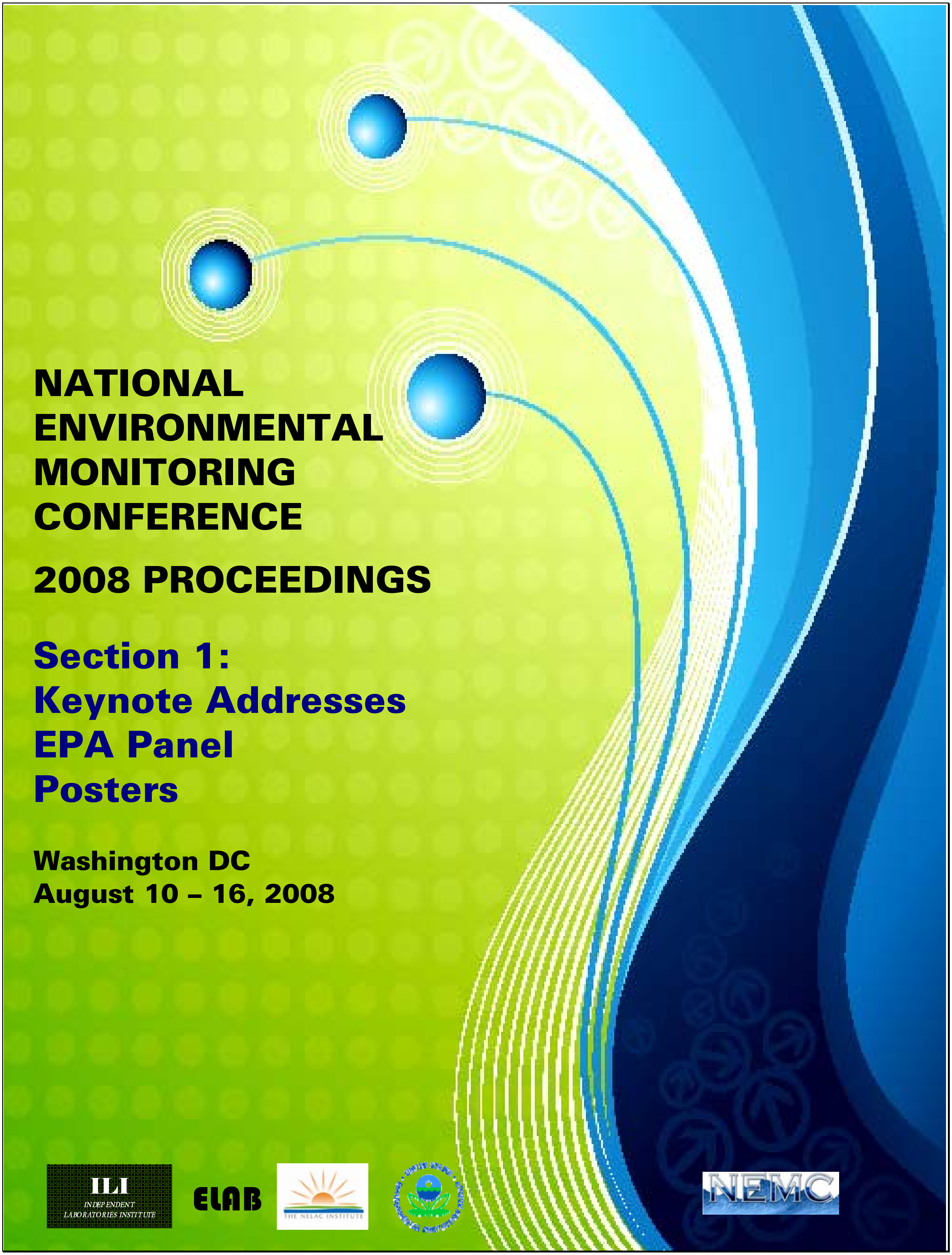

NATIONAL ENVIRONMENTAL MONITORING CONFERENCE

2008 PROCEEDINGS

Section 1:
Keynote Addresses
EPA Panel
Posters

Washington DC
August 10 – 16, 2008

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NEMC 2008
CONFERENCE HIGHLIGHTS

The Environmental Measurement Symposium, a combined meeting of the National Environmental Monitoring Conference (NEMC) and The NELAC Institute (TNI) was held August 10 – 16, 2008 in Washington DC, just blocks from the nation's capitol. The conference was co-sponsored by the US Environmental Protection Agency, the Independent Laboratories Institute, and The NELAC Institute.

A total of 469 people attended the 2008 Forum, which was a 9% increase in attendance over 2007. The meeting included:

- 19 technical breakout sessions with 100 presentations;
- a 2-day poster program with 23 posters;
- 4 keynote presentations;
- 3 EPA general sessions with 13 presentations;
- 13 TNI committee meetings;
- an assessment forum;
- a laboratory mentoring session;
- an accreditation body forum;
- a meeting of the Environmental Laboratory Advisory Board;
- 5 training workshops; and
- a 3-day exhibit program with 43 exhibitors and sponsors.

Highlights of the week included the following keynote speakers:

- Dr. Jorg Feldman from the University of Aberdeen who spoke on elemental speciation in environmental monitoring;
- Dr. Heidelore Fielder from the UN Environmental Program who spoke on global monitoring of persistent organic pollutants;
- Dr. J. Clarence Davies from Resources for the Future who spoke on EPA and nanotechnology; and
- TNI's own Bob Wyeth who spoke on moving forward on national accreditation.

NATIONAL ENVIRONMENTAL MONITORING CONFERENCE PROCEEDINGS 2008

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KEYNOTE ADDRESSES

Do We Need Elemental Speciation in Environmental Monitoring?

Jorg Feldmann
University of Aberdeen
Meston Walk
Aberdeen, UK AB243EU
+44-1224-272911
j.feldmann@abdn.ac.uk

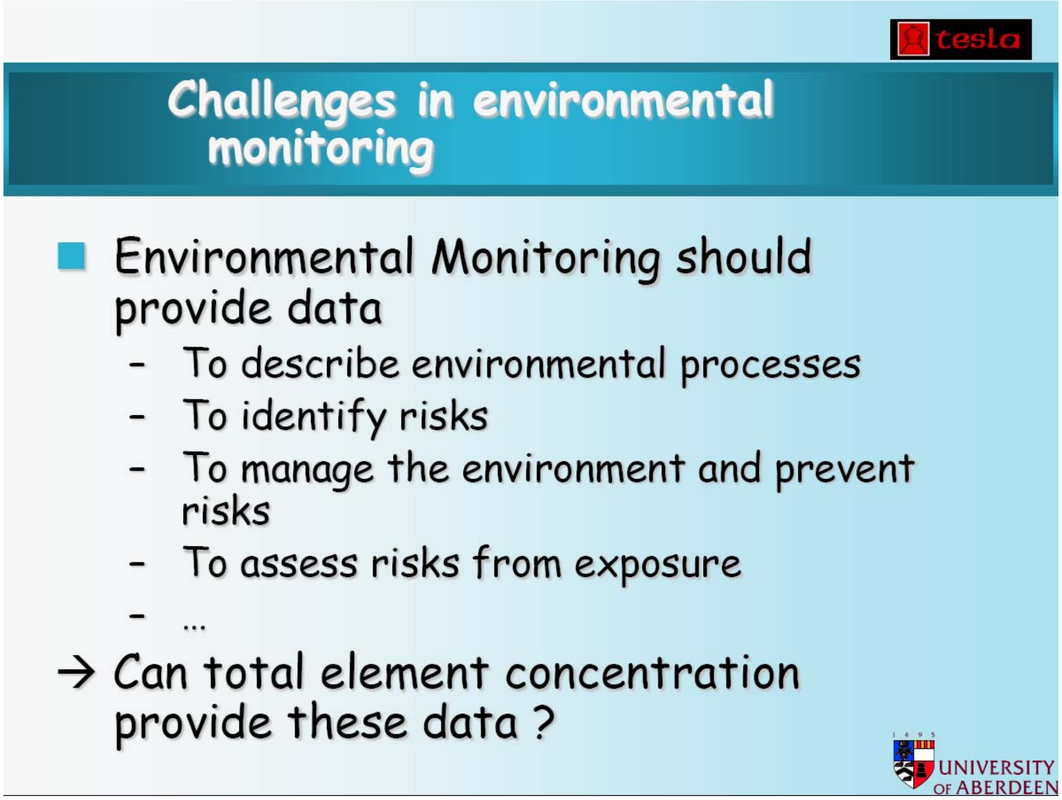
ABSTRACT

The lecture will give an overview about the elemental species of concern in environmental monitoring. It will highlight the implementation into legislation, and focus on analytical difficulties concerning accurate and precise determination of relevant element species. For some elements, relevant target species are well-identified while for other elements it is not clear which are the relevant species. The lecture will illustrate why elemental speciation not only is a key for risk assessment, but also is important to understand how elements are transported from contaminated soil into plants, and how they translocate. Reference will be given to the world-wide unique combination of elemental and molecular mass spectrometry simultaneously hyphenated to HPLC in Aberdeen (UK) and how it is used for identification and quantification of arsenic species at the soil/plant interface. As an example the data collected from food surveys in the UK, Bangladesh and USA on arsenic in rice products will be presented and discussed within the context of arsenic exposure.

Do We Need Elemental Speciation in Environmental Monitoring?

Jörg Feldmann

University of Aberdeen, Scotland



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Challenges in environmental monitoring

- Environmental Monitoring should provide data
  - To describe environmental processes
  - To identify risks
  - To manage the environment and prevent risks
  - To assess risks from exposure
  - …

→ Can total element concentration provide these data ?

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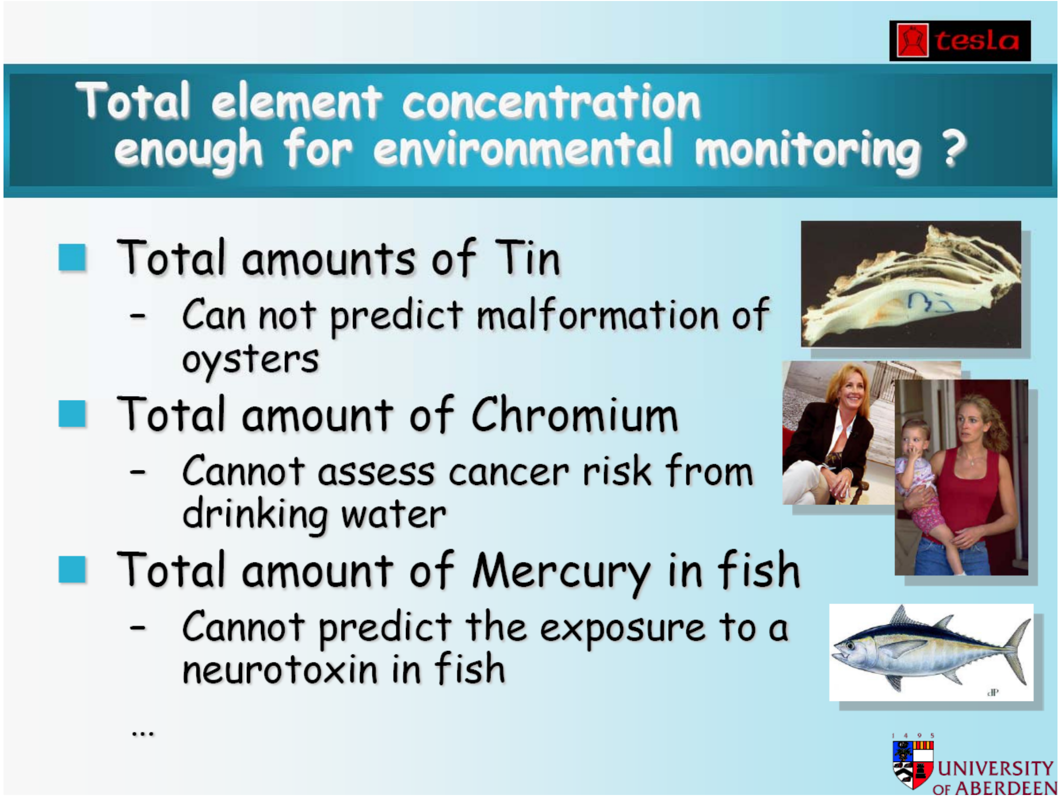

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Total element concentration enough for environmental monitoring ?

- Total amounts of Tin
  - Can not predict malformation of oysters
- Total amount of Chromium
  - Cannot assess cancer risk from drinking water
- Total amount of Mercury in fish
  - Cannot predict the exposure to a neurotoxin in fish

…

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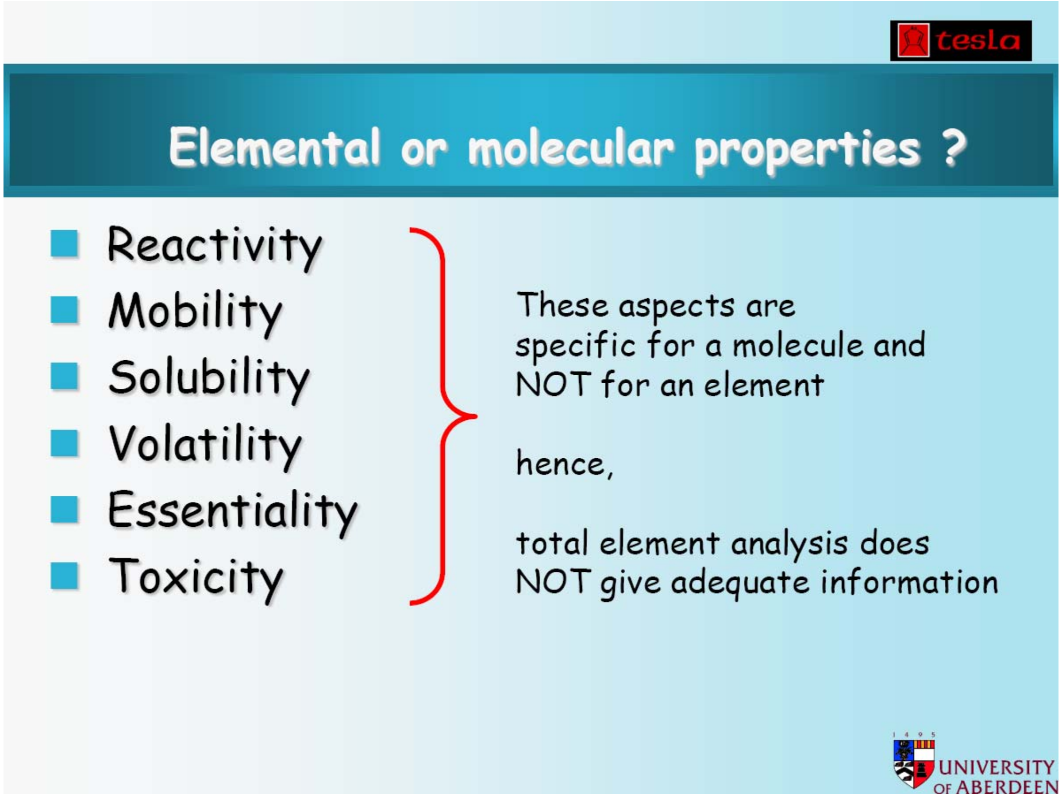

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Elemental or molecular properties ?

- Reactivity
- Mobility
- Solubility
- Volatility
- Essentiality
- Toxicity

These aspects are specific for a molecule and NOT for an element

hence,

total element analysis does NOT give adequate information

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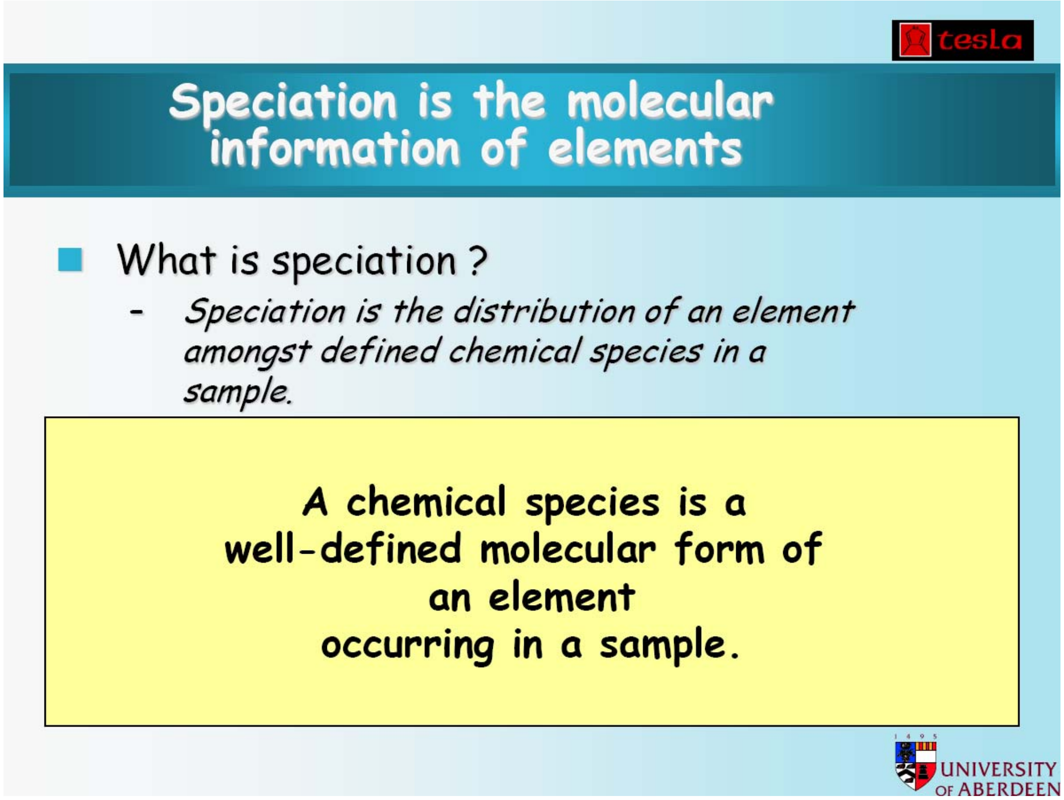
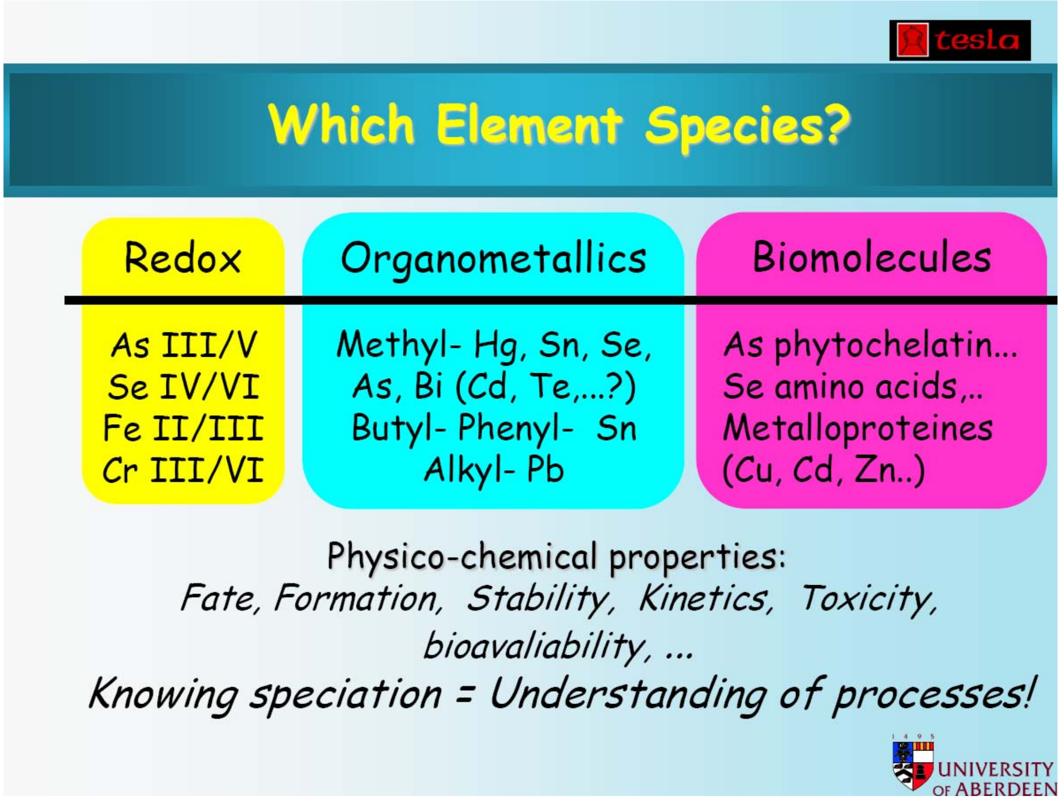


Speciation is the molecular information of elements

- What is speciation ?
  - *Speciation is the distribution of an element amongst defined chemical species in a sample.*

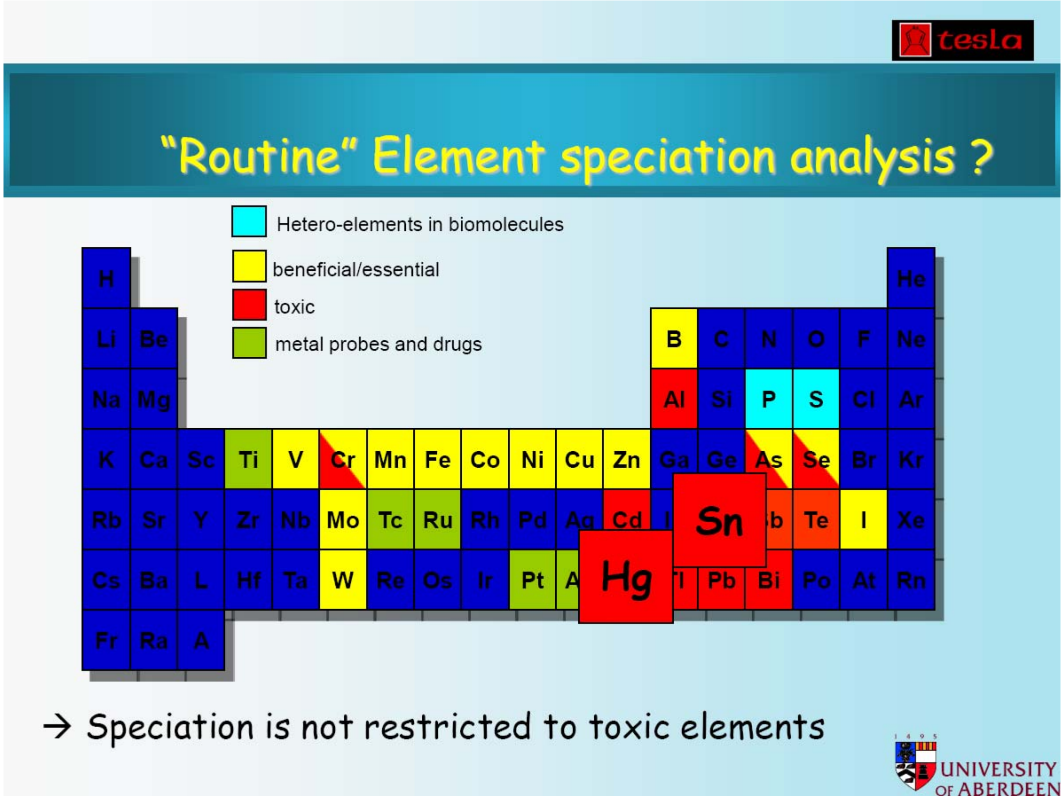
A chemical species is a
well-defined molecular form of
an element
occurring in a sample.



Which Element Species?

Redox	Organometallics	Biomolecules
As III/V Se IV/VI Fe II/III Cr III/VI	Methyl- Hg, Sn, Se, As, Bi (Cd, Te,...?) Butyl- Phenyl- Sn Alkyl- Pb	As phytochelatin... Se amino acids,.. Metalloproteines (Cu, Cd, Zn..)

