexities of the field sampling and measurement "world", the committee must continue to focus its energies on thinking "outside the box", encouraging and listening to new ideas, and creating an environment where these new ideas can flourish. Your participation in the FAC activities is vital for the production of data suitable for its intended use **and** may have an influence on the future path of INELA as a consensus standards development organization. All are encouraged to join INELA and to get involved! More information on the efforts of the INELA FAC may be found on the INELA web site (www.inela.org).

© Authored by Dawn D. Thomas, ASQ CQM, 2005. Thomas is a corporate Quality Assurance Manager for Professional Service Industries, Inc., a national engineering consulting firm, in Orlando, Florida and is certified by the American Society for Quality (ASQ) as a Quality Manager (CQM). She is the current Chairperson of the INELA Field Activities Committee and is a member of the INELA Board of Directors.

### LABORATORY SUBSAMPLING STRATEGIES FOR SOLID SAMPLES: CONTROLLING ANALYTICAL MEASUREMENT UNCERTAINTY

### ABSTRACT

Traditional approaches to environmental laboratory subsampling usually result in inaccurate analytical measurement results. Correct subsampling that includes multiple increment sampling helps control sampling error. A material preparation and subsampling strategy that takes into account compositional and distribution heterogeneity of the sample matrix and environmental analytes can be developed to minimize analytical measurement uncertainty.

### BIO

Bill Ingersoll is a Chemist with the Naval Sea Systems Command, Programs Field Office (SEA 04XQ/LABS) located in Charleston, South Carolina. Mr. Ingersoll has worked as a chemist in environmental sampling and analysis for 15 years. His work for NAVSEA includes technical and scientific assistance for Navy laboratory and engineering personnel, and environmental laboratory assessment to support the Navy IR QA program. Bill is a member of the DoD joint Environmental Data Quality Work Group (EDQW), Institute for National Environmental Laboratory Accreditation (INELA) and serves on the Proficiency Testing Board of the National Environmental laboratory Accreditation Conference (NELAC).

### BACKGROUND

Scientifically valid environmental decisions require quality data. Data quality can be defined as the ability of data to provide information that meets the data-users requirements. Generation of quality data for environmental decision-makers and data-users is the goal of environmental laboratories. For laboratories to achieve quality data, the data must be accurate. Minimizing analytical measurement uncertainty and bias helps ensure that accurate data are generated in the environmental laboratory.

General particulate sampling theory developed by Pierre Gy attributes sampling error to:

- Large-scale errors (trends and cycles),
- Materialization errors<sup>1</sup> (material preparation, delimitation, and extraction errors), and
- Small-scale errors (fundamental error, and grouping and segregation error).

Materialization error, and fundamental error and grouping and segregation error affect laboratory uncertainty and bias. Generally, the laboratory components of analytical measurement can be broken down into material preparation and subsampling, and chemical preparation and instrumental analysis. Material preparation and subsampling are frequently the overlooked components in the equation for determining and controlling laboratory analytical measurement uncertainty. Traditionally, resources are concentrated on more and more sophisticated instrumental techniques while subsampling is relegated to entry-level personnel with minimal training. Sophisticated instrumental analyses combined with incorrect subsampling

<sup>1</sup> Both primary field sampling and secondary laboratory subsampling affect materialization error. However, the laboratory usually cannot control field-sampling effects on materialization error.

techniques results in inaccurate data. Correct subsampling requires as sophisticated a strategy as instrumental analysis to control analytical measurement uncertainty and bias. Choosing the correct subsampling approach minimizes uncertainty and bias and improves data quality.

Subsampling bias and uncertainty result from heterogeneity of the sampled material. Fluids such as gases and liquids are collections of large numbers of randomly distributed molecules. Single-phase aqueous samples therefore do not present a significant challenge to collecting representative samples. However, soils, sediments, and solid wastes are usually heterogeneous collections of compositionally and distributionally variable arrangements of particles. Heterogeneity (compositional and distributional) of soils makes representative sampling and subsampling a significant challenge. Compositional heterogeneity results when particles with different sizes, shapes, densities, and chemical properties have different environmental analyte concentrations. Distributional heterogeneity results when analytes tend to clump and stratify within the sample material.