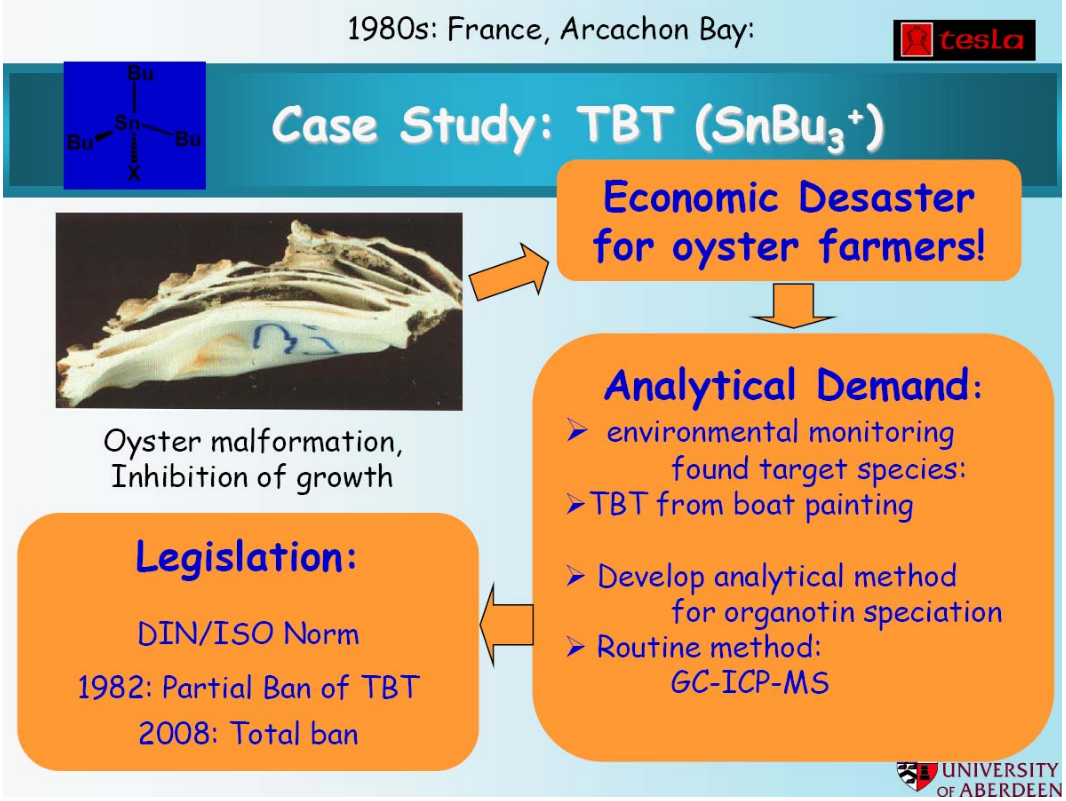
Physico-chemical properties:
Fate, Formation, Stability, Kinetics, Toxicity, bioavaliability, ...
Knowing speciation = Understanding of processes!



"Routine" Element speciation analysis ?

- 🟦 Hetero-elements in biomolecules
- 🟨 beneficial/essential
- 🟥 toxic
- 🟩 metal probes and drugs

→ Speciation is not restricted to toxic elements



1980s: France, Arcachon Bay:

Case Study: TBT ($SnBu_3^+$)

Oyster malformation,
Inhibition of growth

Economic Desaster for oyster farmers!

Analytical Demand:

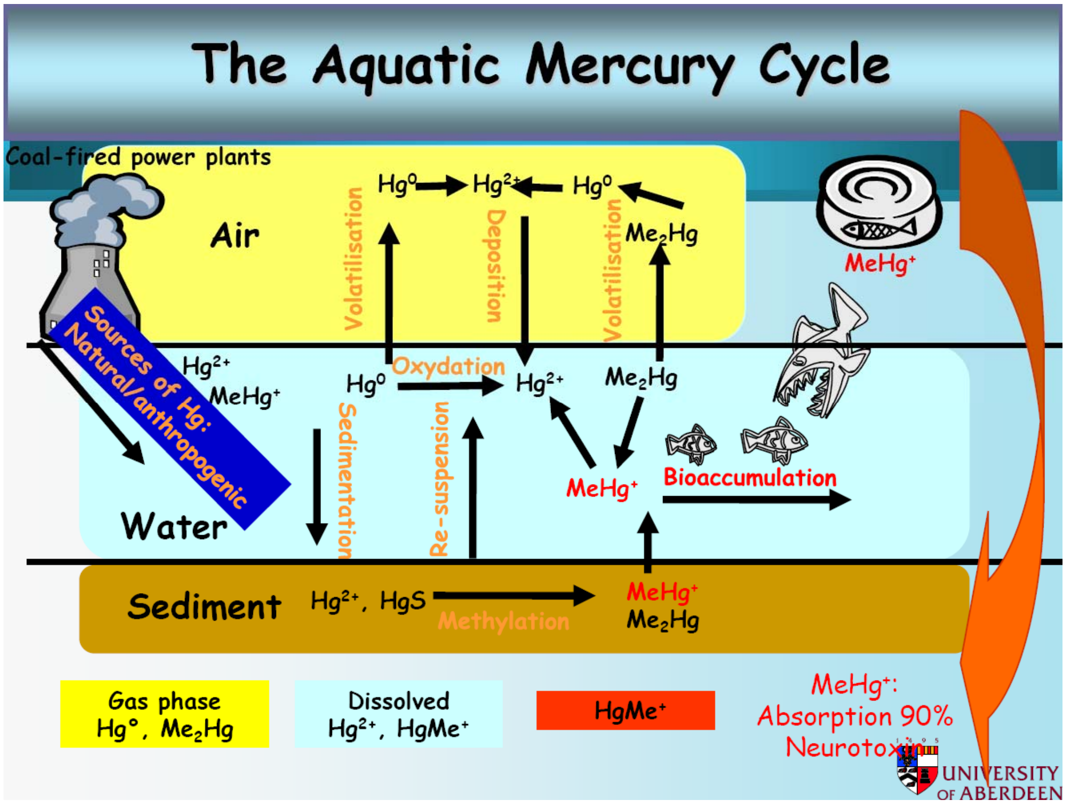
- environmental monitoring
  found target species:
- TBT from boat painting
- Develop analytical method
  for organotin speciation
- Routine method:
  GC-ICP-MS

Legislation:

DIN/ISO Norm

1982: Partial Ban of TBT

2008: Total ban



The Aquatic Mercury Cycle

Gas phase $Hg°$, Me_2Hg	Dissolved Hg^{2+}, $HgMe^+$	$HgMe^+$

$MeHg^+$:
Absorption 90%
Neurotoxin

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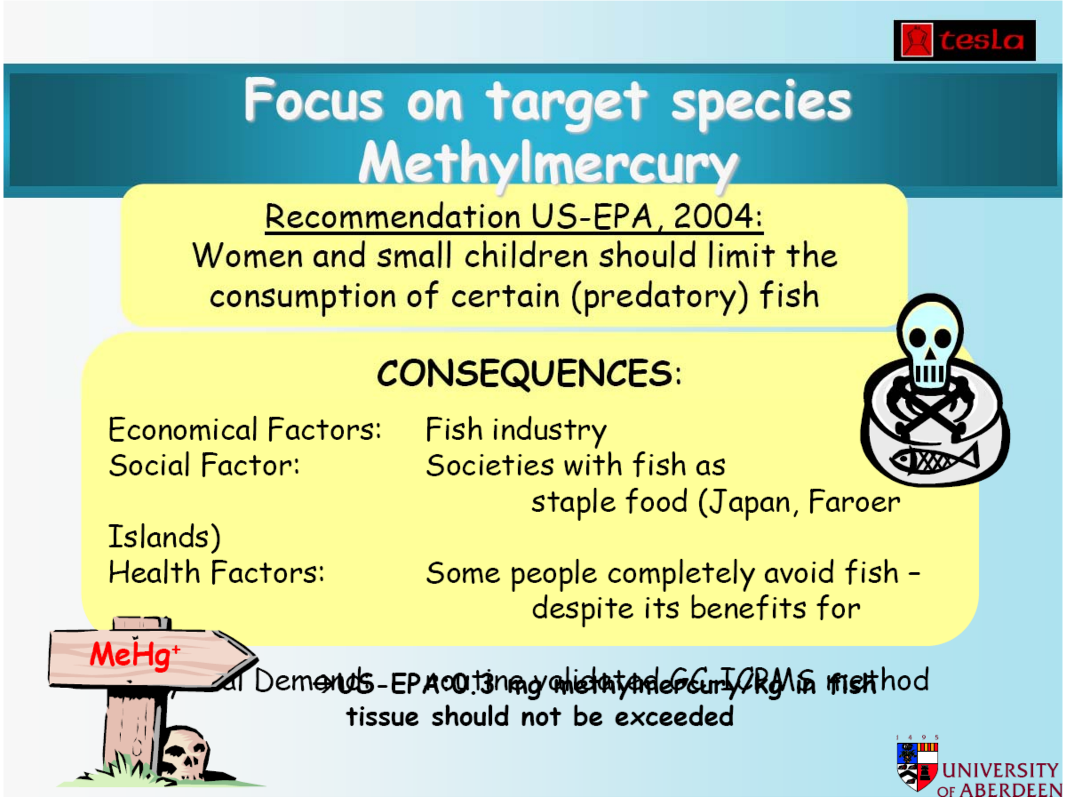

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Focus on target species Methylmercury

Recommendation US-EPA, 2004:
Women and small children should limit the consumption of certain (predatory) fish

CONSEQUENCES:

Economical Factors: Fish industry
Social Factor: Societies with fish as staple food (Japan, Faroer Islands)
Health Factors: Some people completely avoid fish - despite its benefits for

al Demands → routine validated GC-ICPMS method

US-EPA: 0.3 mg methylmercury/kg in fish tissue should not be exceeded

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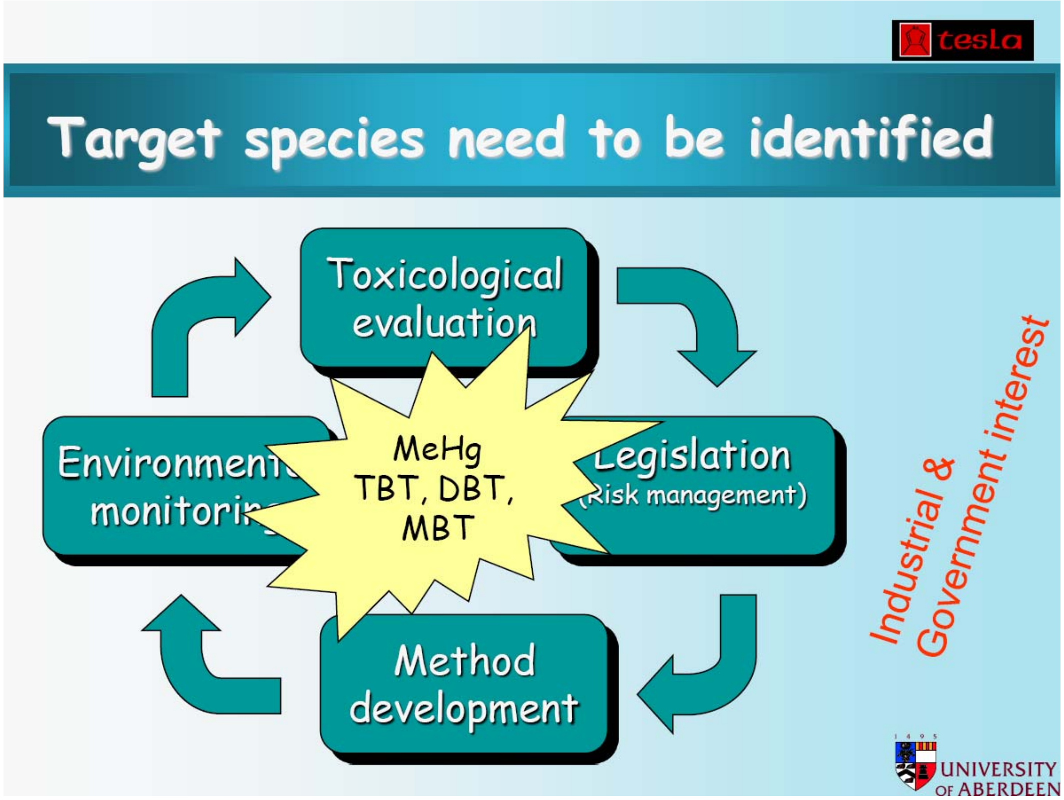


Target species need to be identified

Toxicological evaluation

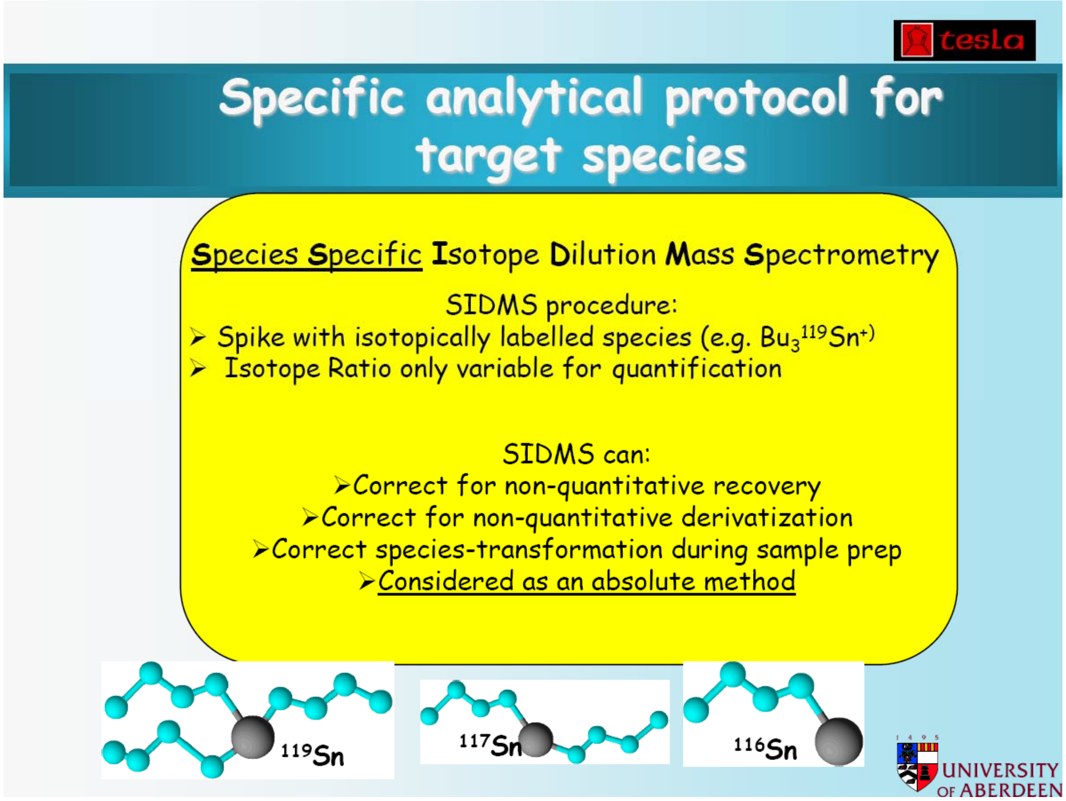
Legislation (Risk management)

Method development

Environmen monitorin

MeHg
TBT, DBT,
MBT

Industrial & Government interest



Specific analytical protocol for target species

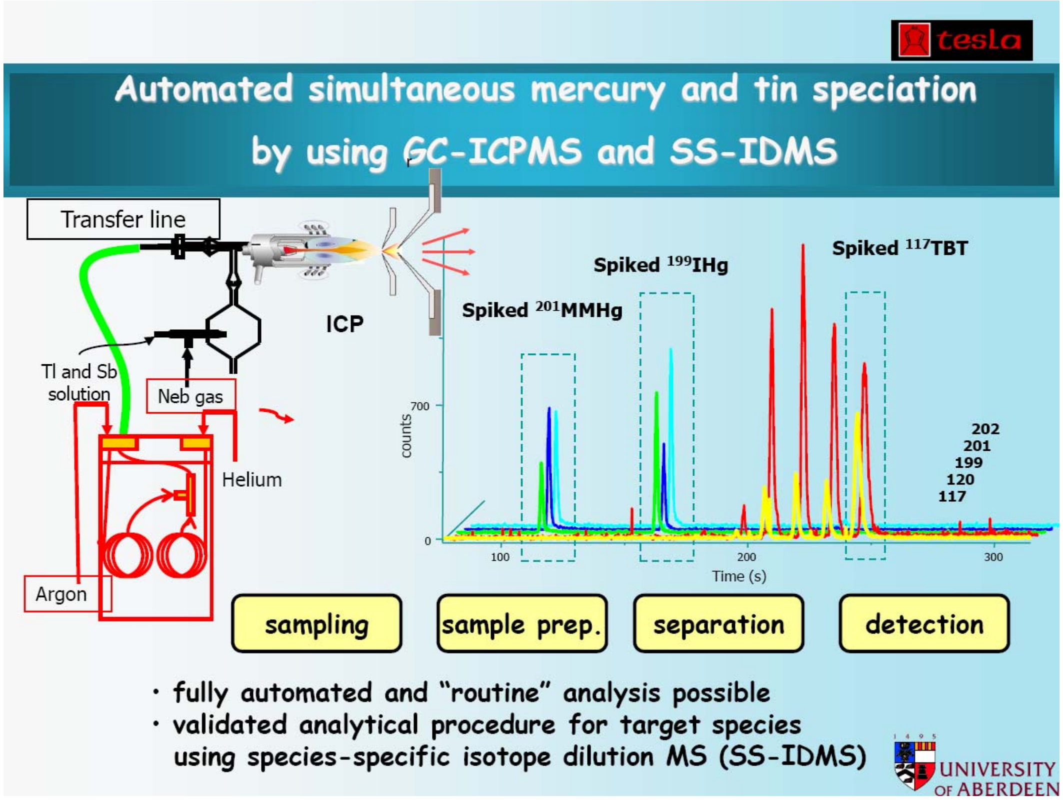
<u>**S**pecies **S**pecific</u> **I**sotope **D**ilution **M**ass **S**pectrometry

SIDMS procedure:

- Spike with isotopically labelled species (e.g. Bu$_3$^{119}Sn^{+})
- Isotope Ratio only variable for quantification

SIDMS can:

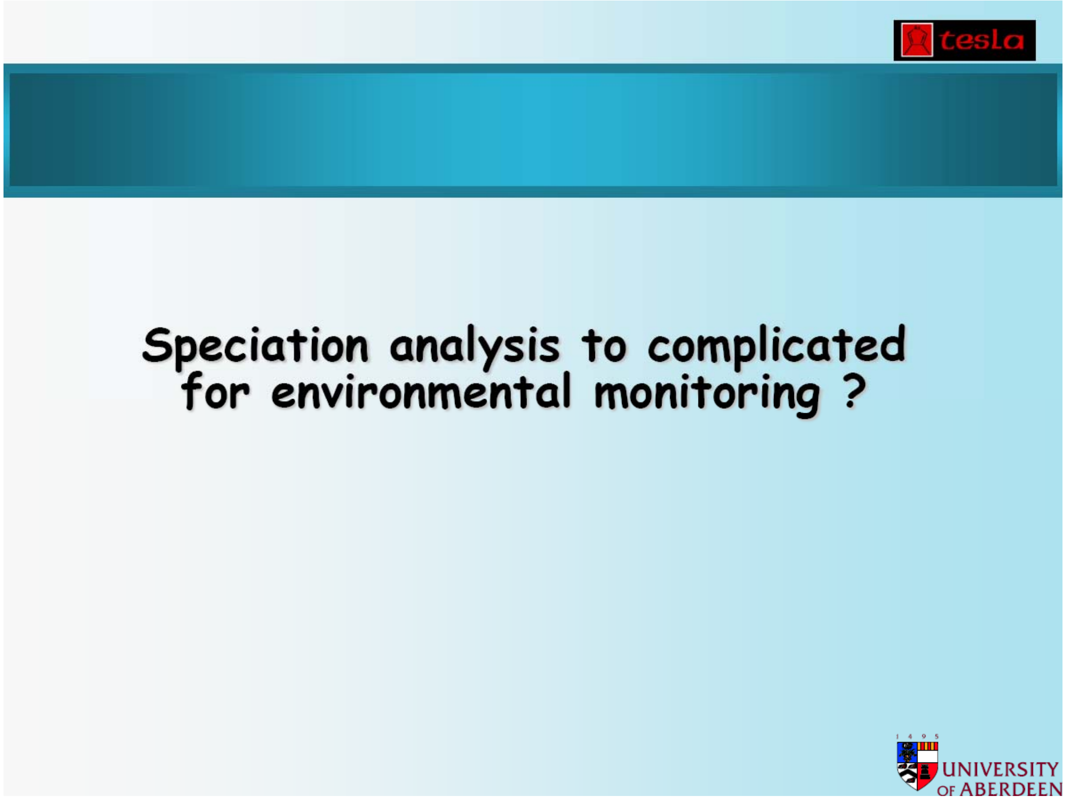
- Correct for non-quantitative recovery
- Correct for non-quantitative derivatization
- Correct species-transformation during sample prep
- <u>Considered as an absolute method</u>



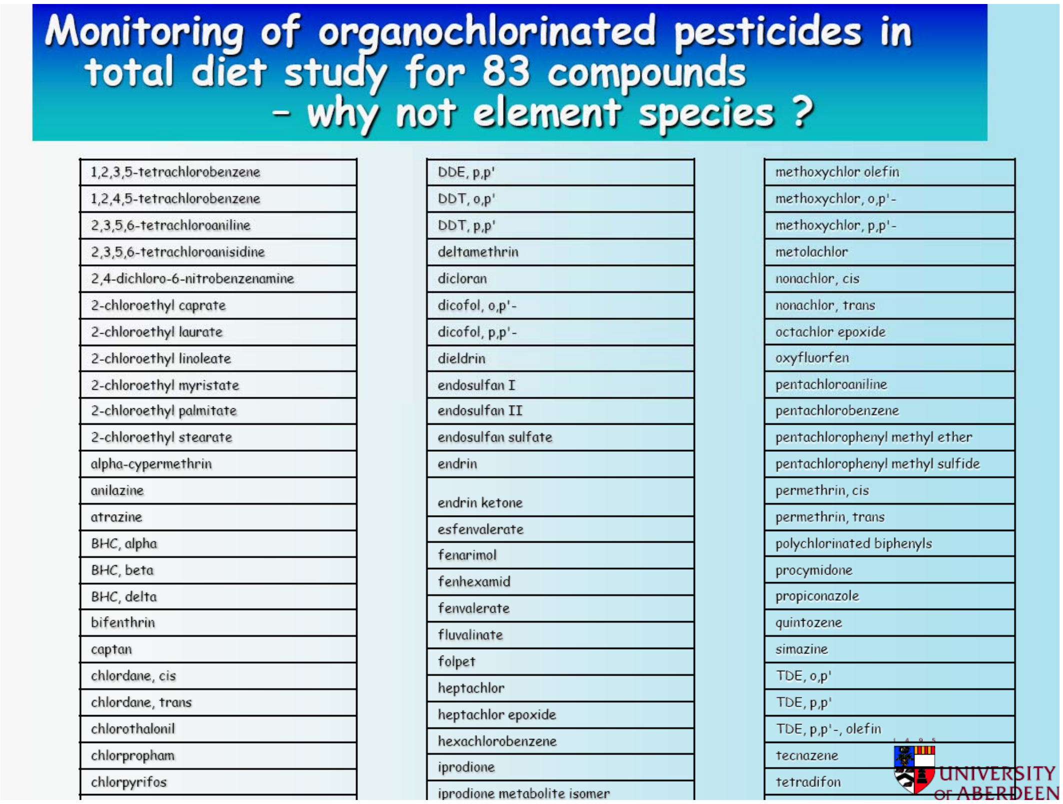
Automated simultaneous mercury and tin speciation by using GC-ICPMS and SS-IDMS

sampling | sample prep. | separation | detection

- fully automated and "routine" analysis possible
- validated analytical procedure for target species using species-specific isotope dilution MS (SS-IDMS)



Speciation analysis to complicated for environmental monitoring ?



Monitoring of organochlorinated pesticides in total diet study for 83 compounds – why not element species ?

- 1,2,3,5-tetrachlorobenzene
- 1,2,4,5-tetrachlorobenzene
- 2,3,5,6-tetrachloroaniline
- 2,3,5,6-tetrachloroanisidine
- 2,4-dichloro-6-nitrobenzenamine
- 2-chloroethyl caprate
- 2-chloroethyl laurate
- 2-chloroethyl linoleate
- 2-chloroethyl myristate
- 2-chloroethyl palmitate
- 2-chloroethyl stearate
- alpha-cypermethrin
- anilazine
- atrazine
- BHC, alpha
- BHC, beta
- BHC, delta
- bifenthrin
- captan
- chlordane, cis
- chlordane, trans
- chlorothalonil
- chlorpropham
- chlorpyrifos

- DDE, p,p'
- DDT, o,p'
- DDT, p,p'
- deltamethrin
- dicloran
- dicofol, o,p'-
- dicofol, p,p'-
- dieldrin
- endosulfan I
- endosulfan II
- endosulfan sulfate
- endrin
- endrin ketone
- esfenvalerate
- fenarimol
- fenhexamid
- fenvalerate
- fluvalinate
- folpet
- heptachlor
- heptachlor epoxide
- hexachlorobenzene
- iprodione
- iprodione metabolite isomer

- methoxychlor olefin
- methoxychlor, o,p'-
- methoxychlor, p,p'-
- metolachlor
- nonachlor, cis
- nonachlor, trans
- octachlor epoxide
- oxyfluorfen
- pentachloroaniline
- pentachlorobenzene
- pentachlorophenyl methyl ether
- pentachlorophenyl methyl sulfide
- permethrin, cis
- permethrin, trans
- polychlorinated biphenyls
- procymidone
- propiconazole
- quintozene
- simazine
- TDE, o,p'
- TDE, p,p'
- TDE, p,p'-, olefin
- tecnazene
- tetradifon

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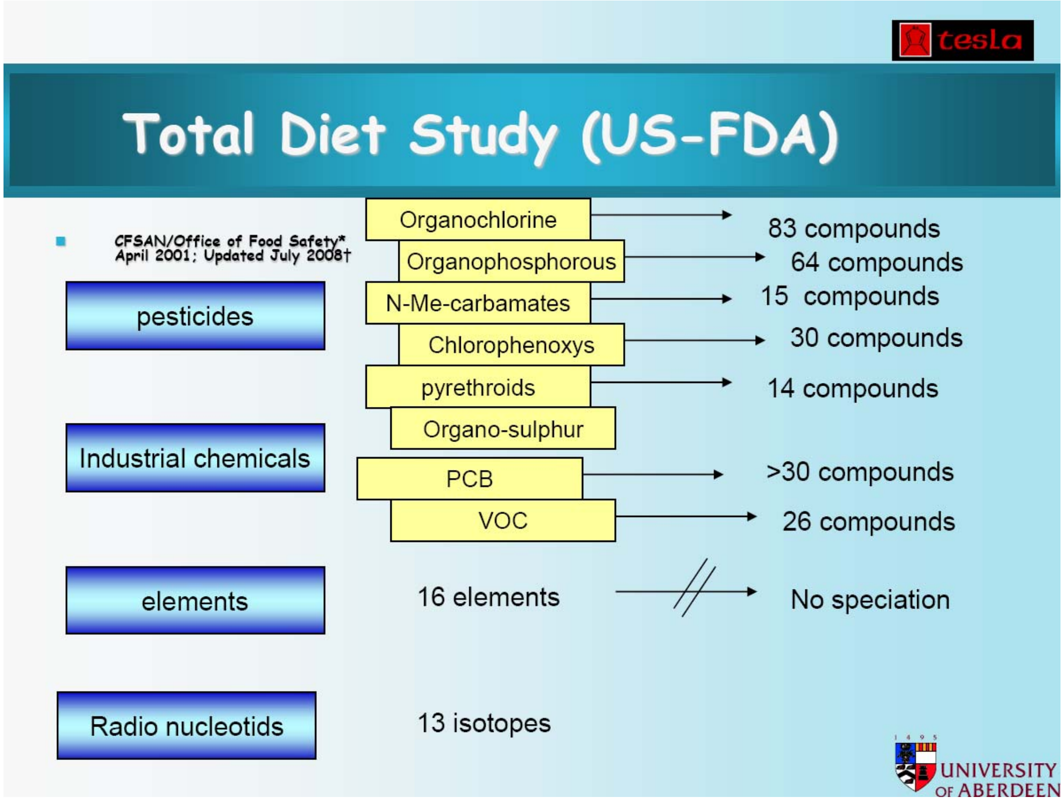

tesla

Total Diet Study (US-FDA)

- CFSAN/Office of Food Safety*
  April 2001; Updated July 2008†

pesticides

Organochlorine → 83 compounds

Organophosphorous → 64 compounds

N-Me-carbamates → 15 compounds

Chlorophenoxys → 30 compounds

pyrethroids → 14 compounds

Organo-sulphur

Industrial chemicals

PCB → >30 compounds

VOC → 26 compounds

elements

16 elements → No speciation

Radio nucleotids

13 isotopes

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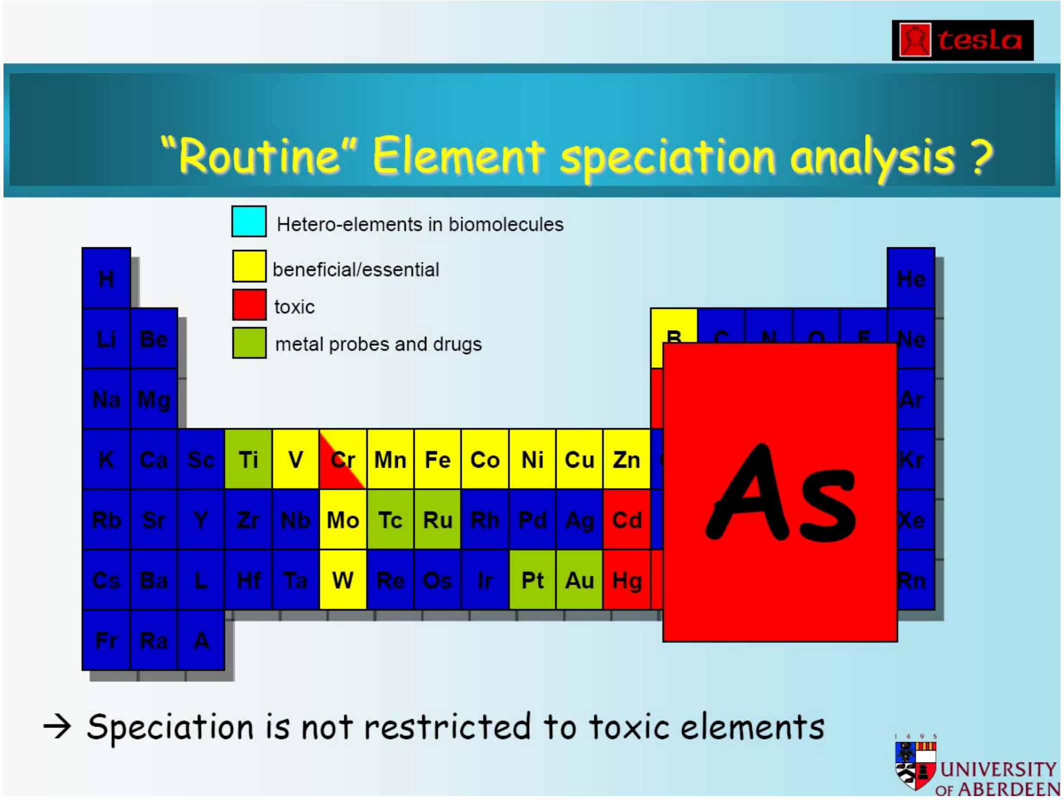


"Routine" Element speciation analysis ?

🟦 Hetero-elements in biomolecules
🟨 beneficial/essential
🟥 toxic
🟩 metal probes and drugs

→ Speciation is not restricted to toxic elements

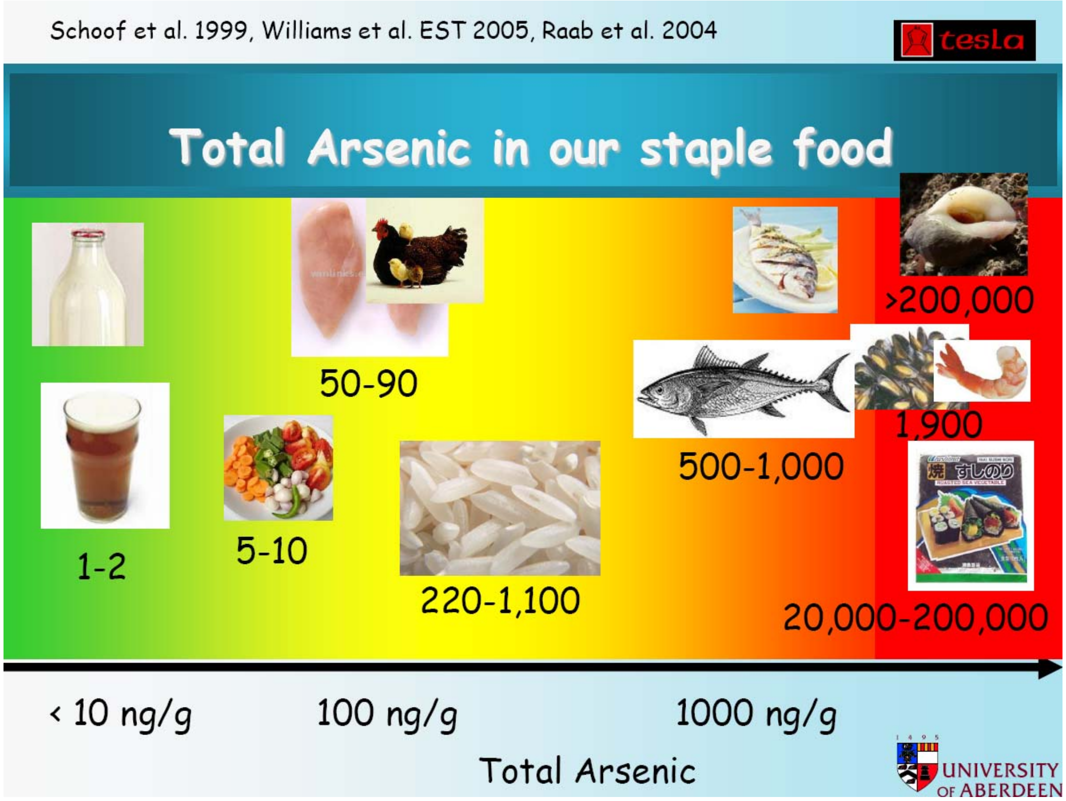
More than 60 Million people are exposed to elevated levels of Arsenic in their drinking water

Fig. 1. Skin lesions due to arsenic poisoning

Fig. 2. Children near a tube-well disconnected due to contamination of water with arsenic

US-EPA, WHO level of As in water: < 10 µg/L
Daily intake should be less than 0.01 mg !!!

- Arsenic
  - Problem 1: total arsenic in food does not reflect risk
  - Problem 2: total arsenic in soil does not reflect the arsenic in crops
  - Problem 3: total arsenic in urine does not reflect the exposure to toxic arsenic



Schoof et al. 1999, Williams et al. EST 2005, Raab et al. 2004

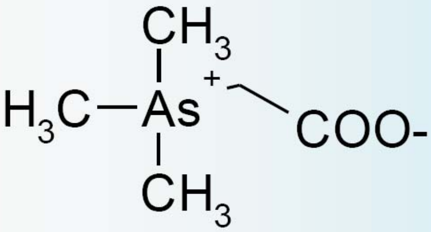
Total Arsenic in our staple food

"Fish arsenic"

UK guideline since 1959: Regulation of arsenic in food:

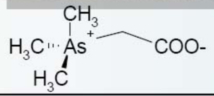
… *any food other than fish, edible seaweed* *may not contain arsenic in a proportion exceeding 1 mg/kg.*

→ Arsenic in fish has been considered as harmless !!!



Main species: Arsenobetaine:

Arsenic toxicity – species dependant



arsenite
arsenate

Arsenobetaine

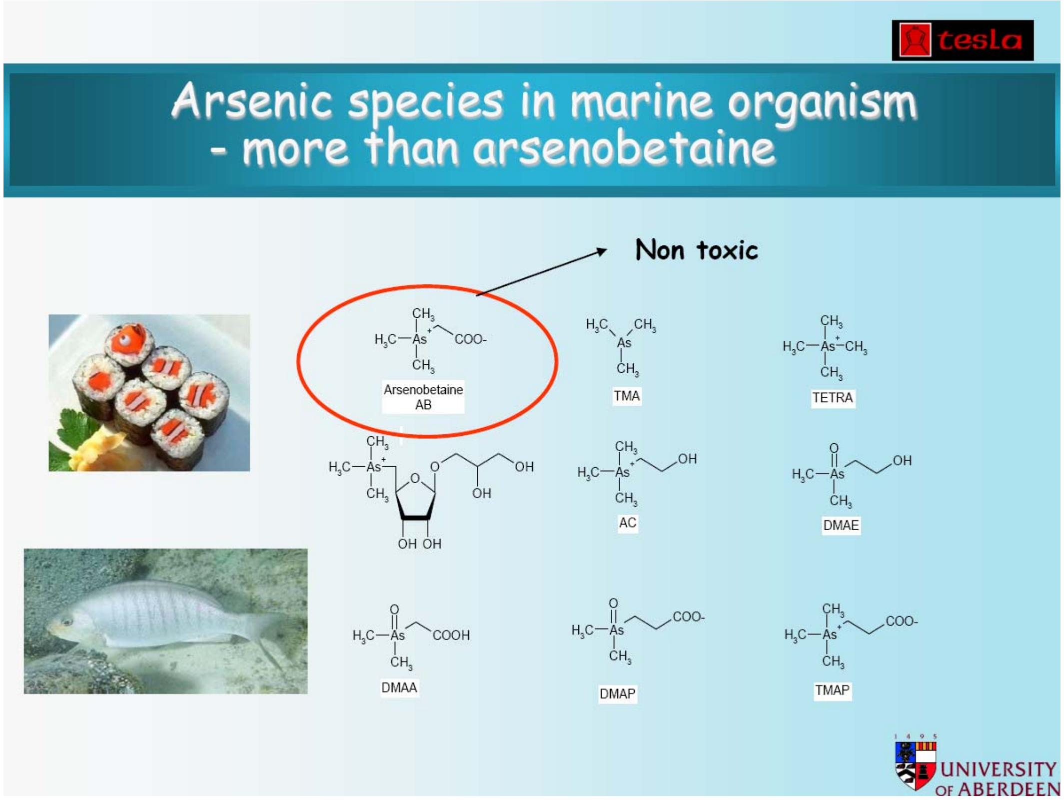
- Fish, shellfish
- No acute toxi
  10,000 mg/kg
- No chronic to
- No accumulati
  and skin
- Not metaboliz
- Excreted unch

/ arsenate

and in vegetables

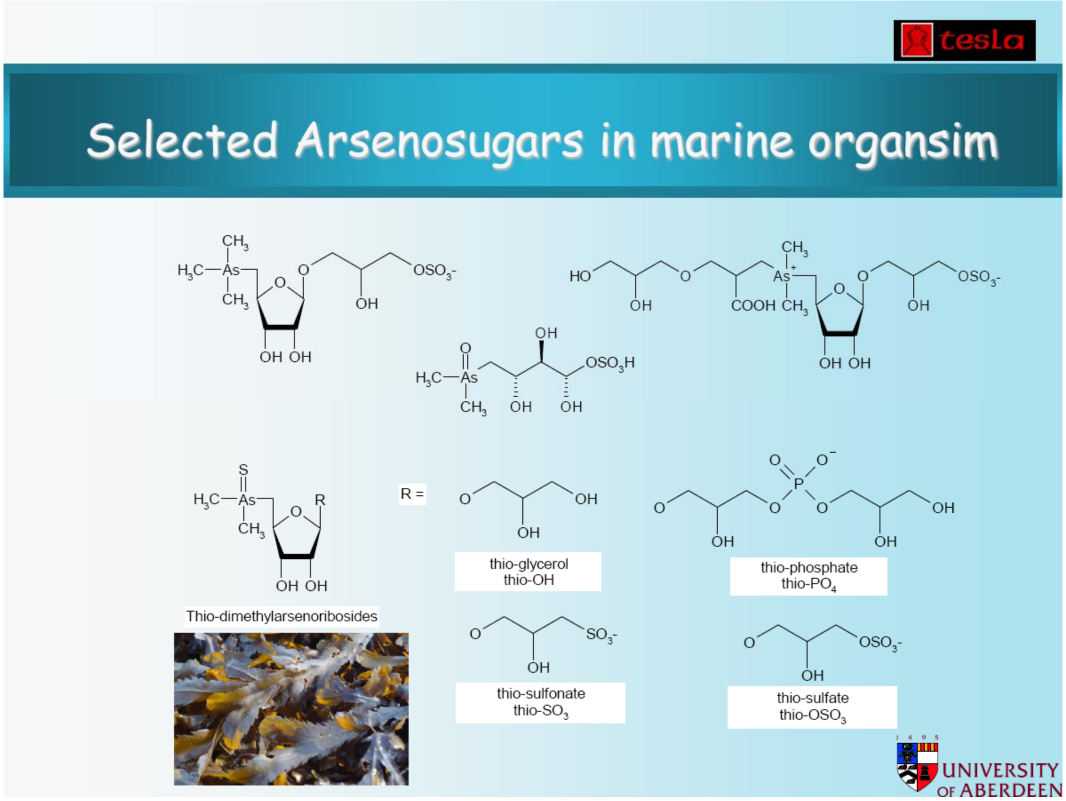
e toxicity (LD_{50}< 50
)

nic toxic effect
te in hair, nail and skin
sed by humans to DMA,
hylated arsenics)
via urine



Arsenic species in marine organism - more than arsenobetaine

Non toxic



Selected Arsenosugars in marine organsim

Thio-dimethylarsenoribosides

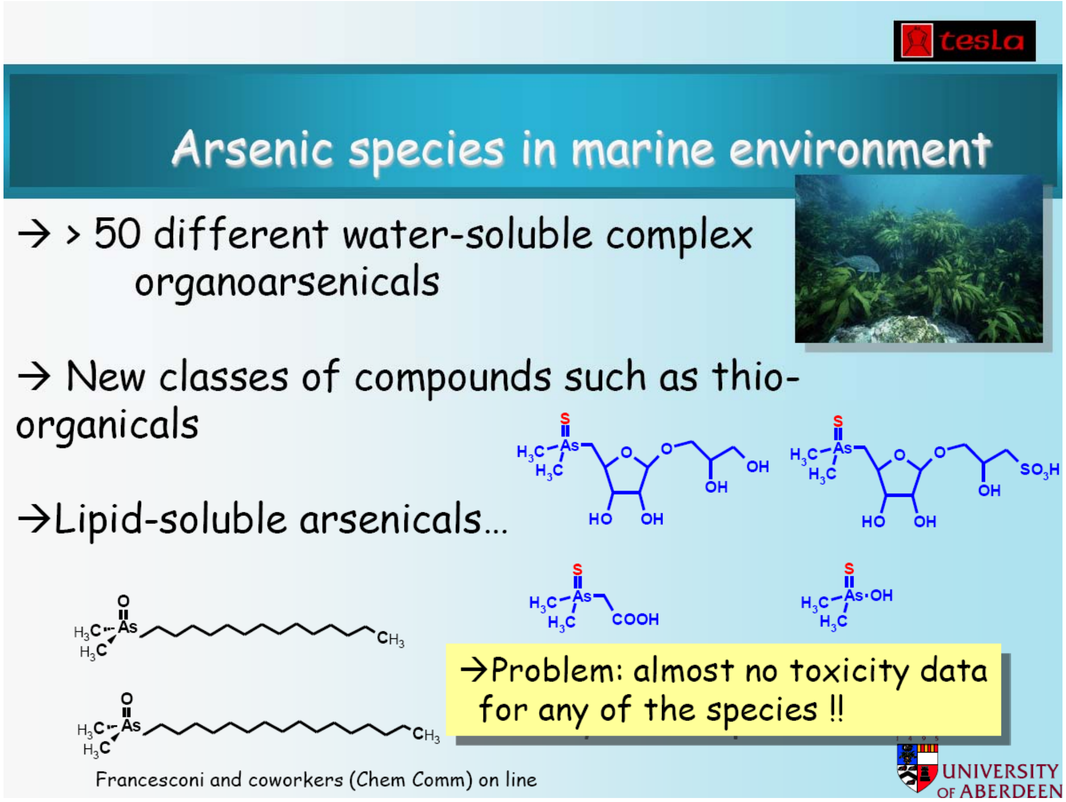
R =

thio-glycerol
thio-OH

thio-phosphate
thio-PO_4

thio-sulfonate
thio-SO_3

thio-sulfate
thio-OSO_3



Arsenic species in marine environment

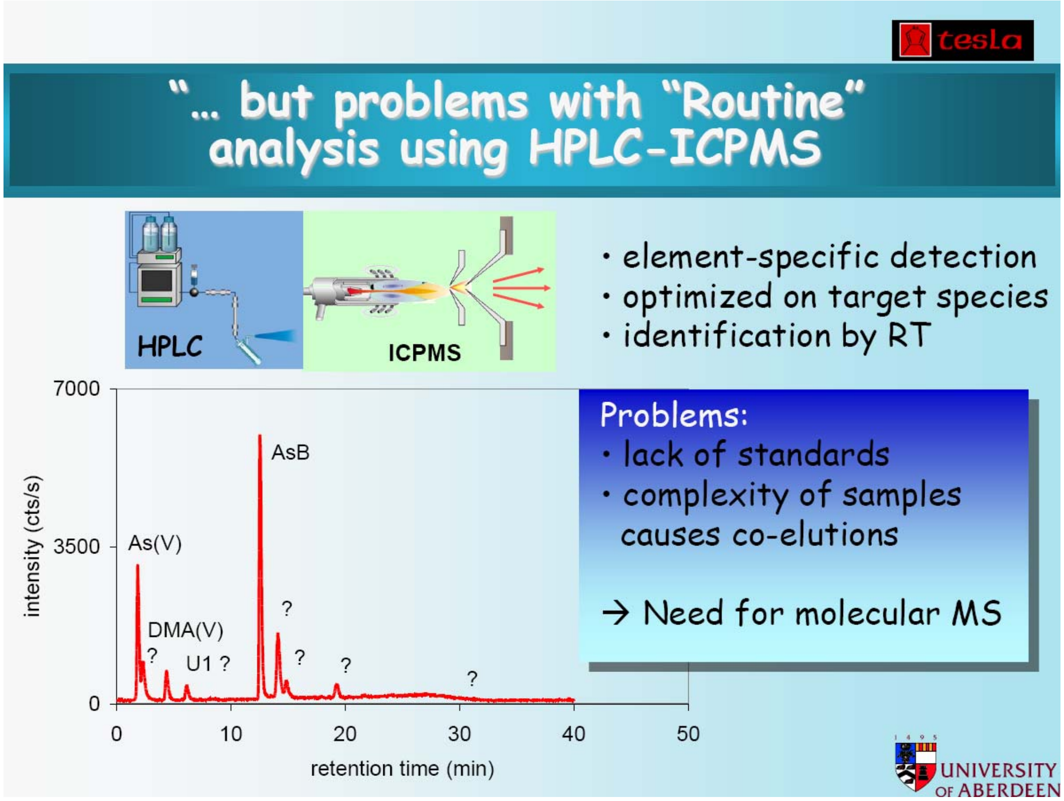
→ > 50 different water-soluble complex organoarsenicals

→ New classes of compounds such as thio-organicals

→Lipid-soluble arsenicals…

→Problem: almost no toxicity data for any of the species !!