### SUBSAMPLING STRATEGIES

The strategy for selecting a laboratory subsample from a field sample requires an approach that ensures representativeness. Traditional subsampling techniques such as selecting a single 1-gram increment "off the top" of the sample container is usually non-representative. To achieve representative subsampling, every particle must have equal probability of selection. That includes the entire sample container subsampled from top to bottom. Laboratory techniques including drying, mixing, and grinding, and multiple increment subsampling can be used to ensure that representative subsamples are selected. Multiple increment subsampling is the technique where many small portions of the sample are selected from the entire sample material to make up the subsample. Materialization error in the laboratory can be controlled by correctly delimiting and extracting increments without cross-contamination or losses. The choice of the material preparation and subsampling techniques depends upon the sample matrix, the type of sample preparation and analysis performed, the environmental analyte(s), and the measurement quality objectives for a specific project. No technique or suite of techniques is correct for all matrices and analytes. For example, samples tested for volatiles cannot be dried, mixed or milled.

### VOLATILES

For correct sampling of volatile organic compounds the sample must not be exposed to air or elevated temperature. Traditionally, the collection of solid matrices for volatile analyses resulted in significant negative bias from volatilization during the sampling processes. Research indicates that losses of as much as 3-orders of magnitude between replicates result from incorrect volatiles sampling.

Correct sampling requires minimizing exposure of the sampled material to ambient air or elevated temperature. Traditional sampling techniques disturbed the soil structure integrity. The material disturbance increases the surface area exposed to air resulting in volatilization, and the disturbance promotes degradation by oxidation and microbial activity of the volatile organic compounds. Method 5035 was developed to reduce VOC losses attributable to volatilization and degradation during traditional sample collection, handling, and preparation techniques. This approach provides a closed-system for collection in vials that minimizes disturbing soil structure. EnCore® study data demonstrates that sampling variability is negligible when field

cores are sealed in closed-system vials. Variability of analytical measurements for replicates decreased from 3-orders of magnitude to a relative standard deviation at the 95% confidence level ( $RSD_{95\%}$ ) of ~ 20%. The 20% can be attributed to the instrumental analysis variability independent of matrix interference.

### SEMIVOLATILES

For correct subsampling of semivolatile organic compounds the sample must be homogenized before subsampling. This can be accomplished by mixing the sample material before multiple increment sampling. Mixing breaks up grouping and segregation of the sample matrix and analytes, and disperse the analytes randomly throughout the sample prior to subsampling. Although exposing sample material to air during the subsampling process does not significantly volatilize semivolatile organic compounds (SVOCs), wet samples cannot be dried at elevated temperature (above ambient temperature). The problem of material preparation and subsampling of wet samples can be overcome by drying the sample with anhydrous sodium sulfate. Mixing anhydrous sodium sulfate with the sample until the material is free-flowing helps in homogenization.

In a study conducted by Analytics Environmental Laboratory LLC of New Hampshire, soil samples were subsampled for PCB testing. Aroclor 1260 concentrations for the different samples ranged from 500 ppb to 15,000 ppb. Without mixing the soil samples, the RSD<sub>95%</sub> was 240%. However, when to samples were homogenized by hand mixing, the RSD<sub>95%</sub> was reduced to 30%. The 30% can be attributed to the chemical preparation and instrumental analysis variability, and the subsampling contribution to analytical measurement uncertainty was negligible.

### SOLID PARTICLES

For correct subsampling of solid particles the sample must be homogenized and particles size must be reduced before subsampling. The compositional heterogeneity increases with the increase in particle size. To decrease compositional (and distributional) heterogeneity particle size must be reduced. This can be accomplished by milling or grinding the sample. Particle size reduction minimizes fundamental error that is caused by compositional heterogeneity. The compositional heterogeneity increases with the increase in particle size. To decrease compositional heterogeneity increases with the increase in particle size. To decrease compositional heterogeneity increases with the increase in particle size. To decrease compositional (and distributional) heterogeneity particle size must be reduced or the analytical subsample size increased. Analyzing a larger subsample can also help minimize fundamental error. However, larger subsamples may not be practical because of matrix interferences affecting chemical preparation and instrumental analysis.