Francesconi and coworkers (Chem Comm) on line

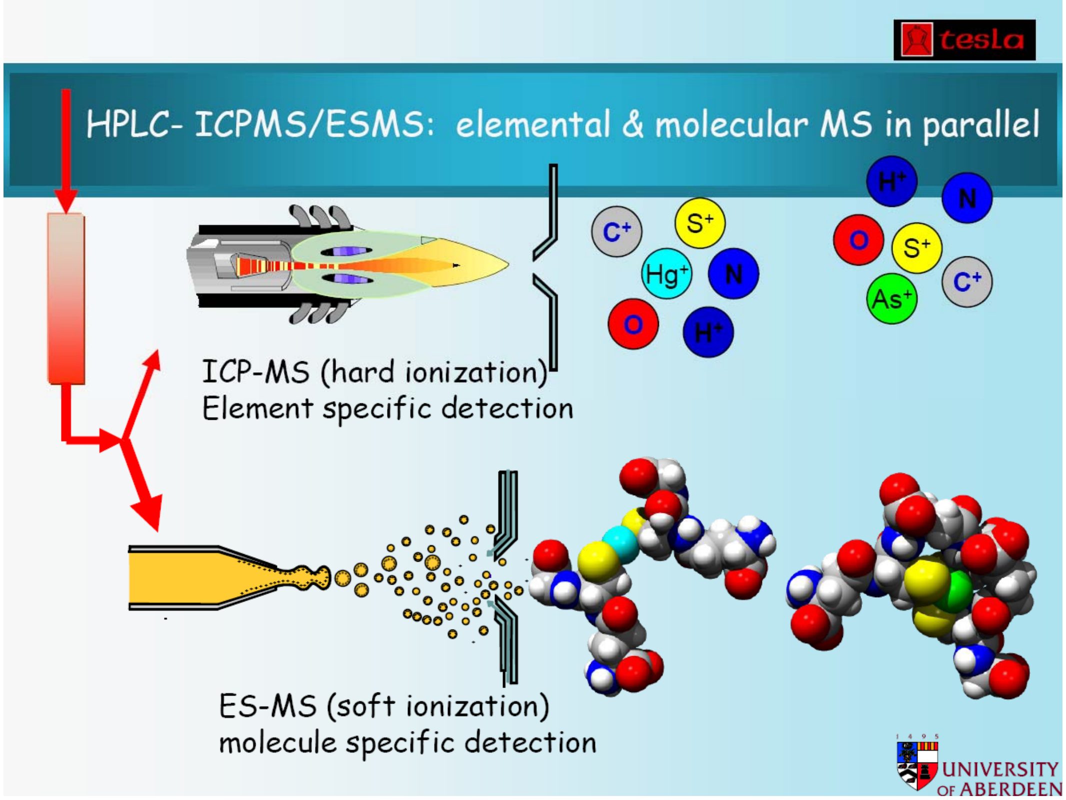


"… but problems with "Routine" analysis using HPLC-ICPMS

- element-specific detection
- optimized on target species
- identification by RT

Problems:
- lack of standards
- complexity of samples causes co-elutions

→ Need for molecular MS



HPLC- ICPMS/ESMS: elemental & molecular MS in parallel

ICP-MS (hard ionization)
Element specific detection

ES-MS (soft ionization)
molecule specific detection

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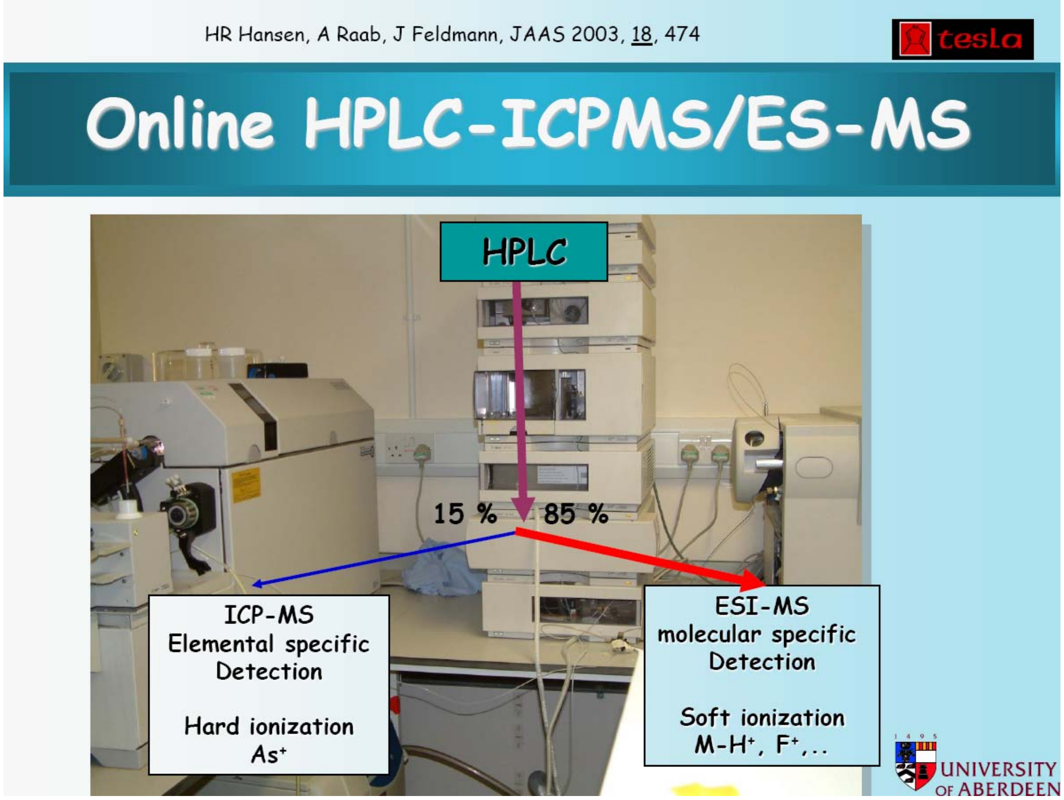

HR Hansen, A Raab, J Feldmann, JAAS 2003, 18, 474

Online HPLC-ICPMS/ES-MS

HPLC

15 %

85 %

ICP-MS
Elemental specific
Detection

Hard ionization
As⁺

ESI-MS
molecular specific
Detection

Soft ionization
M-H⁺, F⁺,..

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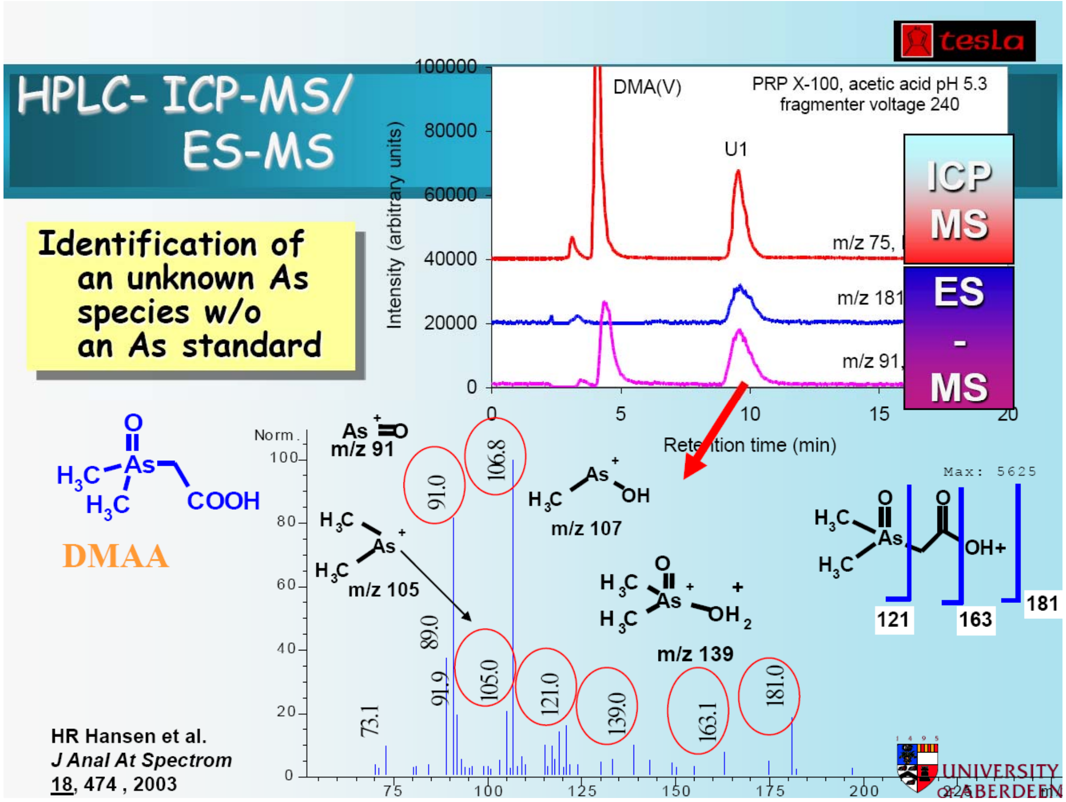


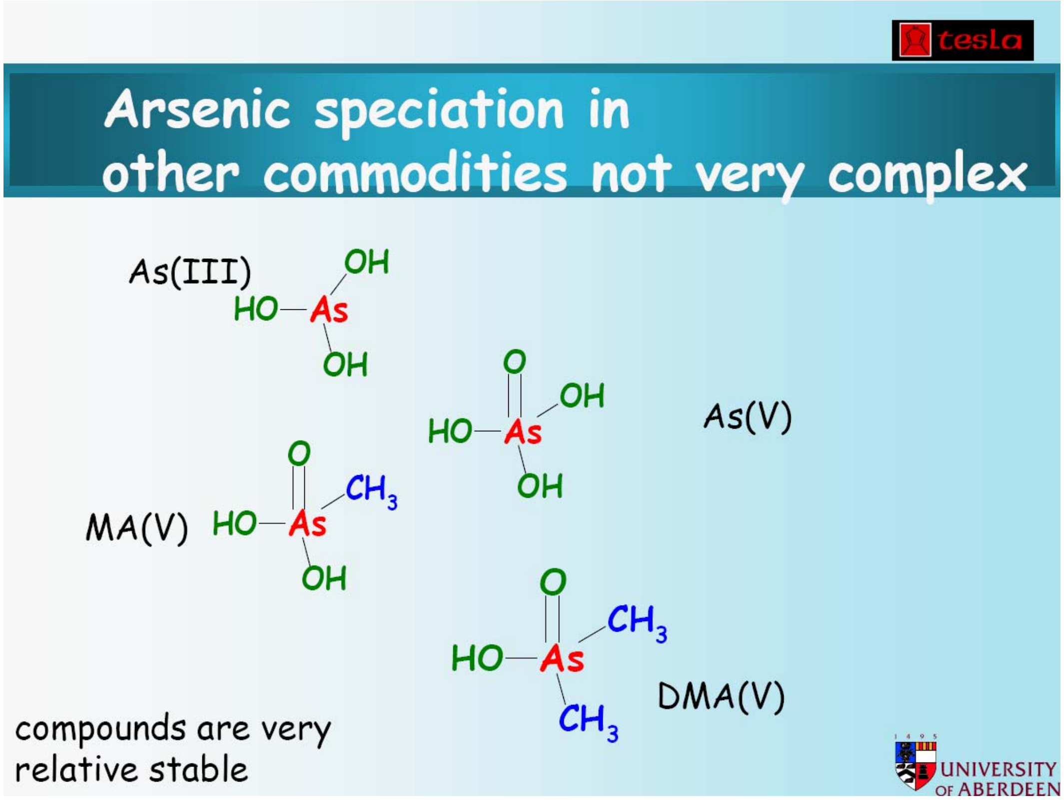
HPLC- ICP-MS/ ES-MS

Identification of an unknown As species w/o an As standard

HR Hansen et al.
J Anal At Spectrom
18, 474 , 2003

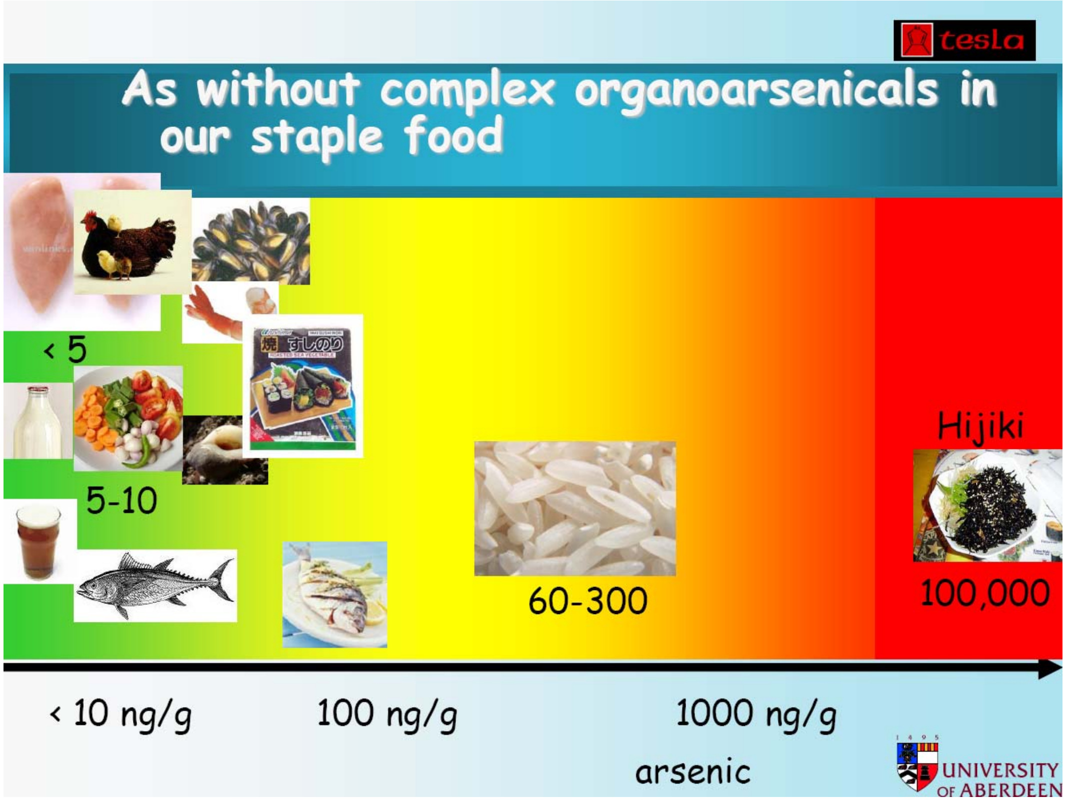
Summary: As in marine samples

- Arsenic speciation in marine samples very complex
  - Analysis needs sophisticated methods
  - Target species not yet identified
- As speciation of seafood (incl. metabolic studies) is difficult to be implemented into large monitoring programs.

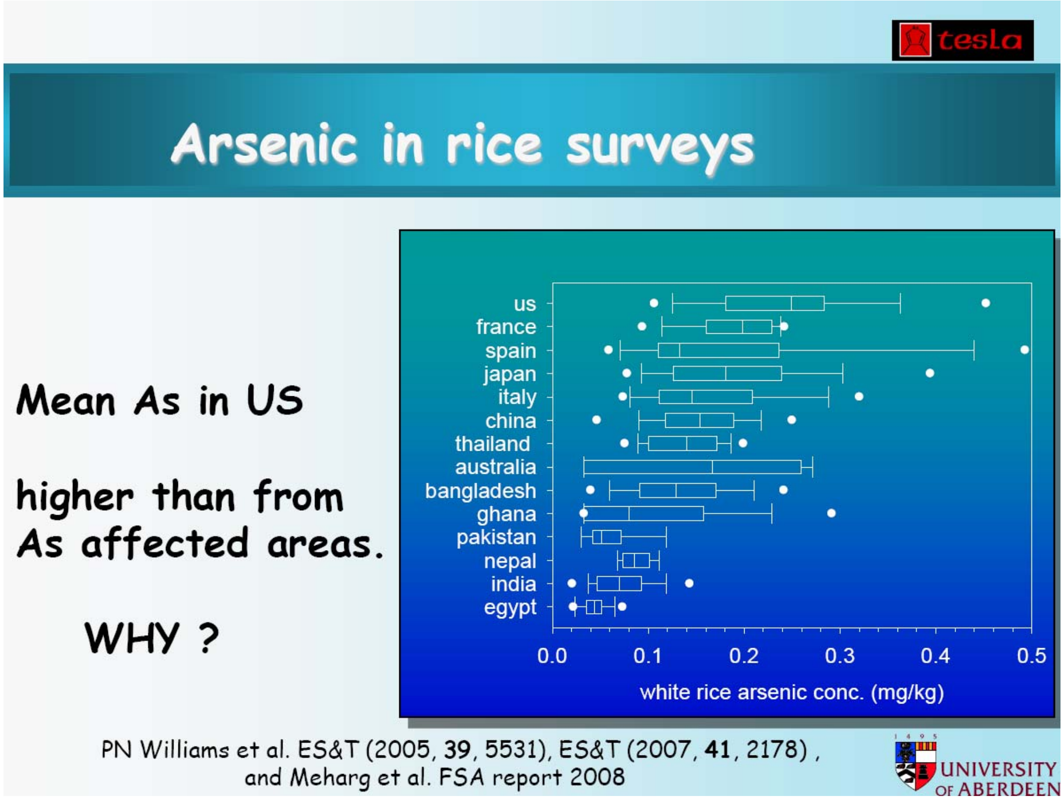


Arsenic speciation in other commodities not very complex

compounds are very relative stable



As without complex organoarsenicals in our staple food



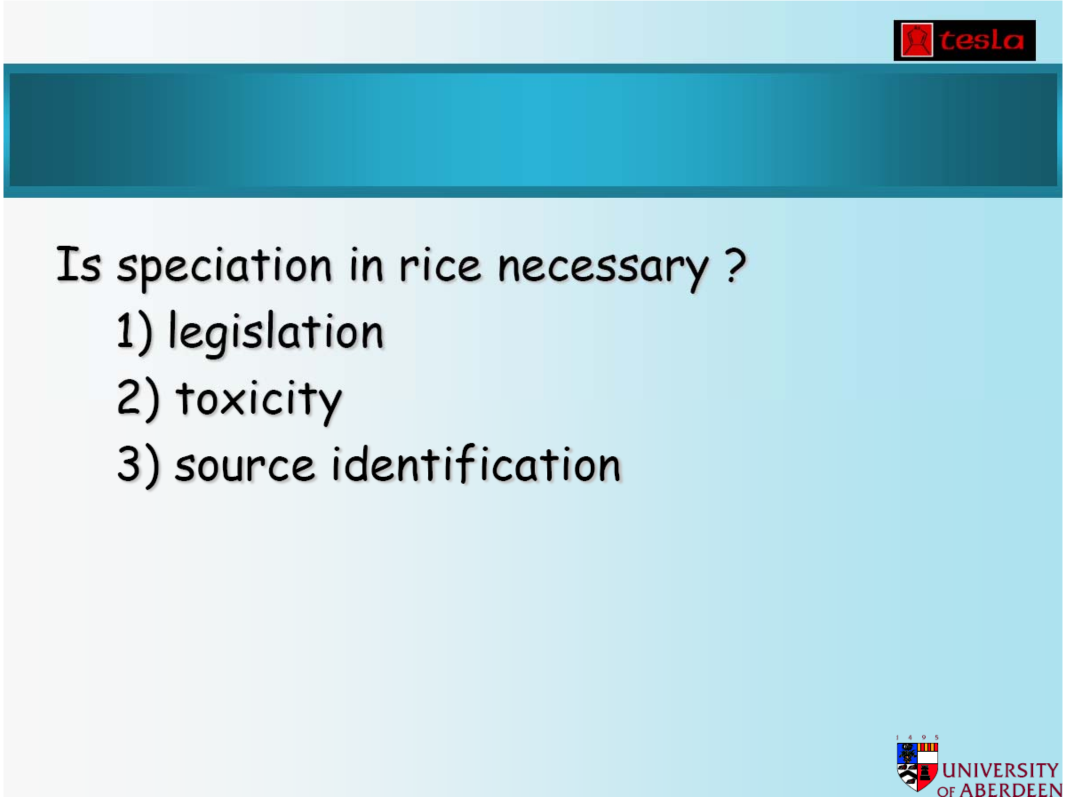
Arsenic in rice surveys

Mean As in US

higher than from As affected areas.

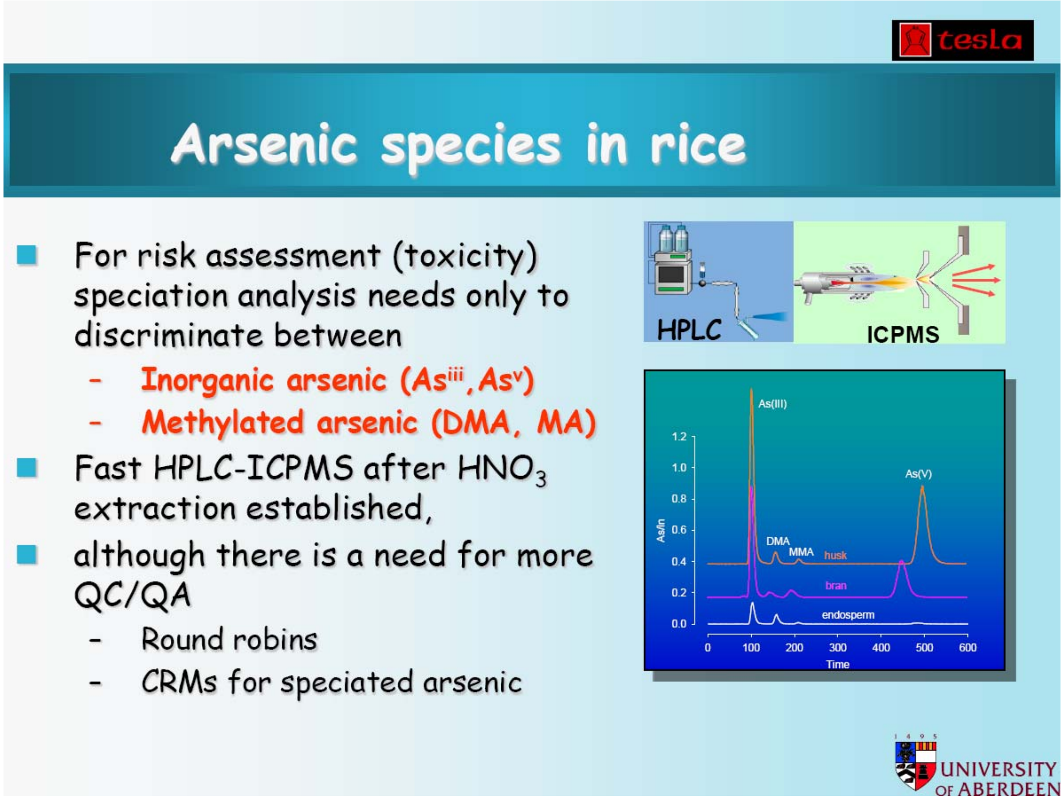
WHY ?

PN Williams et al. ES&T (2005, **39**, 5531), ES&T (2007, **41**, 2178) , and Meharg et al. FSA report 2008



Is speciation in rice necessary ?