Metallic lead fragments and explosive residue are examples of solid environmental analytes that may range from dust size to gravel size particles. In a USACE-CRREL study for explosive residue subsampling, the analytical measurement variability was minimized by particle size reduction. When the soil sample was not ground prior to subsampling, the RSD<sub>95%</sub> was 600% for TNT. However, when the sample was ground in a milling machine, the RSD<sub>95%</sub> was reduced to 12%. The 12% can be attributed to the chemical preparation and instrumental analysis variability, and the subsampling contribution to analytical measurement uncertainty was negligible. Increasing the subsample mass from 2 grams to 50 grams (without particle size reduction) only reduced the RSD<sub>95%</sub> to 300%.

# Intrinsic Reliability — A Metric for Describing Confidence in Measurements

### Molly Isbell, David L. Lewis

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### ABSTRACT

A critical need for any environmental sampling program is to describe the reliability of measurement results. For traditional environmental sampling and monitoring programs where the focus is on quantitative measurements of chemicals in the environment, measurement reliability metrics (e.g., precision, quantitative accuracy, detection limits) are typically well established. However, due to a fundamental difference in objectives, these metrics are not always directly applicable in the context of monitoring for threat agents. In a program designed to detect threat agents in the environment, it is necessary to know the likelihood that a positive detection is a false alarm. The concept of *Predictive Value* addresses the need to address such questions and is widely accepted in the clinical testing context, where positive predictive value (PPV) describes the probability that a patient actually has a disease, given a positive test result. In the context of threat-agent detection, PPV describes the likelihood that an agent is present, given a positive test result.

Calculation of predictive value requires knowledge of false-positive and false-negative rates, which can be determined through positive and negative control samples and through proficiency tests and validation testing. However, predictive value also requires information about the *a*-*priori* likelihood of the threat agent in the environment, which is often very uncertain.

Predictive value equations can be expressed such that they are separable into one component that describes the *a-priori* likelihood that the target is present and one that describes the reliability of the measurement process. Because the two components can be separated, it is possible to compute a statistic (*Intrinsic Reliability*) based only on the reliability of the measurement process, which can be determined experimentally and does not depend on *a-priori* information. This Intrinsic Reliability statistic therefore describes the value of a reported detection in determining the likelihood that the threat agent is actually present.

I prefer to give an oral presentation.

### Vanishing Zero Defects

### Dr. John Long

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### ABSTRACT

Resourceful environmental monitoring has to be a consequence of realistic policy protocols that. in turn, are developed from sound scientific information. Beyond the conclusions that can be drawn from toxicological data, many other factors can play important roles in determining both advisory and legislative guidelines. These include uncertainty factors, which may give special consideration to test regimens, extrapolation of data from animal to human conditions, or accountability to certain subpopulations, for example. However, one particularly important factor has been changing with time and needs to be made a priority by the scientific community: the growing distance between the sensitivity of toxicological studies (typically parts per million of target substrate) and the thresholds of analytical instrumentation used to measure the substrate (frequently parts per trillion). This "detection limit creep" and the divergence of the data trails have made it increasingly difficult to relate some environmental conditions to an accurate (or unbiased) assessment of probable risk. The greater the gap between the respective data thresholds, the greater the opportunity for manipulation based on political or personal motivations, and subsequent abuse of scientific methods. The example of perchlorate in the environment is instructive: eminent toxicologists have gone on record indicating that exposure in the general U.S population to perchlorate at levels equivalent to 0.2 parts per million should be considered safe, while other voices have demanded that the public be protected to one part per billion. The EPA's February, 2005 determination that 0.7 microgram per kg of body weight represents a safe dose for human ingestion of perchlorate. This presentation takes a deeper look at all the factors at work in the controversial and very political process that led to this decision.

I prefer to give an oral presentation.

Session 13

# Advances in Preparation and Analysis of Organic Compounds

NEMC 2005 Proceedings



## Pursuit of Practical Particle Size Reduction and Sub-sampling for the Environmental Testing Laboratory

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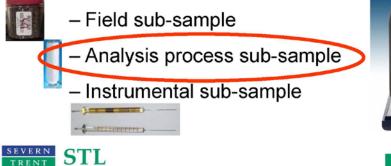
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NEMC 2005 Washington D.C. July 27, 2005

Consultation with experts is recommended, do not rely solely on this presentation for guidance.

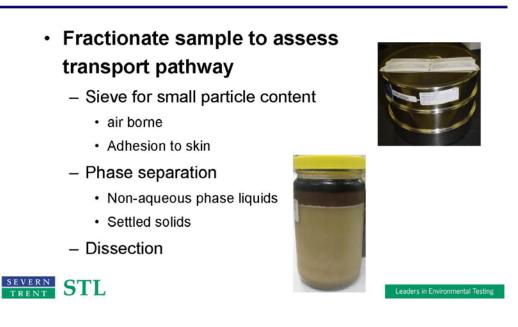
# Why Sub-sample?

 Impossible to put the entire test area or quantity through the measurement process





# Why Sub-sample?



# **EPA Guidance Document**

- Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples.
- EPA/600/R-03/027
- November 2003
- 134 pages
- Non-volatile analytes only



# **Representative Sub-sample**

### Laboratory sub-sample

- Tug-o-war match

statisticians ------ analyst -----waste, cost & labor minimizers



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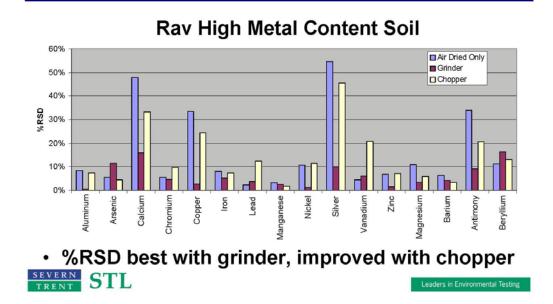
# **Representative Subsample**

- How do we keep everybody happy?
- Particle size reduction
- Mixing
  - Smaller sample
    - Represents bigger sample
    - Better precision
    - Accuracy
      - same analytes
  - same concentrations



Leaders in Environmental Testing

# **Precision Improvement**

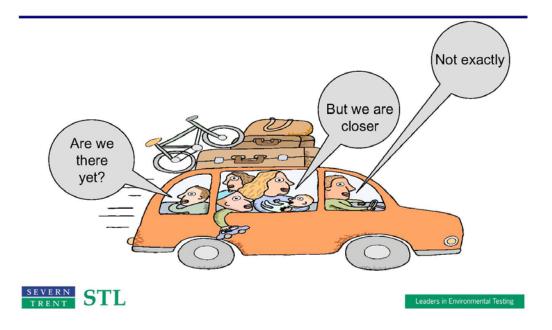


# The Ideal

- Place sample jar as received from the field into a magic homogenizer
- Run for a few minutes
- Then any 0.5 to 30 g aliquot accurately represents the whole
  - i.e. dig-a-spot technique works



# **Status Report**



## How To Do Particle Size Reduction?

- Form suitable to grind, crush or chop
  - Dry sample
    - Room temperature
    - Oven
  - Wet sample
    - Slurry grinding or chopping
  - Freeze sample
    - Dry ice



# What does homogenization and sub-sampling mean?

- Stir and dig multiple spots
- Air dry, grind, stir & dig
- Sieve, cone & quarter, line & scoop
- · Air dry, chop, sieve, shake, dig