1) legislation
2) toxicity
3) source identification



tesla

Arsenic species in rice

- For risk assessment (toxicity) speciation analysis needs only to discriminate between
  - **Inorganic arsenic (As^{iii}, As^{v})**
  - **Methylated arsenic (DMA, MA)**
- Fast HPLC-ICPMS after HNO_3 extraction established,
- although there is a need for more QC/QA
  - Round robins
  - CRMs for speciated arsenic

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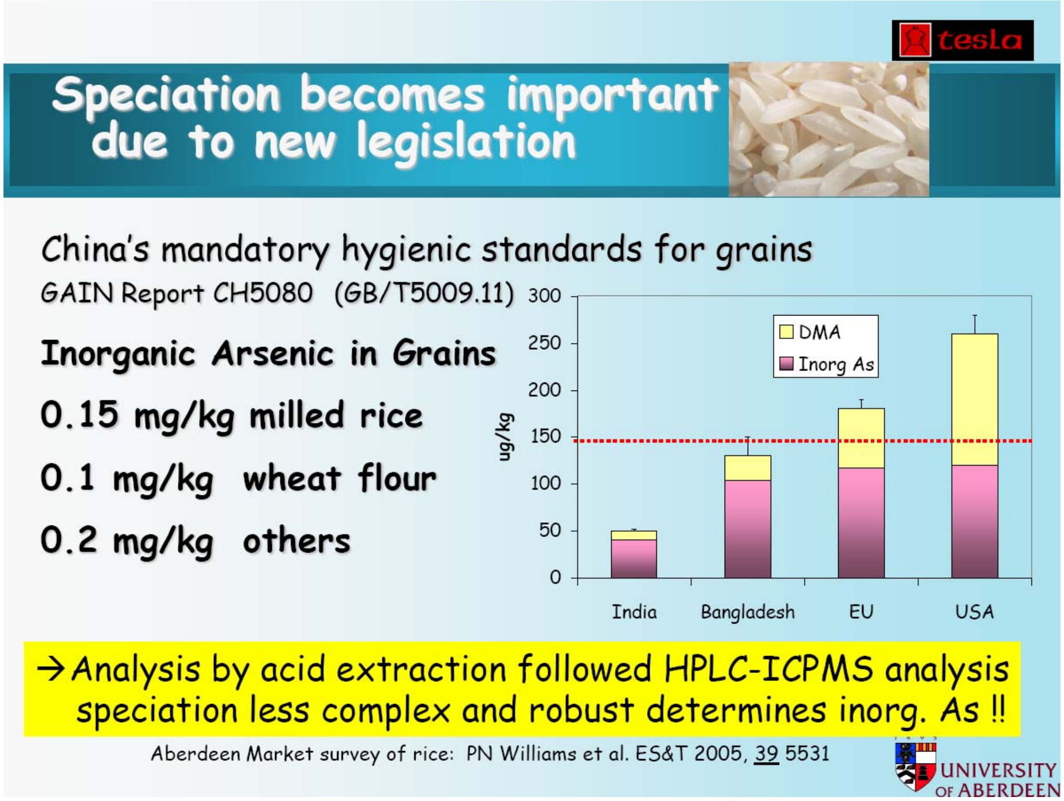

tesla

Speciation becomes important due to new legislation

China's mandatory hygienic standards for grains

GAIN Report CH5080 (GB/T5009.11)

Inorganic Arsenic in Grains

0.15 mg/kg milled rice

0.1 mg/kg wheat flour

0.2 mg/kg others

→Analysis by acid extraction followed HPLC-ICPMS analysis speciation less complex and robust determines inorg. As !!

Aberdeen Market survey of rice: PN Williams et al. ES&T 2005, 39 5531

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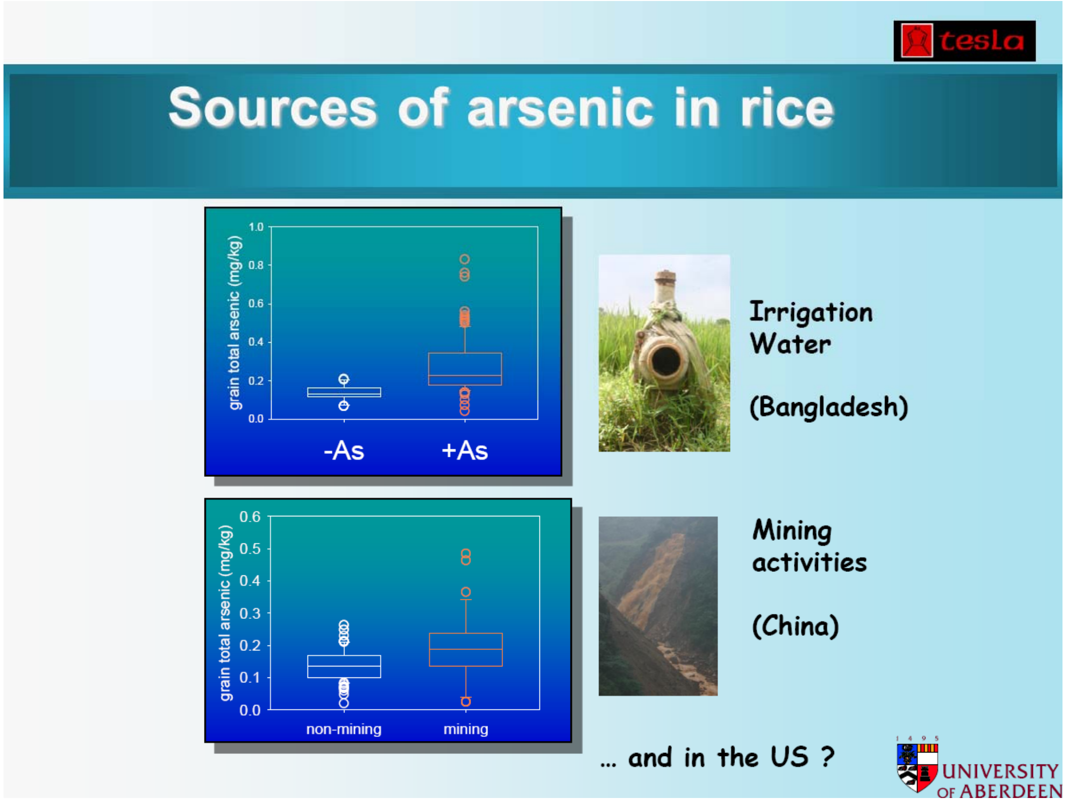


Sources of arsenic in rice

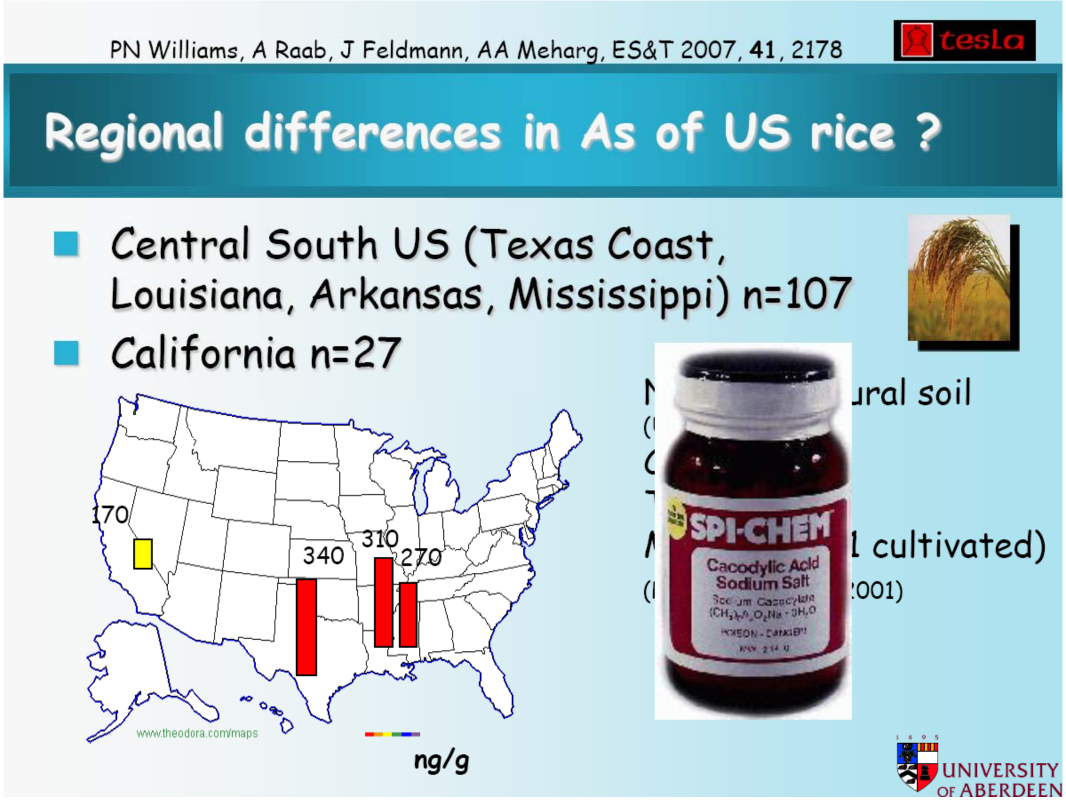
Irrigation Water

(Bangladesh)

Mining activities

(China)

… and in the US ?

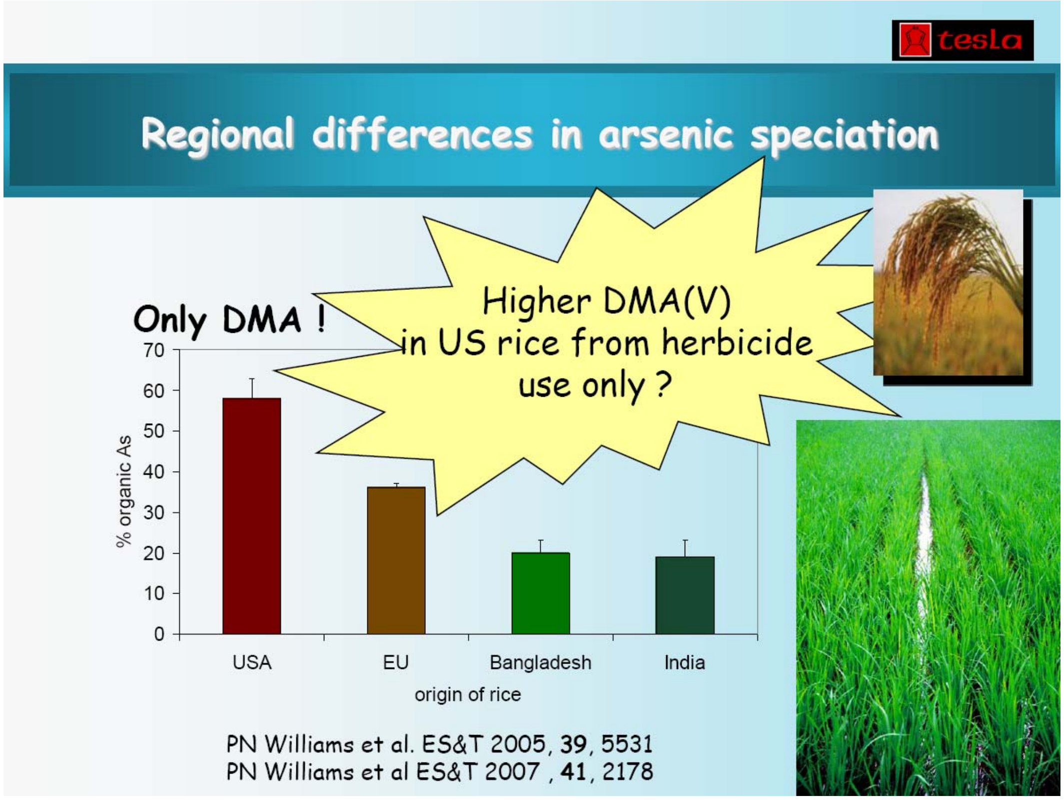


PN Williams, A Raab, J Feldmann, AA Meharg, ES&T 2007, **41**, 2178

Regional differences in As of US rice ?

- Central South US (Texas Coast, Louisiana, Arkansas, Mississippi) n=107
- California n=27

N ural soil
(
C
T
M 1 cultivated)
(:001)

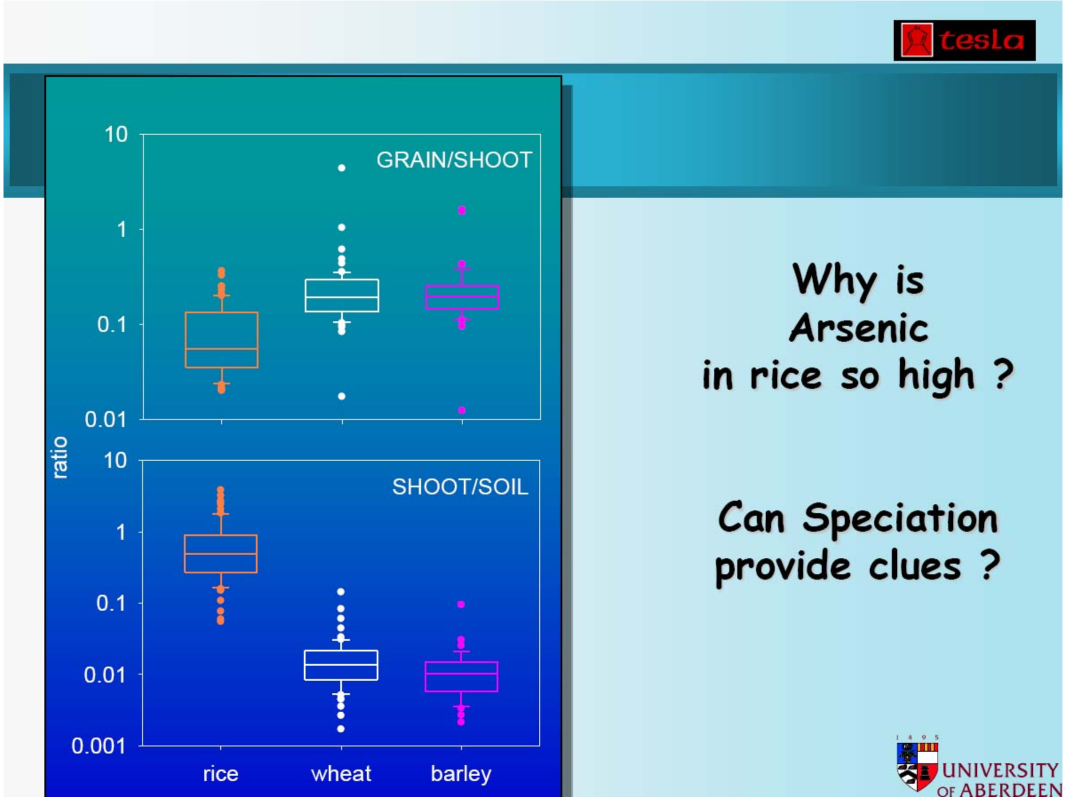


Regional differences in arsenic speciation

Only DMA !

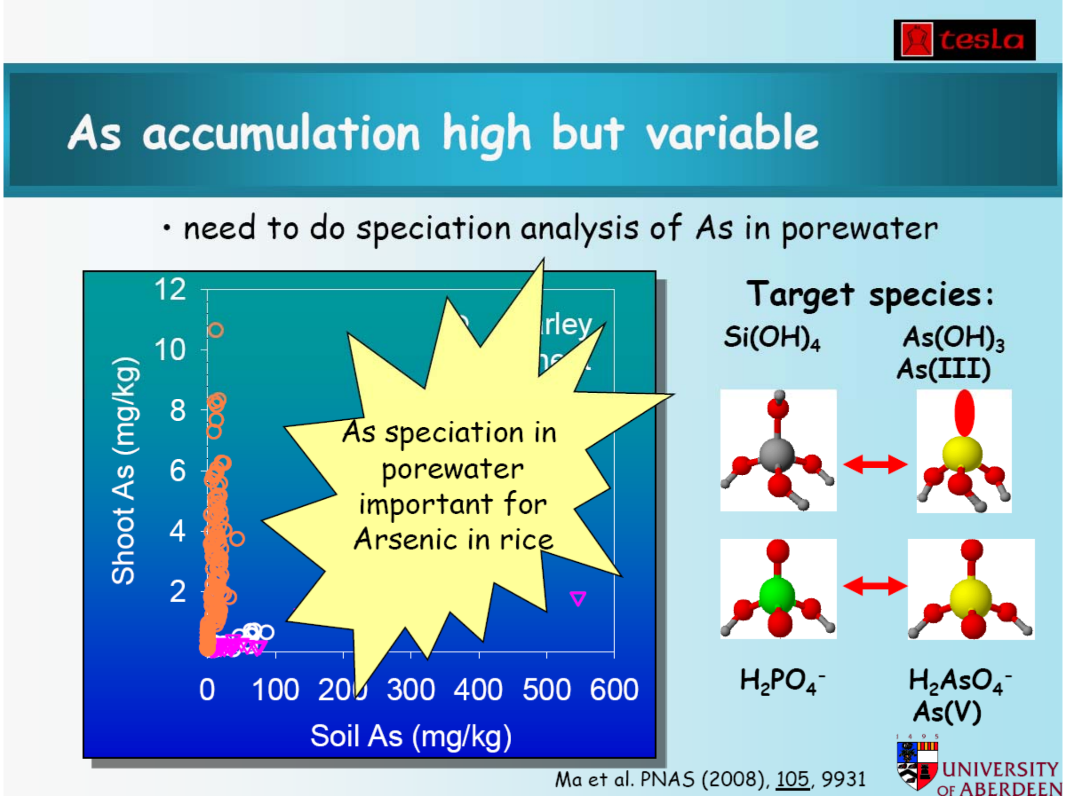
Higher DMA(V)
in US rice from herbicide
use only ?

PN Williams et al. ES&T 2005, **39**, 5531
PN Williams et al ES&T 2007 , **41**, 2178



Why is
Arsenic
in rice so high ?

Can Speciation
provide clues ?



As accumulation high but variable

- need to do speciation analysis of As in porewater

As speciation in porewater important for Arsenic in rice

Target species:

$Si(OH)_4$ ↔ $As(OH)_3$ As(III)

$H_2PO_4^-$ ↔ $H_2AsO_4^-$ As(V)

Ma et al. PNAS (2008), 105, 9931

Conclusion: As speciation in rice

- Less complex
- Robust analytical methodology available
  - to differentiate between inorganic As and DMA/MA in grains
  - to differentiate between As(III) and As(V) in soil porewater
- Speciation is needed
  - Legislation
  - Assessing the risk of accumulation and toxicity

Take home message

- Hg, Sn : target species identified and validated methodology developed
  → Robust enough for environmental monitoring
- As, Se: complex
  - Target species needs to be identified for type of sample
  - Validation of HPLC-ICPMS methods needs molecular information using additional ES-MS

  →today only robust method with limited scope can be implemented in large scale programs

Acknowledgments

TESLA: Trace Elemental Speciation Laboratory Aberdeen

http://www.abdn.ac.uk/chemistry/research/tesla/

Special thanks:
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METCO

EPA and Nanotechnology: Oversight for the 21st Century

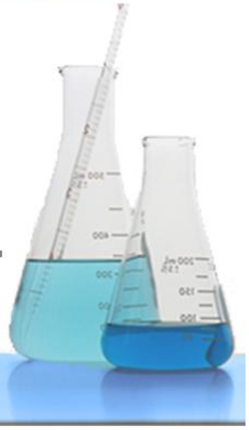
J. Clarence Davies
Resources for the Future
1616 P Street NW
Washington, DC 20036
202-328-5080
davies@rrf.org

ABSTRACT

Nanotechnology has enormous potential to improve everyone's life. For the potential to be realized, the new technology must be subject to an adequate oversight system, a system designed to identify and prevent any adverse effects of nano on health or the environment. The accelerating pace of nano discoveries and new products means that time is running short for the establishment of an oversight system. Action is needed now. EPA has a key role in nanotechnology oversight. The only existing general law that could provide the basis for overseeing the new technology is the Toxic Substances Control Act, although EPA has declined to use the law for this purpose. Many of the other laws administered by the agency also are essential to nano oversight, although the lack of scientific information about nanomaterials and the inadequacy of methods for monitoring workplace and ambient levels of the materials are major impediments. There are a large number of steps that can and should be taken now to provide oversight for nano. However, future steps will depend on learning more about the risks of nano, on our ability to monitor nanomaterials, and on developments in nanotechnology itself.

THE NELAC INSTITUTE

MOVING FORWARD ON NATIONAL ACCREDITATION



Bob Wyeth

Columbia Analytical Services, Inc.

THE NELAC INSTITUTE

Who is TNI?

- A 501(c)3 non-profit organization.
- A member organization managed by a Board of Directors.
- A voluntary consensus standards development organization accredited by the American National Standards Institute (ANSI).

Our Heritage

- National Environmental Laboratory Accreditation Conference (NELAC), 1995 – 2008:
  - An unincorporated organization of federal and state officials.
- Institute for National Environmental Laboratory Accreditation (INELA), 2002 – 2006:
  - An incorporated 501(c)3 non-profit organization.
- The NELAC Institute was formed on November 6, 2006 by:
  - combining the operations of the two organizations,
  - adopting new Bylaws,
  - establishing a transition Board of Directors, and
  - modifying the INELA Articles of Incorporation.

TNI: A 501(c)3 Organization

Board of Directors

Self-Governing Programs
- ☐ NELAP
- ☐ LASP
- ☐ TAP
- ☐ CSDP
- ☐ PTP
- ☐ Forum
- ☐ NEMC

Staff

Administrative Committees
- ☐ Policy
- ☐ Advocacy
- ☐ Website
- ☐ Conference Planning

Individual Members

Mission and Vision

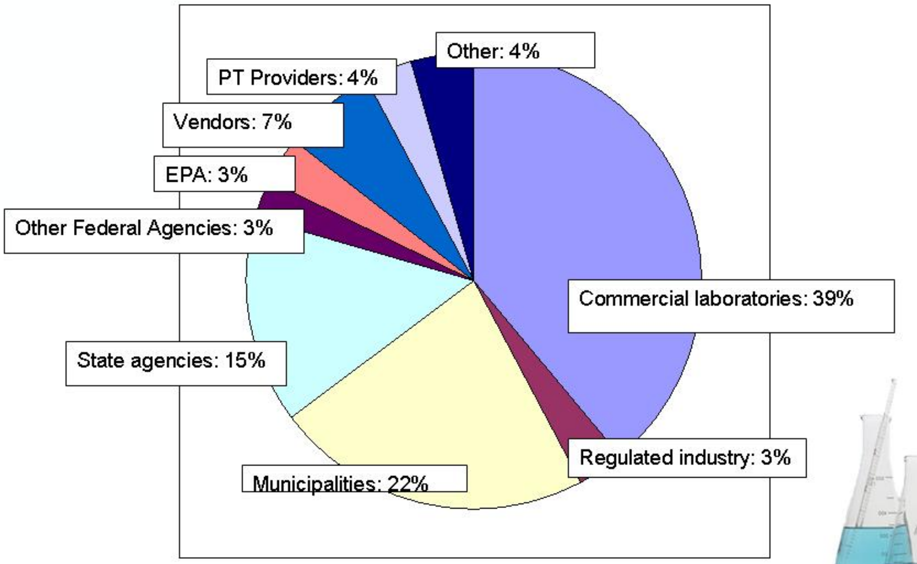
Foster the generation of environmental data of known and documented quality through an open, inclusive and transparent process that is responsive to the needs of the community.

All entities generating environmental data in the United States will be accredited to consensus national standards.

Who Are Our Members?

- **Organizations that accredit laboratories**
  - Recognized accreditation bodies
  - States that are not recognized accreditation bodies
  - Federal agencies that operate accreditation programs
- **Accredited laboratories**
  - Commercial, Municipal, University, State, Federal, etc.
- **Others**
  - State and federal agencies that do not operate accreditation programs
  - Data users, consultants, PT Providers, vendors, etc.
  - Anyone interested in laboratory accreditation

Member Demographics



Members

- Individual Members
  - May vote
  - May serve on committees or the Board
  - Entitled to member discounts
  - Access to members area of website
- Organizational Members
  - May appoint individual members
  - Are recognized
  - May not vote

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What do we Provide?

- An infrastructure for stakeholders to discuss issues.
- A consensus process for the operation of accreditation programs:
  - Requirements for Accreditors,
  - Requirements for laboratories that are accredited, and
  - Requirements for a private sector proficiency testing program.
- A process to formally recognize accreditation programs.
- Assistance to laboratories and regulatory agencies

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Programs of TNI

- ☐ Consensus Standards Development
- ☐ Laboratory Accreditation System
- ☐ National Environmental Laboratory Accreditation
- ☐ Proficiency Testing
- ☐ Technical Assistance
- ☐ Forum on Laboratory Accreditation
- ☐ National Environmental Monitoring Conference (NEMC)

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Consensus Standards Development Program

- Develop standards for the accreditation of environmental laboratories.

Expert Committees

- Accreditation Body
- Field Activities
- On-site Assessment
- Proficiency Testing
- Quality Systems

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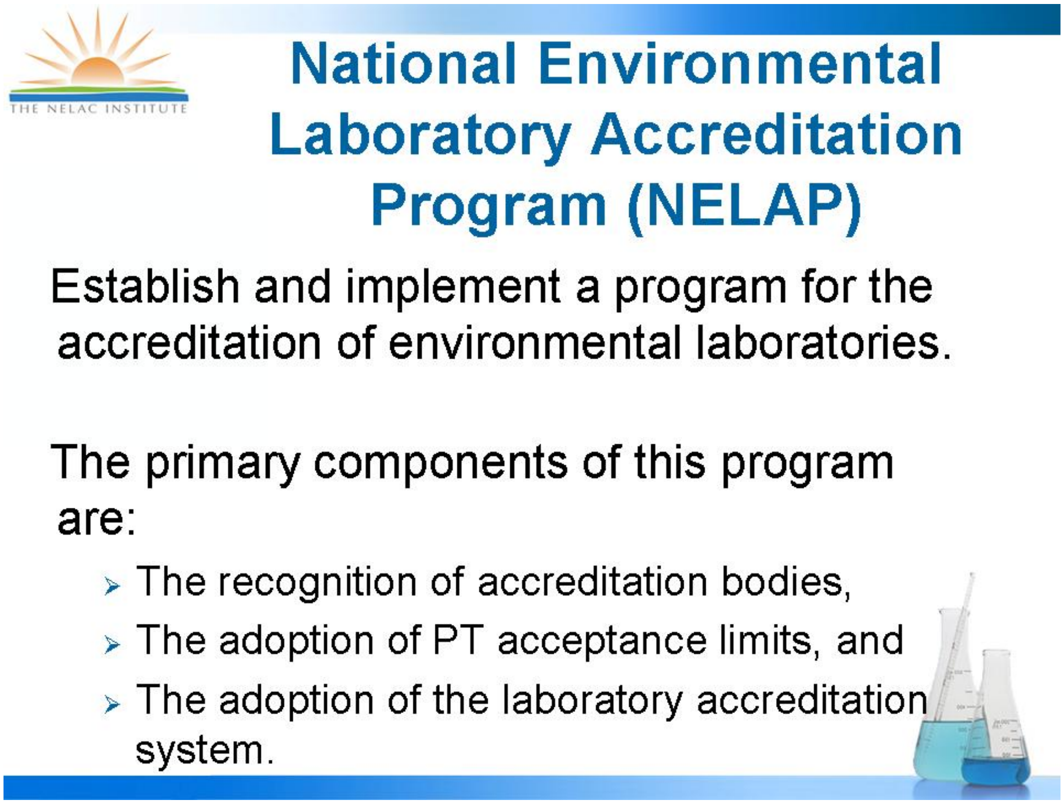
Laboratory Accreditation System Program

- Develop a system for the accreditation of environmental laboratories:
    - policies and procedures, interpretations, & guidance documents
- National Database Subcommittee



National Environmental Laboratory Accreditation Program (NELAP)

Establish and implement a program for the accreditation of environmental laboratories.

The primary components of this program are:

- The recognition of accreditation bodies,
- The adoption of PT acceptance limits, and
- The adoption of the laboratory accreditation system.

NELAP Board

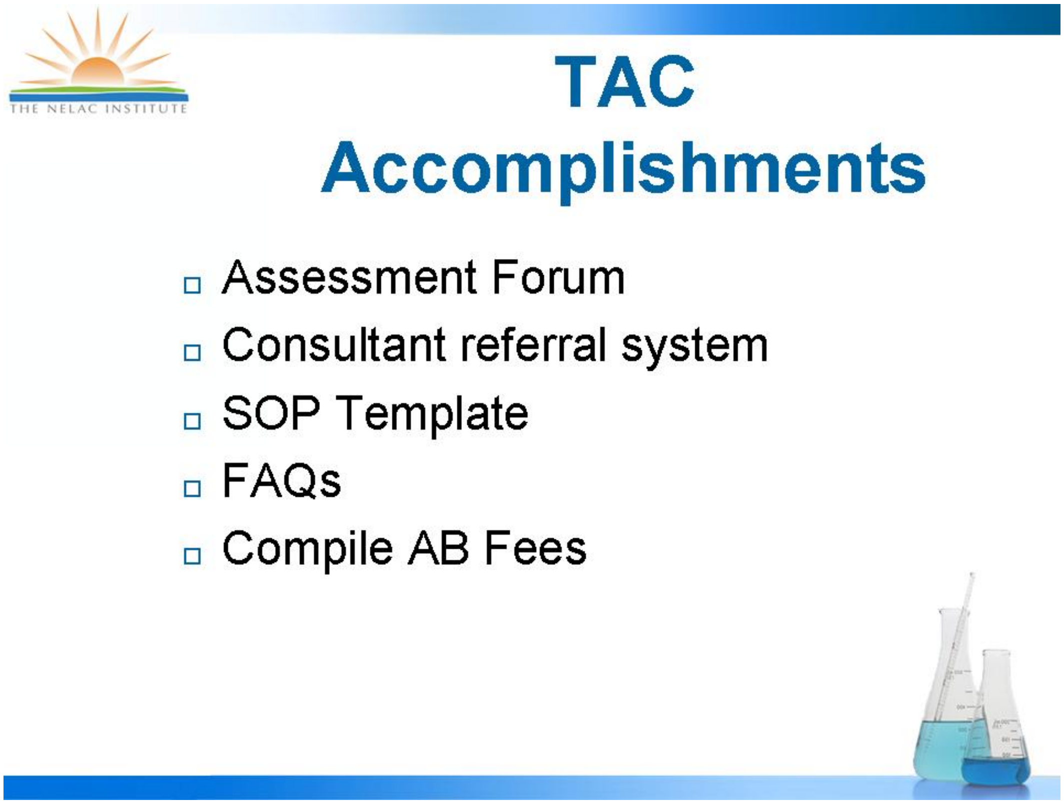
- One representative and one alternate appointed by each Accreditation Body.
- Final authority for implementation of the program.

Proficiency Testing (PT) Board

- Selection of a PT Provider Accreditor (PTPA).
- Monitors the PTPA to assure compliance with TNI.
- Facilitate annual caucus on proficiency testing.
- Evaluate PT data for the appropriateness of study limits.
- Provide recommendations to the NELAP Board as to acceptance limits.
- PT Board develops requirements, provides oversight of independent providers and sets policy.

Technical Assistance Program

- Develop tools and templates to assist laboratories and accreditation bodies.
- Ensure that relevant training programs are provided.
- Ensure that laboratory assessors have a forum to discuss common issues.
- Develop a mentoring program to assist both laboratories and accreditation bodies with implementing accreditation programs.



TAC Accomplishments

- Assessment Forum
- Consultant referral system
- SOP Template
- FAQs
- Compile AB Fees

National Environmental Monitoring Conference

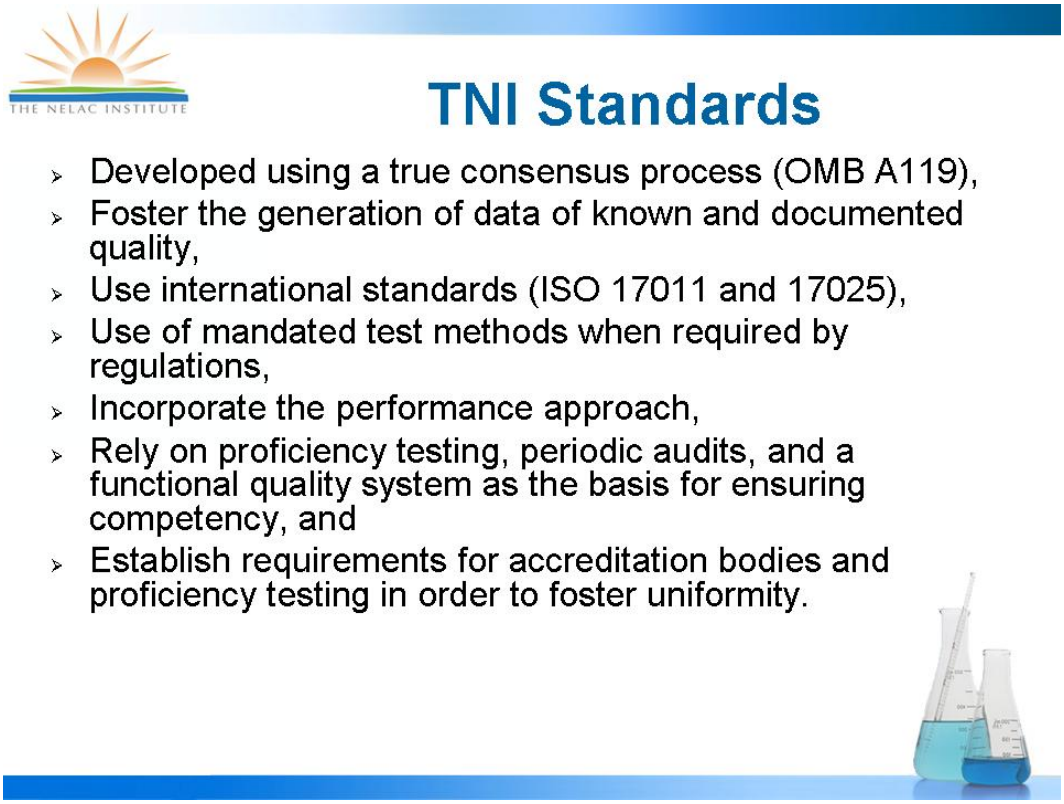
- Annual technical meeting focused on the latest innovations in environmental monitoring
- Co-hosted with EPA and the Independent Laboratories Institute
- August 11-15, 2008 Washington, DC
- www.nemc.us

Forum on Laboratory Accreditation

- Semiannual meeting where TNI committees, members, and others meet to discuss common issues
- August 11-15, 2008, Washington, DC
- January 12-16, 2009, Miami, FL
- August 10-14, 2009, San Antonio!

TNI Standards

Where are we now?



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TNI Standards

- Developed using a true consensus process (OMB A119),
- Foster the generation of data of known and documented quality,
- Use international standards (ISO 17011 and 17025),
- Use of mandated test methods when required by regulations,
- Incorporate the performance approach,
- Rely on proficiency testing, periodic audits, and a functional quality system as the basis for ensuring competency, and
- Establish requirements for accreditation bodies and proficiency testing in order to foster uniformity.

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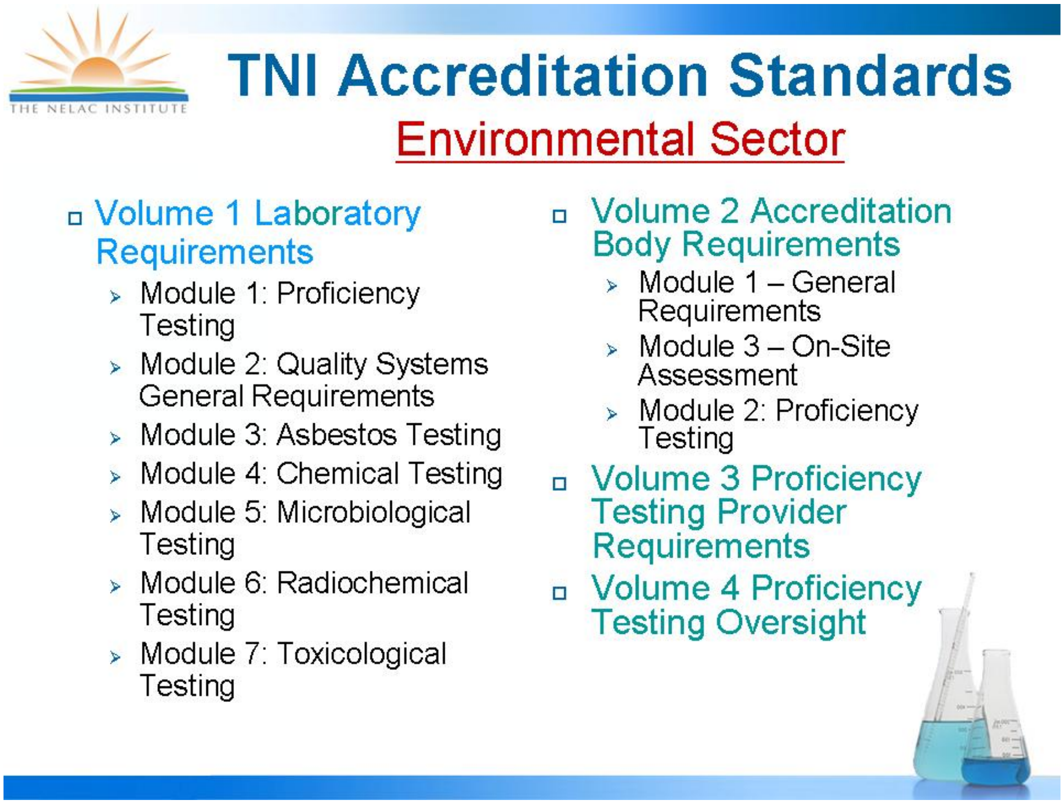
Status of TNI Standards

- All standards have been approved and are being considered for adoption into the program
- Standards are available on the TNI website
  - ISO version available for purchase
- TNI Standards will replace the 2003 NELAC standard in ~2010
  - Implementation of the TNI Standard is a major on-going effort of all programs and committees



TNI Accreditation Standards

Environmental Sector

- Volume 1 Laboratory Requirements
  - Module 1: Proficiency Testing
  - Module 2: Quality Systems General Requirements
  - Module 3: Asbestos Testing
  - Module 4: Chemical Testing
  - Module 5: Microbiological Testing
  - Module 6: Radiochemical Testing
  - Module 7: Toxicological Testing
- Volume 2 Accreditation Body Requirements
  - Module 1 – General Requirements
  - Module 3 – On-Site Assessment
  - Module 2: Proficiency Testing
- Volume 3 Proficiency Testing Provider Requirements
- Volume 4 Proficiency Testing Oversight

TNI Accreditation Standards

Field Sampling and Measurement Organization Sector

Volume 1: General requirements for FSMOs

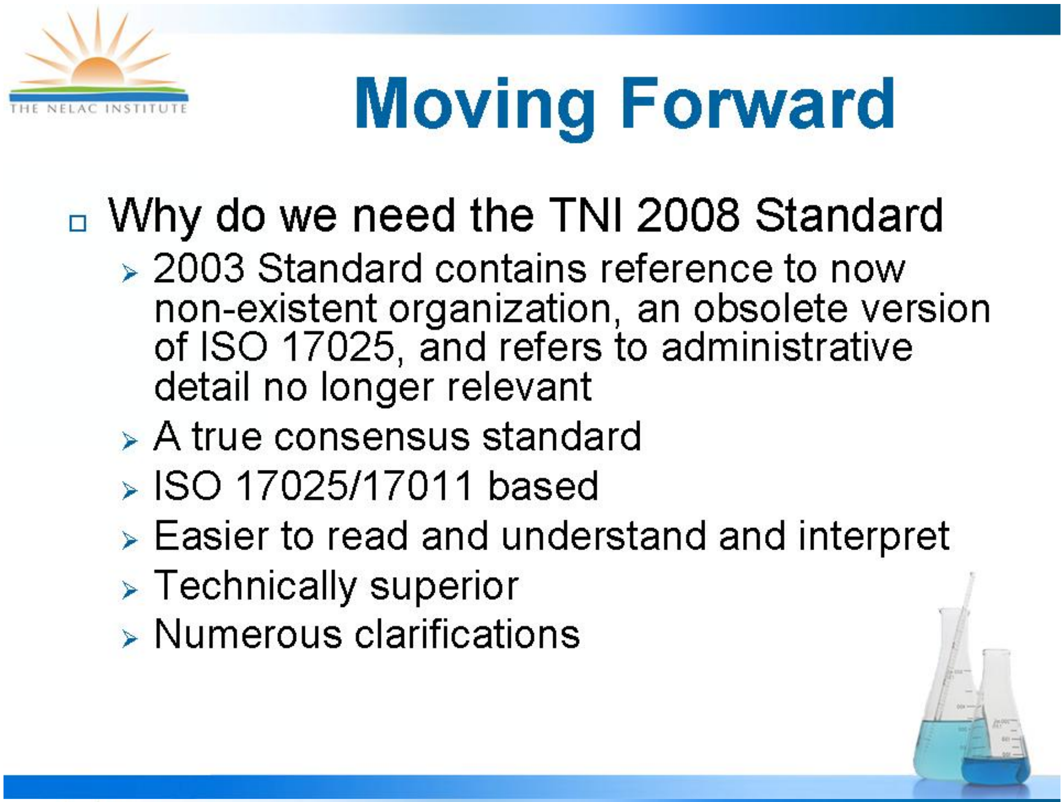
Volume 2: General Requirements for Accreditation Bodies Accrediting FSMOs

Moving Forward

- Need to address concerns of the accreditation effort.
  - True national program not yet achieved
  - Some stakeholders still do not support the national accreditation effort
  - No standards beyond 2003 adopted by NELAP
  - Requirements appear onerous to small laboratories

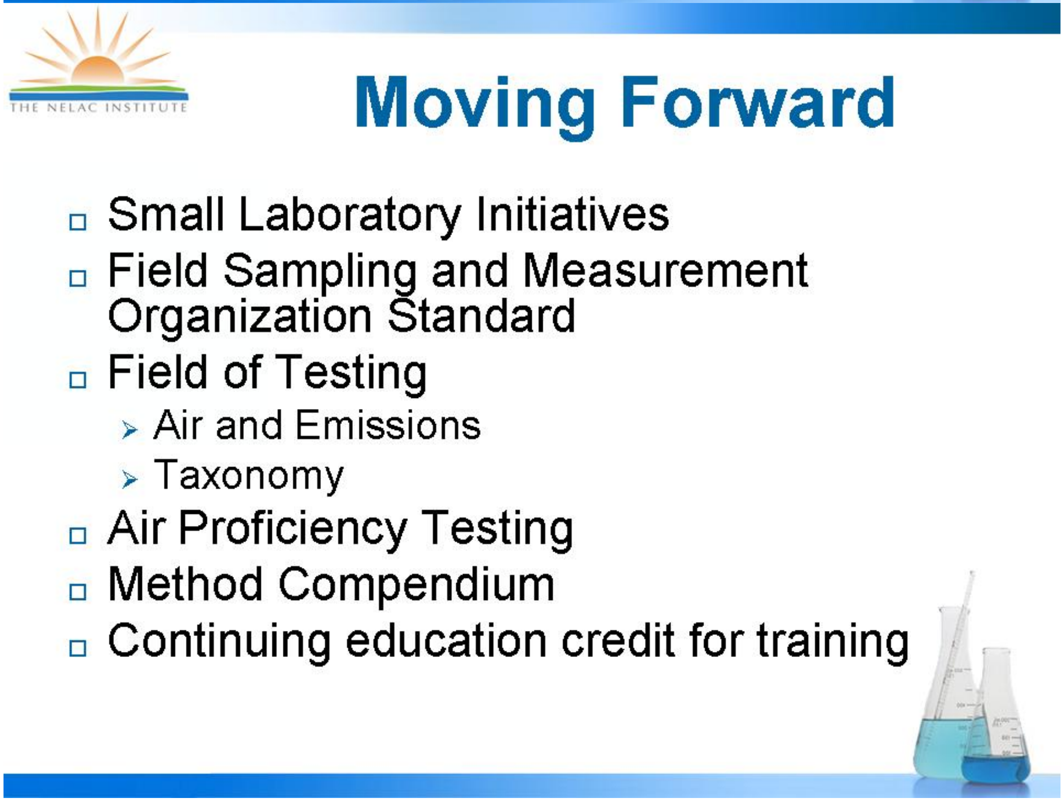
Advocacy Committee

- Promote a national program for the accreditation of environmental laboratories
  - Establish relationships with other organizations (e.g., ACIL, AWWA, APHL, WEF) that have an interest in accreditation issues.
  - Establish relationships with EPA program offices.
  - Develop presentations and papers to promote national accreditation.



Moving Forward

- Why do we need the TNI 2008 Standard
  - 2003 Standard contains reference to now non-existent organization, an obsolete version of ISO 17025, and refers to administrative detail no longer relevant
  - A true consensus standard
  - ISO 17025/17011 based
  - Easier to read and understand and interpret
  - Technically superior
  - Numerous clarifications



Moving Forward

- Small Laboratory Initiatives
- Field Sampling and Measurement Organization Standard
- Field of Testing
  - Air and Emissions
  - Taxonomy
- Air Proficiency Testing
- Method Compendium
- Continuing education credit for training

Summary

- The NELAC Institute is working to take national accreditation to the next level
  - Improve the accreditation requirements
  - Approve more states as accreditation bodies
  - Be responsive to stakeholder needs
  - Provide technical assistance
- We need your help!
  - Join our organization
  - Join a committee

The NELAC Institute

http://www.NELAC-Institute.org

817-598-1624

jerry.parr@nelac-institute.org

Global Monitoring of Persistant Organic Pollutants (POPs): UNEP's Experiences and First Results

Heidelore Fiedler
United Nations Environmental Program
Geneva, Switzerland
+41-22-917-8187
hfiedler@chemicals.unep.ch

ABSTRACT

The Stockholm Convention on Persistent Organic Pollutants (POPs) entered into force on 17 May 2004 and presently has 153 Parties. The initial twelve POPs include eight pesticides, two industrial chemicals, and two unintentional chemicals. The Convention makes provisions for POPs analysis in three areas:

1. Effectiveness evaluation (Article 16). Global Monitoring Plan (GMP) focuses on two core media: mother's milk/human blood to examine human exposure and ambient air for long-range transport;
2. Performance levels for PCDD/PCDF in stack emissions that are associated with the application of best available techniques (Article 5); these performance levels range from 0.1 to 0.5 ng TEQ/Nm³ in stack emissions;
3. Provisional limit values for "low POP content" (Article 6) for POPs wastes according to Technical Guidelines by the Basel Convention: 50 mg/kg for each of the "basic POPs" (aldrin, chlordane, dieldrin, DDT, endrin, heptachlor, hexachlorobenzene, mirex, toxaphene, and PCB) and 15 mg TEQ/kg for PCDD/PCDF.

A UNEP/GEF project has established general guidance for POPs analysis and a databank of operating POPs laboratories. The recommended instrumentation for POPs analysis are capillary gas chromatography coupled to electron capture or mass-selective detectors. The methods applied should be validated and results confirmed through successful participation in international intercalibration studies. The feasibility study with nine laboratories in seven countries has shown the following:

1. Laboratories often work in isolation and family feeling between laboratories should be created;
2. Infrastructure including main instrumentation adequate, but consumables generally of poor quality;
3. Some laboratories were accredited according to ISO 17025;
4. CRMs and LRMs generally not available;
5. Intercalibration studies revealed problems: PCB were more accurately determined than OCPs in all matrices. The two dioxin laboratories performed well for PCDD/PCDF at high concentrations; at lower concentrations and with new matrices some difficulties occurred.

The study was funded through the medium-sized GEF-funded Project "Assessment of Existing Capacity and Capacity Building Needs to Analyse POPs in Developing Countries" with cofinancing from the governments of Canada, Germany, and Japan.