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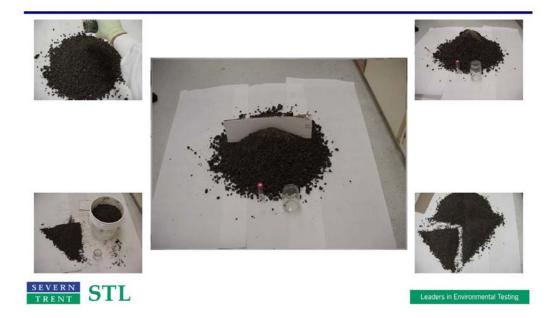
# Air dry, grind, stir & dig



## Sieve, cone & quarter, line & scoop



# Sieve, cone & quarter, line & scoop



## Sieve, cone & quarter, line & scoop



# Air dry, chop, sieve, shake, dig

- Large capacity air drying
- Approx. 500
   1 kg samples





## Air dry, chop, sieve, shake, dig



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### **3 Cup Chopper**



# Air dry, chop, sieve, shake, dig





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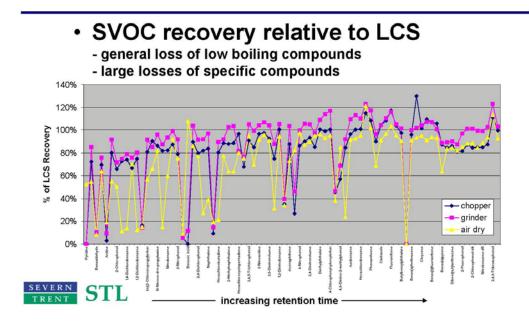
# **Application to Organic Analytes**

- Low vapor pressure organics
  - Explosives
  - PCBs
  - Air drying usually acceptable
- Semi-volatile organics
  - PAHs
  - Substituted phenols
  - Chlorinated aromatics
  - Amines
  - Phthalates



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## **Accuracy Concerns**



### **Accuracy Concerns**

### Lost analytes (negative bias)

- Thermal degradation
  - · heating due to friction
  - · less stable analytes
    - hexachlorocyclopentadiene
    - organophosphorus pesticides
    - endrin & DDT
- Volatilization losses
  - · air drying
  - grind / chop process

low boiling semivolatiles 1,4-dioxane pyridine N-nitrosodimethylamine aniline phenol dichlorobenzenes naphthalene

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# How to Homogenize a Clay Mud Ball?

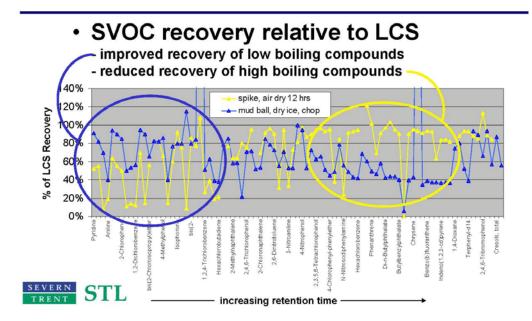
- Flashback to Supercritical Fluid Extraction
  - $-CO_2$
  - Agilent (Hewlett Packard)
  - Dennis Gere
  - Mix CO<sub>2</sub> snow and wet sample in a blender



## Mud Ball >> Mixable Powder



# **Dry Ice Chopping Results**



## "Magic Mill"

- Matrix Spike, single spot on wet clay
- 1.5 minutes homogenization time
- Temperature: 20°C >> ~40°C

   friction
- Moderate air exposure
  - similar to chopper

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# "Magic Mill SVOC Results"

- Average %RSD: 16%
  - Good precision for dig-a-spot sub-sampling

### Analyte recovery

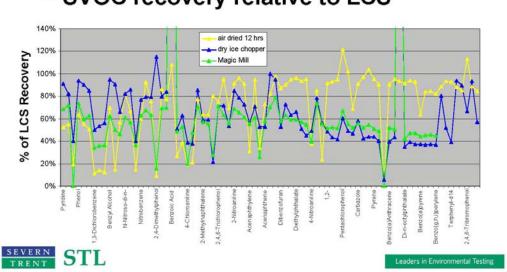
- Low boiling analytes
  - > air dried
  - < dry ice chopped</li>
- High boiling analytes
  - · Low recovery like dry ice chopped