NEMC 2008

Global Monitoring of Persistent Organic Pollutants (POPs): UNEP's Experiences and First Results

Dr. Heidelore Fiedler
United Nations Environment Programme (UNEP)
Chemicals Branch, DTIE
11-13, chemin des Anémones
CH-1219 Châtelaine (GE), Switzerland
E-mail: hfiedler@chemicals.unep.ch

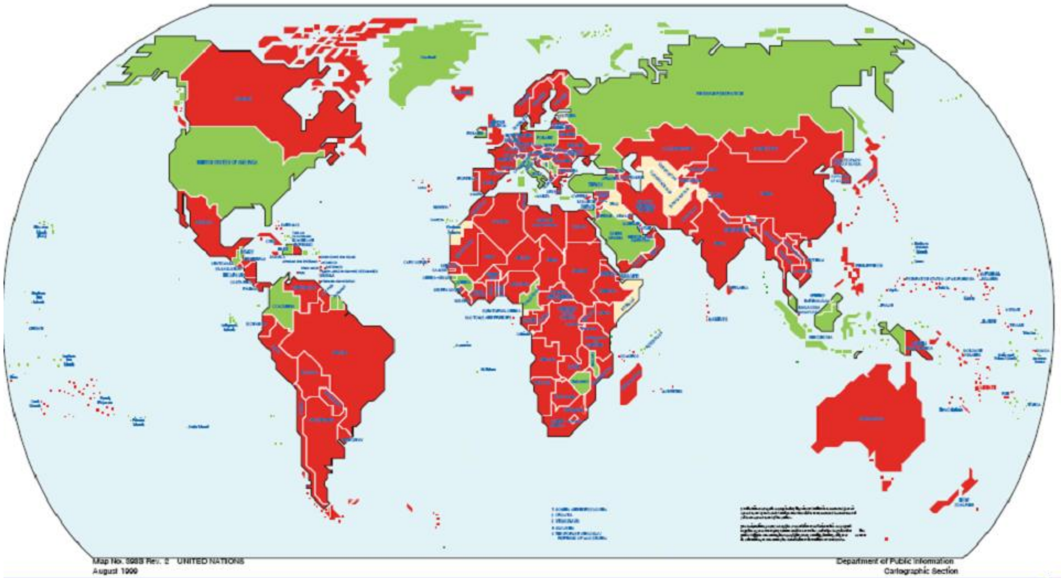
Content

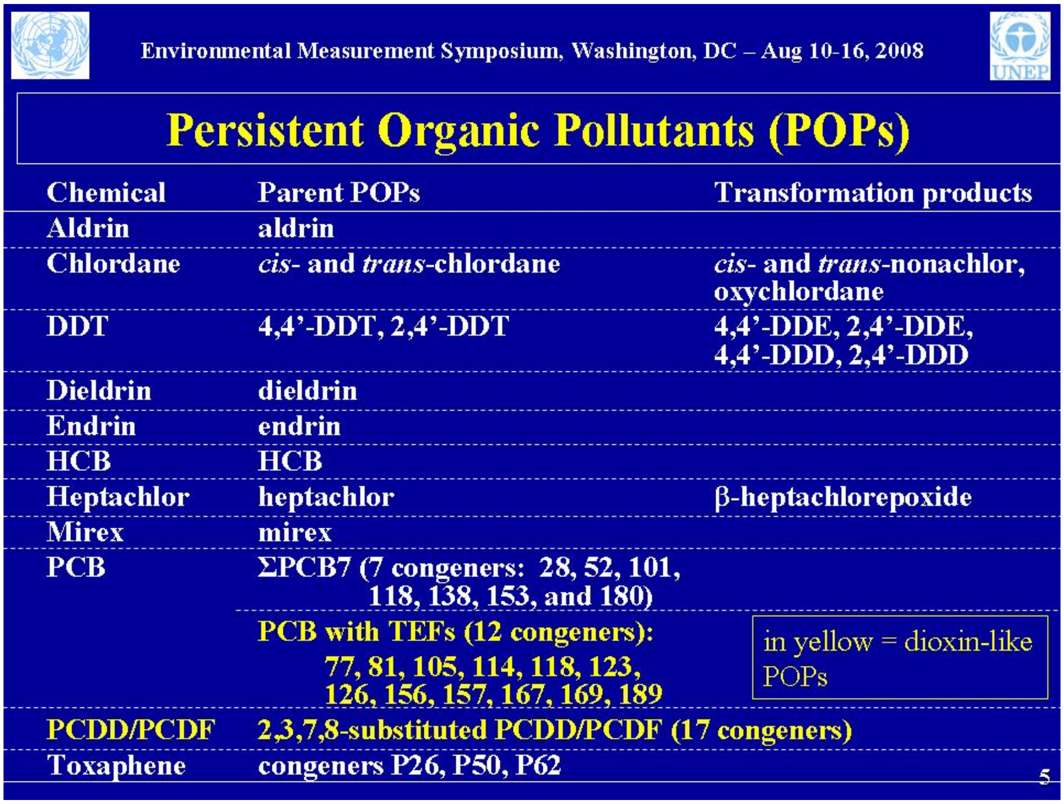
- **Background on Stockholm Convention on Persistent Organic Pollutants**
- **Global Monitoring Plan (GMP)**
- **Guidance Documents**
- **POPs Laboratory Databank**
- **Capacity Building Activities in Developing Countries**
- **Results of Interlaboratory Intercalibration Studies**
- **Conclusions**
- **Outlook**

Stockholm Convention on POPs

- **POPs = persistent organic pollutants;**
- **POPs**
  - have toxic properties,
  - resist degradation,
  - bioaccumulate and are transported, through air, water and migratory species, across international boundaries, and
  - [are] deposited far from their place of release, where they accumulate in terrestrial and aquatic ecosystems;
- **Stockholm Convention is a legally binding instrument, Secretariat located at UNEP (Geneva-Switzerland);**
- **Conference of the parties (COP) is governing body;**
- **Objectives: to protect human health and the environment from persistent organic pollutants.**

Stockholm Convention on POPs entered into force on 17 May 2004; Today it has 157 parties and 152 signatories

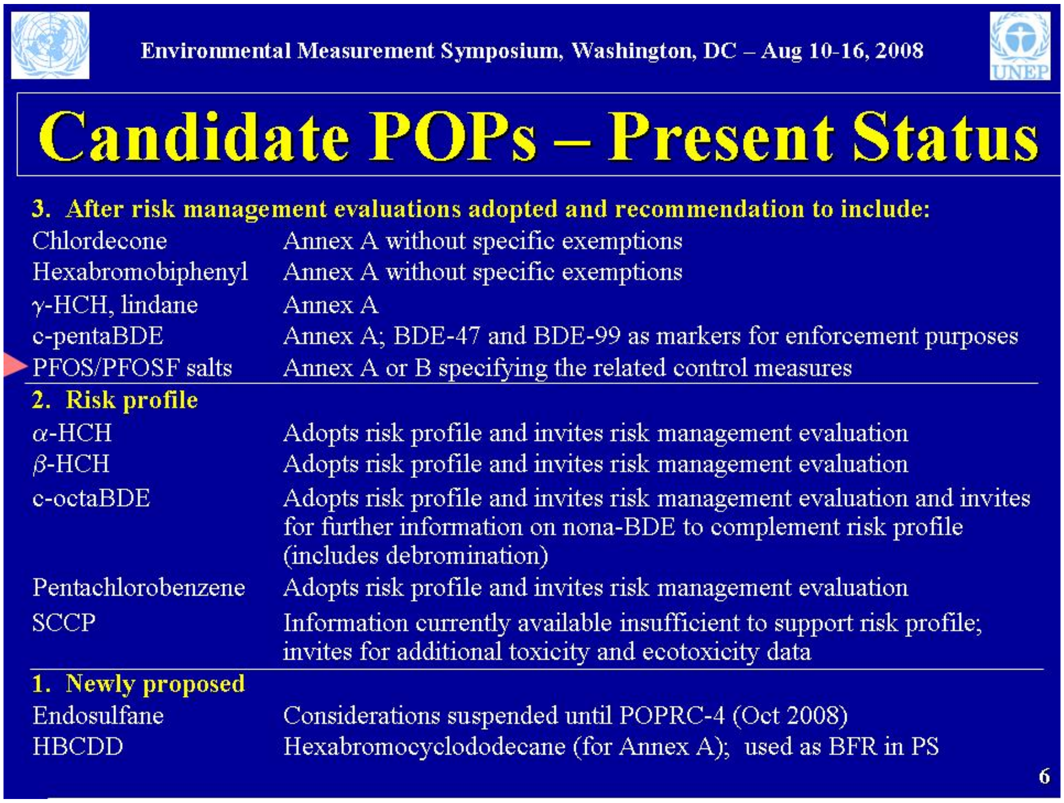




Persistent Organic Pollutants (POPs)

Chemical	Parent POPs	Transformation products
Aldrin	aldrin	
Chlordane	*cis*- and *trans*-chlordane	*cis*- and *trans*-nonachlor, oxychlordane
DDT	4,4'-DDT, 2,4'-DDT	4,4'-DDE, 2,4'-DDE, 4,4'-DDD, 2,4'-DDD
Dieldrin	dieldrin	
Endrin	endrin	
HCB	HCB	
Heptachlor	heptachlor	β-heptachlorepoxide
Mirex	mirex	
PCB	ΣPCB7 (7 congeners: 28, 52, 101, 118, 138, 153, and 180)	
	PCB with TEFs (12 congeners): 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, 189	
PCDD/PCDF	2,3,7,8-substituted PCDD/PCDF (17 congeners)	
Toxaphene	congeners P26, P50, P62	

in yellow = dioxin-like POPs



Candidate POPs – Present Status

3. After risk management evaluations adopted and recommendation to include:

Chlordecone	Annex A without specific exemptions
Hexabromobiphenyl	Annex A without specific exemptions
γ-HCH, lindane	Annex A
c-pentaBDE	Annex A; BDE-47 and BDE-99 as markers for enforcement purposes
PFOS/PFOSF salts	Annex A or B specifying the related control measures

2. Risk profile

α-HCH	Adopts risk profile and invites risk management evaluation
β-HCH	Adopts risk profile and invites risk management evaluation
c-octaBDE	Adopts risk profile and invites risk management evaluation and invites for further information on nona-BDE to complement risk profile (includes debromination)
Pentachlorobenzene	Adopts risk profile and invites risk management evaluation
SCCP	Information currently available insufficient to support risk profile; invites for additional toxicity and ecotoxicity data

1. Newly proposed

Endosulfane	Considerations suspended until POPRC-4 (Oct 2008)
HBCDD	Hexabromocyclododecane (for Annex A); used as BFR in PS

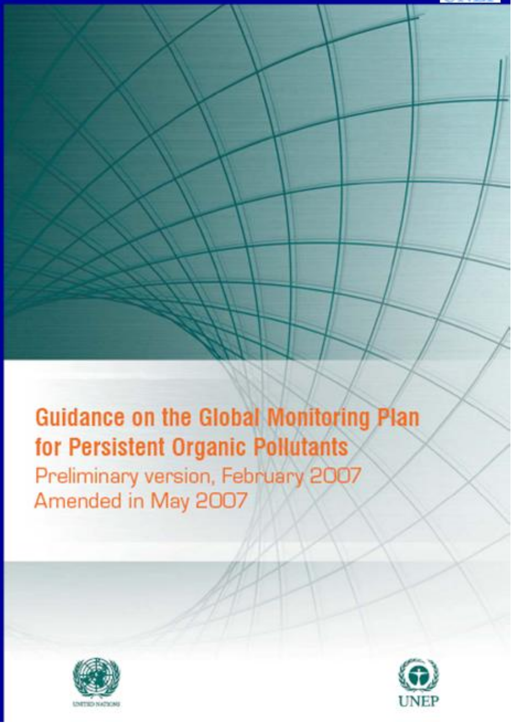
Convention-driven Country Needs

1. Effectiveness evaluation (art. 16) = Monitoring of all 12 POPs in humans (mothers' milk and human blood) and environment (air) for trend analysis
   (temporal = 50% reduction over 10 years; and spatial);
2. Performance levels of PCDD/PCDF in stack emissions of stationary sources (← art. 5) that are associated with BAT (0.1 / 0.2 / 0.5 ng TEQ/Nm³);
3. Limit values for "low POP content" in waste (← art. 6):
   50 mg/kg for aldrin, chlordane, dieldrin, DDT, endrin, heptachlor, HCB, mirex, toxaphene, PCB
   15 µg TEQ/kg for PCDD/PCDF.

Global Monitoring of POPs (GMP)

- Guidelines developed and approved for three core matrices:
  - ambient air,
  - mothers' milk, human blood;
- ⇒ Reports on regional basis for COP-4 (5/2009)

www.pops.int



Guidance on the Global Monitoring Plan for Persistent Organic Pollutants

Preliminary version, February 2007

Amended in May 2007

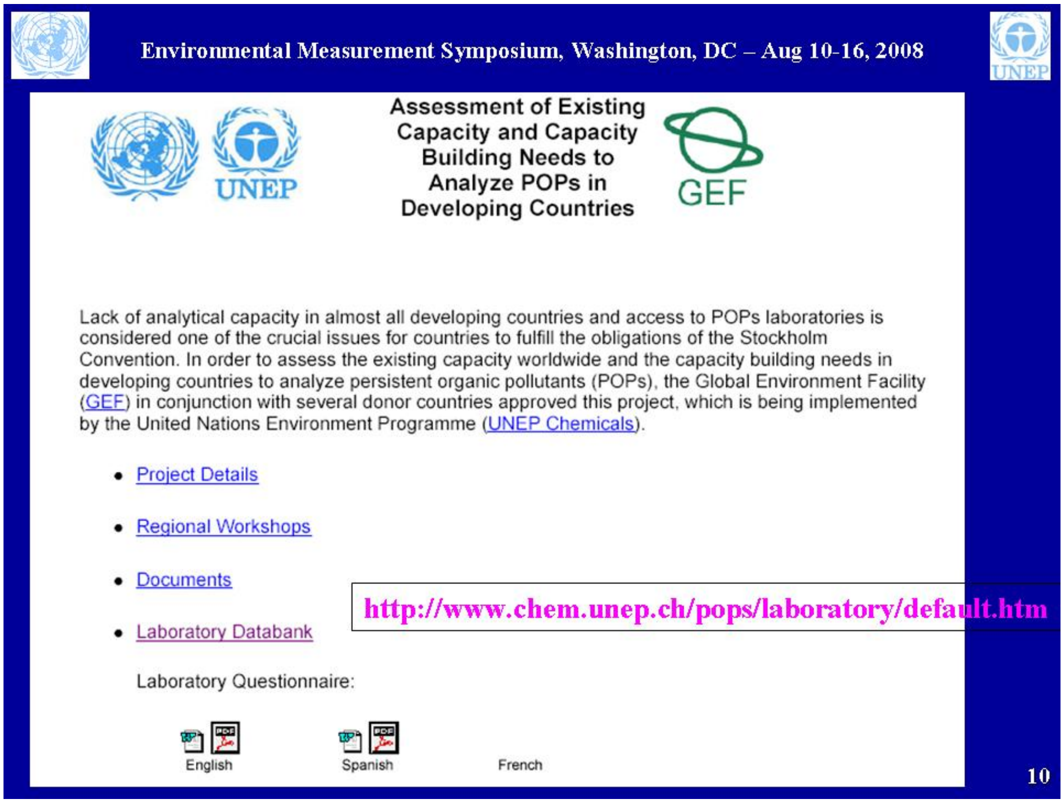
UNEP/GEF Project on POPs Laboratories

Objectives:

- Assessment of Convention-driven country needs for laboratory analysis and conditions necessary to conduct them in a sustainable manner;
- If appropriate on a regional basis;
- Focus on analysis of 12 Stockholm POPs in relevant matrices.

Outcomes:

1. Map of operational laboratories world-wide according to their capabilities to analyze classes of POPs in various matrices. Data to be stored in a searchable and web-accessible databank;
2. Recommended criteria for:
   (a) Sampling, separation, identification, and quantification;
   (b) Sustainability for operating POPs laboratories.



Assessment of Existing Capacity and Capacity Building Needs to Analyze POPs in Developing Countries

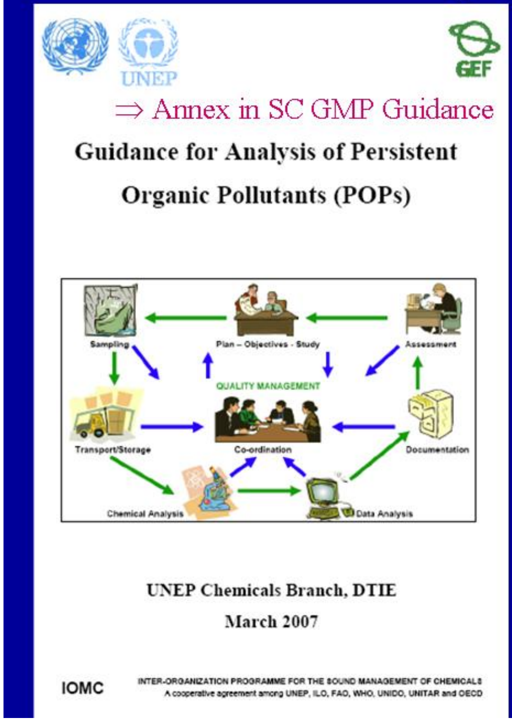
Lack of analytical capacity in almost all developing countries and access to POPs laboratories is considered one of the crucial issues for countries to fulfill the obligations of the Stockholm Convention. In order to assess the existing capacity worldwide and the capacity building needs in developing countries to analyze persistent organic pollutants (POPs), the Global Environment Facility (GEF) in conjunction with several donor countries approved this project, which is being implemented by the United Nations Environment Programme (UNEP Chemicals).

- Project Details
- Regional Workshops
- Documents
- Laboratory Databank

Laboratory Questionnaire:

http://www.chem.unep.ch/pops/laboratory/default.htm

UNEP/GEF Project on POPs Analysis

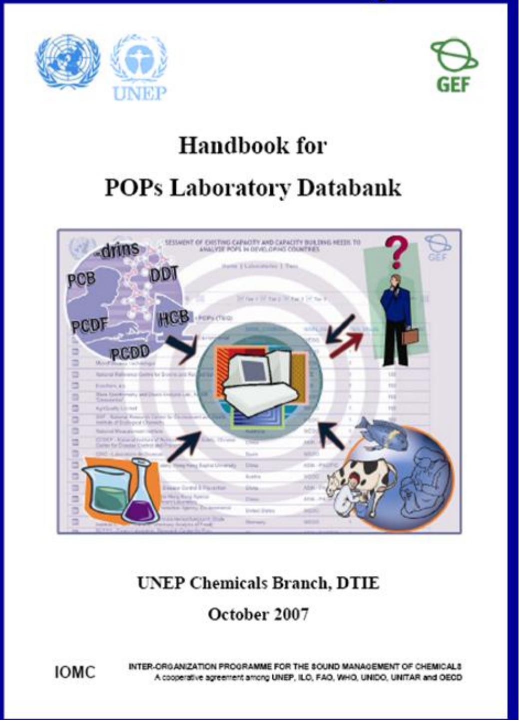


⇒ Annex in SC GMP Guidance

Guidance for Analysis of Persistent
Organic Pollutants (POPs)

UNEP Chemicals Branch, DTIE
March 2007

IOMC INTER-ORGANIZATION PROGRAMME FOR THE SOUND MANAGEMENT OF CHEMICALS
A cooperative agreement among UNEP, ILO, FAO, WHO, UNIDO, UNITAR and OECD



Handbook for
POPs Laboratory Databank

UNEP Chemicals Branch, DTIE
October 2007

IOMC INTER-ORGANIZATION PROGRAMME FOR THE SOUND MANAGEMENT OF CHEMICALS
A cooperative agreement among UNEP, ILO, FAO, WHO, UNIDO, UNITAR and OECD

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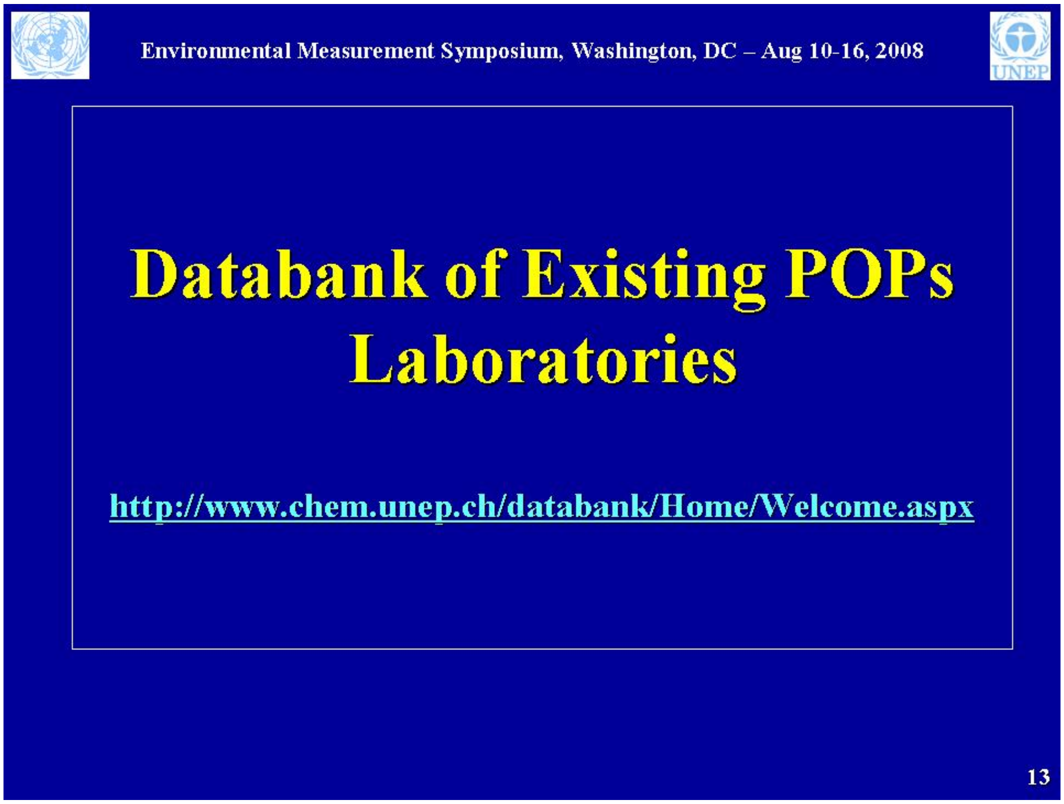
Guidance for POPs Analysis Includes:

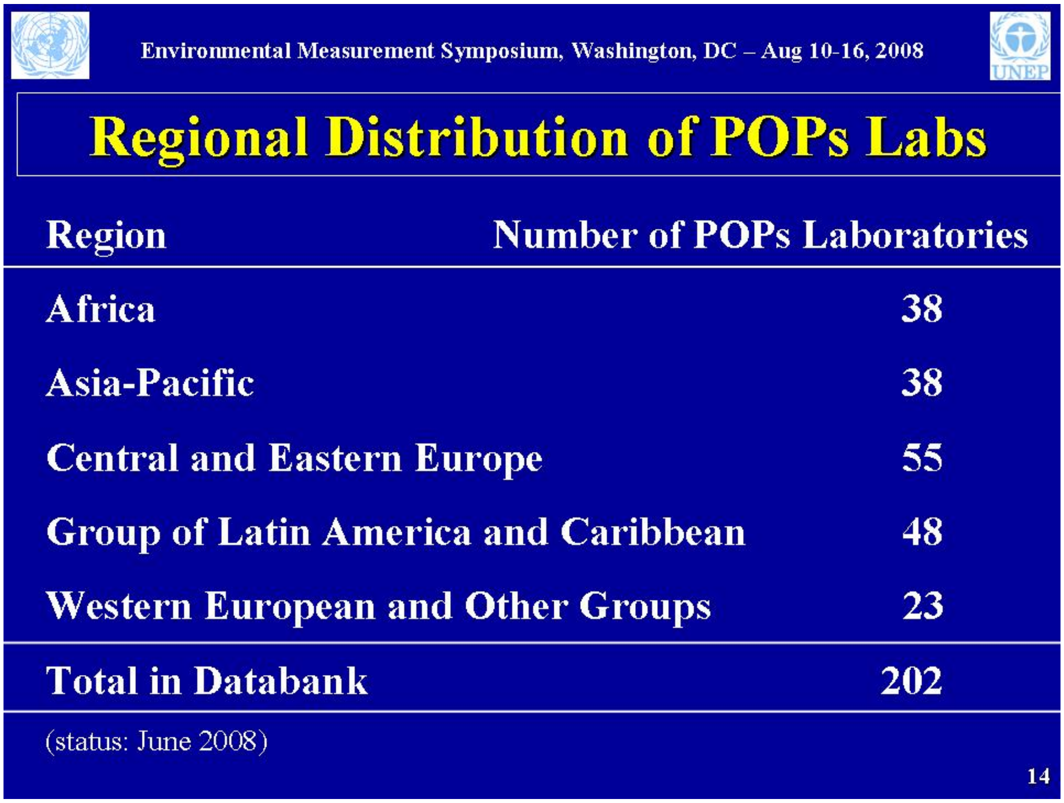
- Extraction recoveries as a general rule:
  - PCB and OCPs: 80 %-120 % (Cl_4/Cl_5: 60 % acceptable)
  - PCDD/PCDF: 50 %-130 % (Cl_7/Cl_8: 40%-150 % acceptable);
- Addition of internal standard for signal/noise at least at 20/1;
- Identification with ECD:
  Confirmation of peaks on 2nd column with different polarity. Alternatively, analyte additions can be used;
- For ECD and PCB: recommended standards = PCB #112, #155, and #198. PCB #209 is not recommended;
- Reporting (quantification) value should be at least 1/5 of the regulatory limit or level of interest or baseline concentration;
- Difference between lower-bound and upper-bound value at the regulatory level/level of interest should be less than 20 %.