### Contamination

- Benzoic acid and bis-2-ethyl hexyl phthalate
  - · Plastic parts in chopper and magic mill



# "Magic Mill Results"



## SVOC recovery relative to LCS

# **Conclusions - Pursuit of Practical**

- Practical now
  - Air drying of samples up to 1 kg
  - Sieving dried samples up to 1 kg for metals, PCBs
  - Chopping dried samples up to 200 g for metals, PCBs



# **Conclusions - Pursuit of Practical**

### Potentially practical

- Chopping dried samples up to 1 kg for metals, PCBs
- Grinding dried samples up to 1 kg for metals, PCBs
- Sub-sampling by cone & quarter, line & scoop



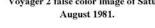
# **Conclusions - Pursuit of Practical**

- Still in pursuit
  - Chopping, grinding, milling of
    - samples with semi-volatile analytes
- Holy Grail
  - Universal homogenization technique
    - in the original sample container



# **Parting Thought**







"It takes work to bring order to a cloud of dust. Applications are being accepted now."



Sandstorm in China, April 2001, ESA

Leaders in Environmental Testing

STL



### **Acknowledgments**

Frank Calovini, Becki Strait, Tom Hula, Darren Miller, Frank Gallegos, Pat O'Meara, Karen Counts, Larry Williams, Al DiPofi, Katy Tucker, John Donat

> Samir Mansy, Paul Zorko, John Jent



US Army Corps of Engineers®

# An Innovative Approach to Automatic Solvent Drying and Concentration of Environmental Extracts

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### ABSTRACT

Two steps that have a major impact on the recoveries for both liquid-liquid (LLE) and solid phase (SPE) extraction techniques are drying and concentrating the extract prior to GC analysis. Residual water must be removed to prevent the extract from separating into multiple phases and back extraction of water soluble analytes. The extract must also be concentrated to improve detection limits by selectively evaporating the extraction solvent, without inducing loss of the more volatile components. Drying extracts has historically been accomplished manually with sodium sulfate. Currently, properly optimized hydrophobic membranes are available that can provide automated removal of residual water. Further, this step can be incorporated into equipment that selectively evaporates the extraction solvent to completely automate sample drying and concentration for GC analysis. The use of such equipment for environmental applications will be discussed. Emphasis will be placed on analyte recovery, carryover, and sample throughput.

# Using Headspace Trapping Technology for Measuring Environmental Volatile Organic Compounds (VOCs) by Method EPA8260B

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### ABSTRACT

Environmental methods, such as 8260B have traditionally been complex and time-consuming to perform. New regulations often add target analytes and lower the concentration levels of interest. In addition, productivity pressures force laboratories to look for more efficient ways to generate quality data. Therefore, successful environmental labs have moved to become automated, fast, and precise.

A large number of EPA methods have required a purge and trap methodology to extract volatile compounds from environmental matrices. Most labs find purge and trap instruments difficult to use, and high in maintenance. However, because of the trace detection levels required in EPA methods it has been difficult to find an alternative extraction method to this system, until now.

New headspace trap technology gives operators the benefits of traditional headspace and now adds a trap option to meet the needs of lower detection limits. This trap technology is capable of sampling up to 100% of the headspace by a pulsed pressure headspace extraction process with analytes refocusing on an adsorbent trap. The system uses a unique dry purge technology to remove the water vapor and uses overlapping thermostating, to produce maximum throughput. Using heating instead of purging to extract the volatile components makes the gas chromatographic run time the time limiting factor, rather than the introduction system.

EPA 8260B results will be presented using the headspace trap equipment connected to a GC/MS. Instrument calibration, repeatability, linearity, response factors, and minimum detection limits will be demonstrated. Fuel oxygenates analysis with the trap system will also be shown. As well as, other results using a salting technique to reach low ppb (parts per billion) detection limits for tough compounds.

### How to Improve Detection Limits, Reduce Maintenance Time and Minimize Breakdown for Pesticide Analysis and Other EPA Method 8270 Analytes using Enhanced Large Volume Injection

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### ABSTRACT

The ability to improve detection limits in environmental analyses is quite advantageous. There are several techniques employed to optimize the analysis of pesticides with Mass Spectrometry (MS) and/or Electron Capture (ECD) detection.

To attain our goals, the parameters employed are Enhanced Large Volume Injection (injector isolation and backflush modes) and simultaneous Full Scan and Single Ion Monitoring.

With Enhanced Large Volume Injection, the analyst has the ability to inject large volumes of sample into the Gas Chromatograph. During the solvent purge time, the injector is completely isolated from the analytical column thereby preventing solvent from reaching the column and detectors. This enables the use of less sample and less extraction solvent while maintaining and exceeding the necessary detection limits.

In addition, isolating the injector, allows for the use of chlorinated solvents with Electron Capture detection. Isolating the injector after the injection has been made, enables the baking and maintaining of the injector port during the analysis.

With a program injector, the analytes of interest (in this case pesticides), experience controlled evaporation minimizing thermal breakdown inherent in classical flash injections.

Two advantages will be discussed -- the ability to increase sensitivity at least 25x, and the ability to enhance integration improving precision.

### Improved Sensitivity and Analysis Time for Semivolatile Organic Compounds Using GC-TOFMS: Can this Analysis really be Performed in less than 10-Minutes?

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### ABSTRACT

The analysis of the semivolatile organic compounds, like those found in USEPA method 8270D, places significant demands on the gas chromatographic column and analytical instrument. Concentrations of target and non-target components may range from low nanograms to milligrams, and the target compound list includes reactive acid and base compounds. Additionally, laboratories constantly try to improve detection limits and analysis time in an effort to differentiate their services while maintaining high sample throughput. In order to significantly improve upon the current state-of-the-art it is necessary to optimize the dimensions of the GC column, and also investigate MS systems that have the ability to accurately characterize the narrow peaks that are obtained in using more rapid GC separations.

This presentation will demonstrate a method improvement that addresses the above concerns. First a split injection is utilized to decrease the amount of material injected onto the column. Second, a narrow i.d. and short length GC column is used to achieve a faster run time, under more efficient separation conditions. Finally, TOFMS is used to achieve accurate peak characterization, over a wider range of calibration standard concentrations. Results of this method will be discussed and compared to the data obtained from a commercial laboratory for a series of sample extracts analyzed by conventional methodology.

# Managing Matrix Interferences in Pesticide Analysis with GC-TOFMS and GCxGC-TOFMS

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### ABSTRACT

Often the main challenges in the analysis of pesticides are to qualitatively and quantitatively determine a pesticide in the presence of sometimes-overwhelming matrix components. Numerous methods are employed to deal with matrix interferences, including off-line cleanup approaches such as solid phase extraction, and/or selected ion recording (SIR) when performing gas chromatography-mass spectrometry (GC-MS). SIR, while valuable for excluding matrix and increasing sensitivity, comes at the expense of a loss of information (versus full mass range acquisition), and may not be that helpful for avoiding interference for pesticides that have mainly low m/z ions in their mass spectra.

Time-of-flight mass spectrometry (TOFMS) offers benefits for pesticide analysis by GC that quadrupole MS systems do not have. Using TOFMS, a full mass spectrum is acquired, with low pg detection of many pesticides. Acquisition speed (up to hundreds of spectra/sec) and spectral continuity (due to the almost instantaneous mass analysis) permit automated peak find and spectral deconvolution algorithms to be included in the data processing software, enhancing pesticide location in complex samples.

A relatively new way to solve separation of matrix components and pesticides is to use comprehensive two-dimensional GC (GCxGC). GCxGC is a way to increase peak capacity by applying two independent separations to a sample in one analysis.

This paper will compare results for the analysis of pesticides in complex matrices using GC-TOFMS and GCxGC-TOFMS.