12



Databank of Existing POPs Laboratories

http://www.chem.unep.ch/databank/Home/Welcome.aspx



Regional Distribution of POPs Labs

Region	Number of POPs Laboratories
Africa	38
Asia-Pacific	38
Central and Eastern Europe	55
Group of Latin America and Caribbean	48
Western European and Other Groups	23
Total in Databank	**202**

(status: June 2008)

Overview on POPs Analyzed (202 Labs)

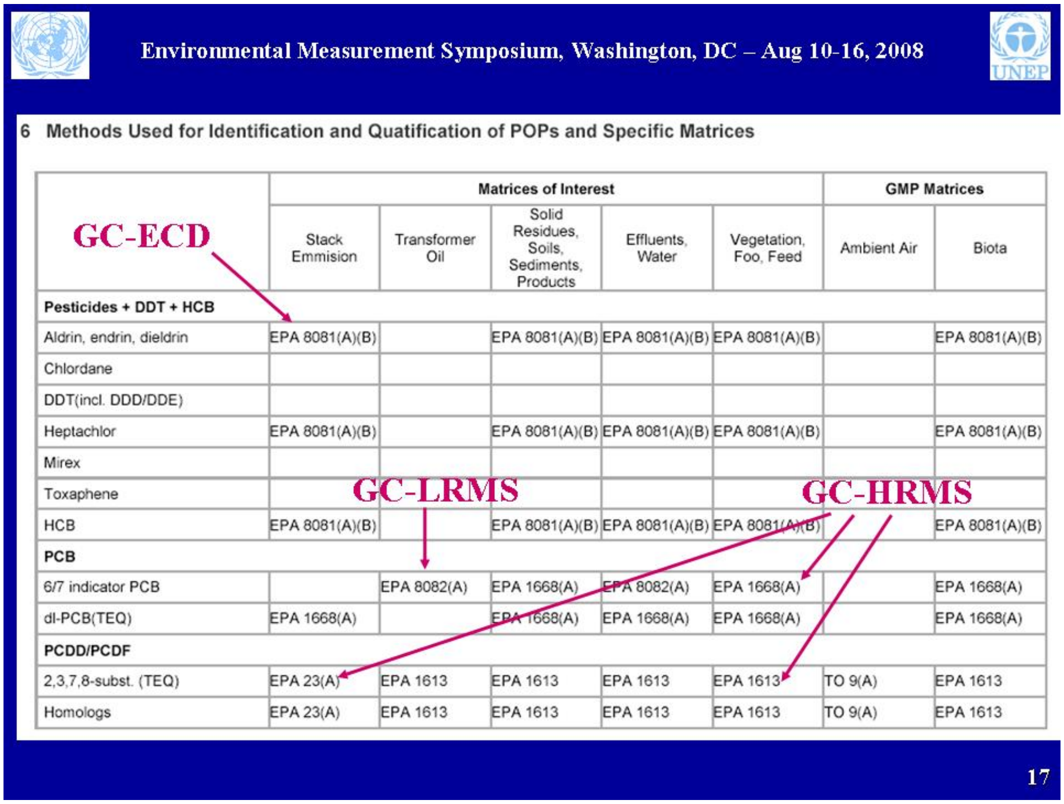
Frequently		Less Frequently	
POP	**No of Labs**	**POP**	**No of Labs**
DDT	155	PCDD/PCDF	56
Heptachlor	147	Toxaphene	72
PCB	145	dl-PCB	74
HCB	145	Mirex	77
-drins	127		
Chlordane	118		

Overview on Matrices Analyzed

Frequently Analyzed		Less Frequently Analyzed	
Matrix	No of Labs	Matrix	No of Labs
Water/effluents	161	Mothers' milk/human blood	53
Soil/sediment/residue	152	Stack emissions	53
Food/feed	124	Ambient air	59
Transformer oils	95	Chemicals/products	70
Vegetation	93		

Core matrices of the first effectiveness evaluation

Basis = 202 labs, status June 2008



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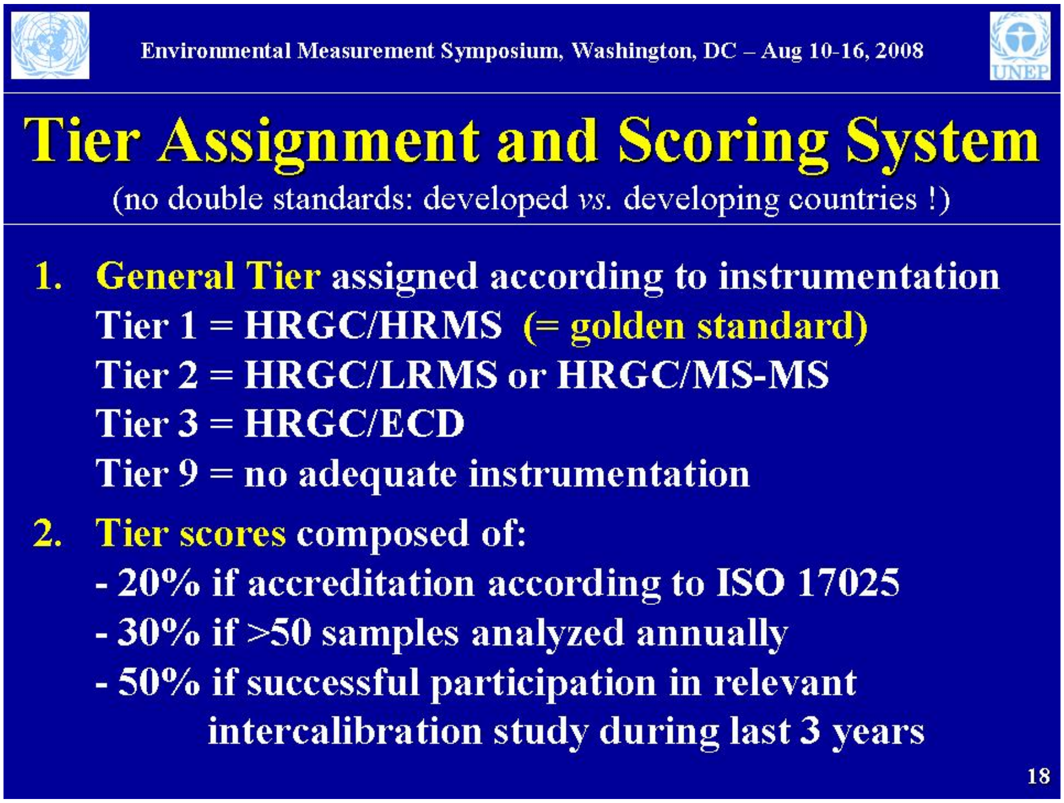
6 Methods Used for Identification and Quatification of POPs and Specific Matrices

	Matrices of Interest					GMP Matrices	
	Stack Emmision	Transformer Oil	Solid Residues, Soils, Sediments, Products	Effluents, Water	Vegetation, Foo, Feed	Ambient Air	Biota
Pesticides + DDT + HCB							
Aldrin, endrin, dieldrin	EPA 8081(A)(B)		EPA 8081(A)(B)	EPA 8081(A)(B)	EPA 8081(A)(B)		EPA 8081(A)(B)
Chlordane							
DDT(incl. DDD/DDE)							
Heptachlor	EPA 8081(A)(B)		EPA 8081(A)(B)	EPA 8081(A)(B)	EPA 8081(A)(B)		EPA 8081(A)(B)
Mirex							
Toxaphene							
HCB	EPA 8081(A)(B)		EPA 8081(A)(B)	EPA 8081(A)(B)	EPA 8081(A)(B)		EPA 8081(A)(B)
PCB							
6/7 indicator PCB		EPA 8082(A)	EPA 1668(A)	EPA 8082(A)	EPA 1668(A)		EPA 1668(A)
dl-PCB(TEQ)	EPA 1668(A)		EPA 1668(A)	EPA 1668(A)	EPA 1668(A)		EPA 1668(A)
PCDD/PCDF							
2,3,7,8-subst. (TEQ)	EPA 23(A)	EPA 1613	EPA 1613	EPA 1613	EPA 1613	TO 9(A)	EPA 1613
Homologs	EPA 23(A)	EPA 1613	EPA 1613	EPA 1613	EPA 1613	TO 9(A)	EPA 1613



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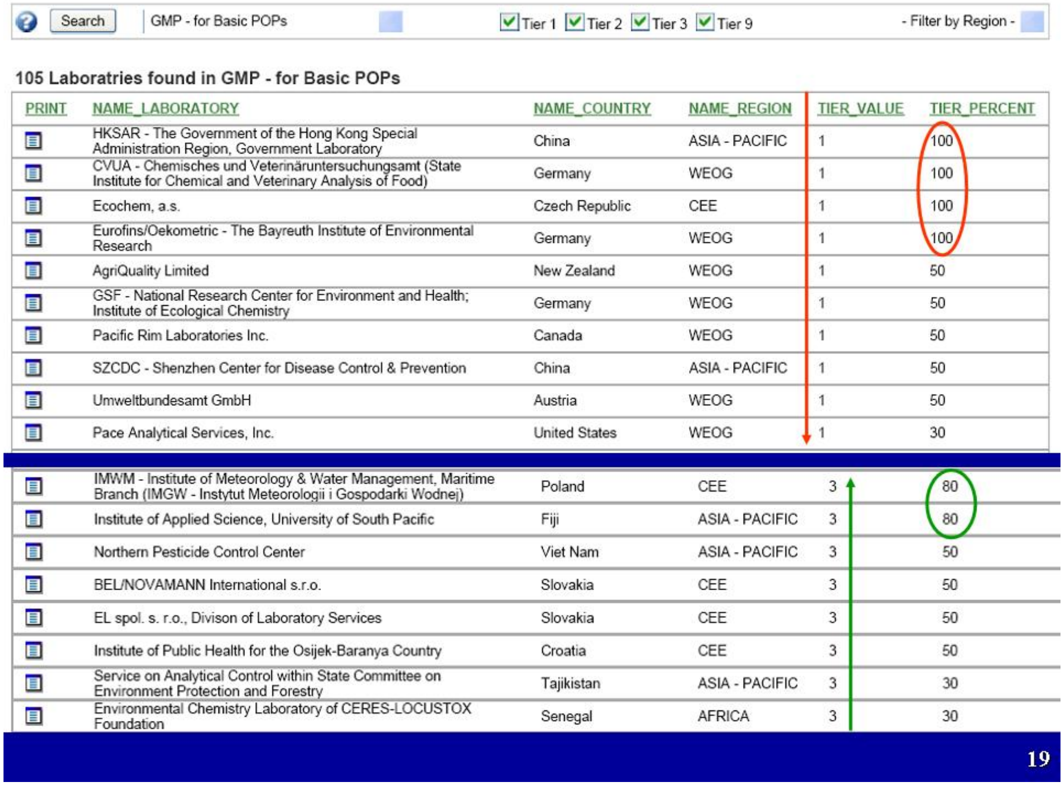
Tier Assignment and Scoring System

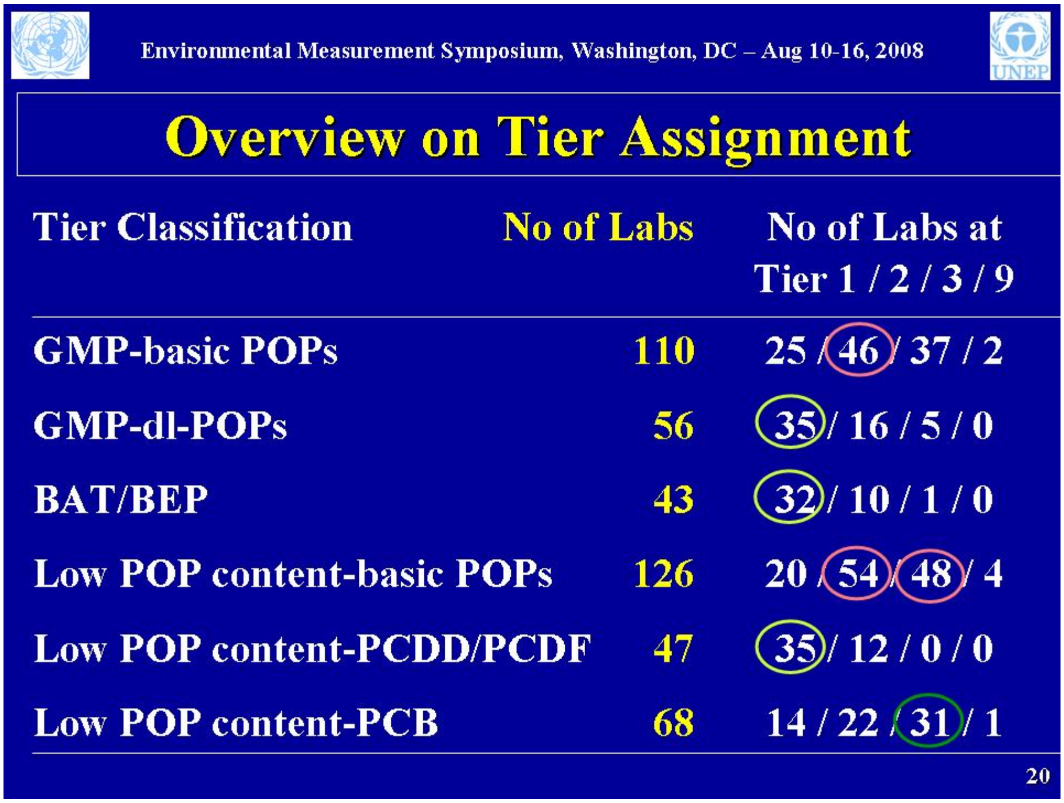
(no double standards: developed *vs.* developing countries !)

1. **General Tier assigned according to instrumentation**
   Tier 1 = HRGC/HRMS (= golden standard)
   Tier 2 = HRGC/LRMS or HRGC/MS-MS
   Tier 3 = HRGC/ECD
   Tier 9 = no adequate instrumentation
2. **Tier scores composed of:**
   - 20% if accreditation according to ISO 17025
   - 30% if >50 samples analyzed annually
   - 50% if successful participation in relevant intercalibration study during last 3 years



105 Laboratries found in GMP - for Basic POPs

PRINT	NAME_LABORATORY	NAME_COUNTRY	NAME_REGION	TIER_VALUE	TIER_PERCENT
▤	HKSAR - The Government of the Hong Kong Special Administration Region, Government Laboratory	China	ASIA - PACIFIC	1	100
▤	CVUA - Chemisches und Veterinäruntersuchungsamt (State Institute for Chemical and Veterinary Analysis of Food)	Germany	WEOG	1	100
▤	Ecochem, a.s.	Czech Republic	CEE	1	100
▤	Eurofins/Oekometric - The Bayreuth Institute of Environmental Research	Germany	WEOG	1	100
▤	AgriQuality Limited	New Zealand	WEOG	1	50
▤	GSF - National Research Center for Environment and Health; Institute of Ecological Chemistry	Germany	WEOG	1	50
▤	Pacific Rim Laboratories Inc.	Canada	WEOG	1	50
▤	SZCDC - Shenzhen Center for Disease Control & Prevention	China	ASIA - PACIFIC	1	50
▤	Umweltbundesamt GmbH	Austria	WEOG	1	50
▤	Pace Analytical Services, Inc.	United States	WEOG	1	30
▤	IMWM - Institute of Meteorology & Water Management, Maritime Branch (IMGW - Instytut Meteorologii i Gospodarki Wodnej)	Poland	CEE	3	80
▤	Institute of Applied Science, University of South Pacific	Fiji	ASIA - PACIFIC	3	80
▤	Northern Pesticide Control Center	Viet Nam	ASIA - PACIFIC	3	50
▤	BEL/NOVAMANN International s.r.o.	Slovakia	CEE	3	50
▤	EL spol. s. r.o., Divison of Laboratory Services	Slovakia	CEE	3	50
▤	Institute of Public Health for the Osijek-Baranya Country	Croatia	CEE	3	50
▤	Service on Analytical Control within State Committee on Environment Protection and Forestry	Tajikistan	ASIA - PACIFIC	3	30
▤	Environmental Chemistry Laboratory of CERES-LOCUSTOX Foundation	Senegal	AFRICA	3	30



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Overview on Tier Assignment

Tier Classification	No of Labs	No of Labs at Tier 1 / 2 / 3 / 9
GMP-basic POPs	110	25 / 46 / 37 / 2
GMP-dl-POPs	56	35 / 16 / 5 / 0
BAT/BEP	43	32 / 10 / 1 / 0
Low POP content-basic POPs	126	20 / 54 / 48 / 4
Low POP content-PCDD/PCDF	47	35 / 12 / 0 / 0
Low POP content-PCB	68	14 / 22 / 31 / 1

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Training Program with Pilot Labs

1. Inspection tour at developing country laboratory (7 countries, 9 labs; by UNEP and back-up expert laboratory);
2. Identification of needs for spares/consumables and standards;
3. Workshop with all participating laboratories;
4. Procurement of spares/consumables/standards and shipment;
5. Hands-on training in developing country laboratory (1 wk.);
6. Exchange of national samples (mirror analysis in expert lab);
7. International intercalibration study on biotic or abiotic matrix;
8. Final workshop with all participating laboratories; evaluation of results, lessons learned; final training at expert laboratory.

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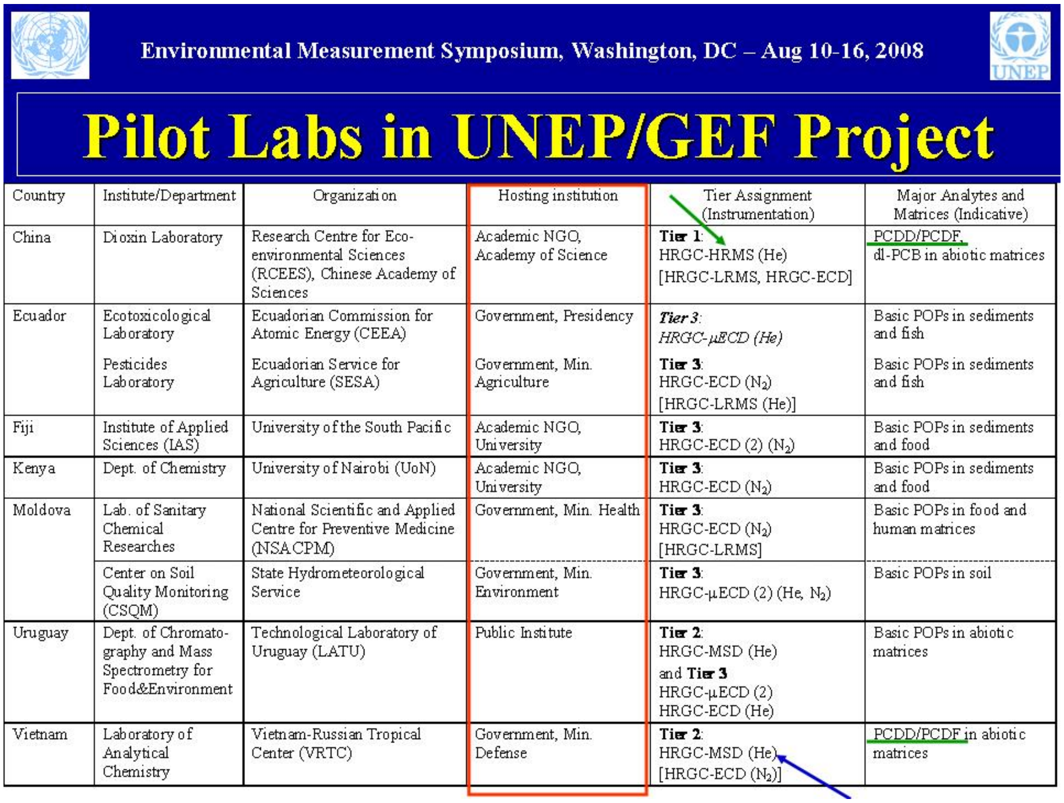

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Pilot Labs in UNEP/GEF Project

Country	Institute/Department	Organization	Hosting institution	Tier Assignment (Instrumentation)	Major Analytes and Matrices (Indicative)
China	Dioxin Laboratory	Research Centre for Eco-environmental Sciences (RCEES), Chinese Academy of Sciences	Academic NGO, Academy of Science	**Tier 1**: HRGC-HRMS (He) [HRGC-LRMS, HRGC-ECD]	PCDD/PCDF, dl-PCB in abiotic matrices
Ecuador	Ecotoxicological Laboratory	Ecuadorian Commission for Atomic Energy (CEEA)	Government, Presidency	***Tier 3***: *HRGC-μECD (He)*	Basic POPs in sediments and fish
	Pesticides Laboratory	Ecuadorian Service for Agriculture (SESA)	Government, Min. Agriculture	**Tier 3**: HRGC-ECD (N_2) [HRGC-LRMS (He)]	Basic POPs in sediments and fish
Fiji	Institute of Applied Sciences (IAS)	University of the South Pacific	Academic NGO, University	**Tier 3**: HRGC-ECD (2) (N_2)	Basic POPs in sediments and food
Kenya	Dept. of Chemistry	University of Nairobi (UoN)	Academic NGO, University	**Tier 3**: HRGC-ECD (N_2)	Basic POPs in sediments and food
Moldova	Lab. of Sanitary Chemical Researches	National Scientific and Applied Centre for Preventive Medicine (NSACPM)	Government, Min. Health	**Tier 3**: HRGC-ECD (N_2) [HRGC-LRMS]	Basic POPs in food and human matrices
	Center on Soil Quality Monitoring (CSQM)	State Hydrometeorological Service	Government, Min. Environment	**Tier 3**: HRGC-μECD (2) (He, N_2)	Basic POPs in soil
Uruguay	Dept. of Chromatography and Mass Spectrometry for Food&Environment	Technological Laboratory of Uruguay (LATU)	Public Institute	**Tier 2**: HRGC-MSD (He) and **Tier 3** HRGC-μECD (2) HRGC-ECD (He)	Basic POPs in abiotic matrices
Vietnam	Laboratory of Analytical Chemistry	Vietnam-Russian Tropical Center (VRTC)	Government, Min. Defense	**Tier 2**: HRGC-MSD (He) [HRGC-ECD (N_2)]	PCDD/PCDF in abiotic matrices

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Observation from Training Sessions

1. **Fiji (ECD lab):**
   - basic training on POPs pesticide analysis
   - GC training
   - Multi-column clean-up introduced
   - Internal standard method introduced
   - Basic QA/QC;
2. **Vietnam (LRMS lab; dioxin-like compounds):**
   - Comparatively well-trained staff
   - EPA-based methods used
   - QA/QC in place
   - Implementation of analysis for dioxin-like (WHO-TEF) PCB
   - Focus on external QA/QC for PCDD/PCDF
   - Instrumentation is limitation.

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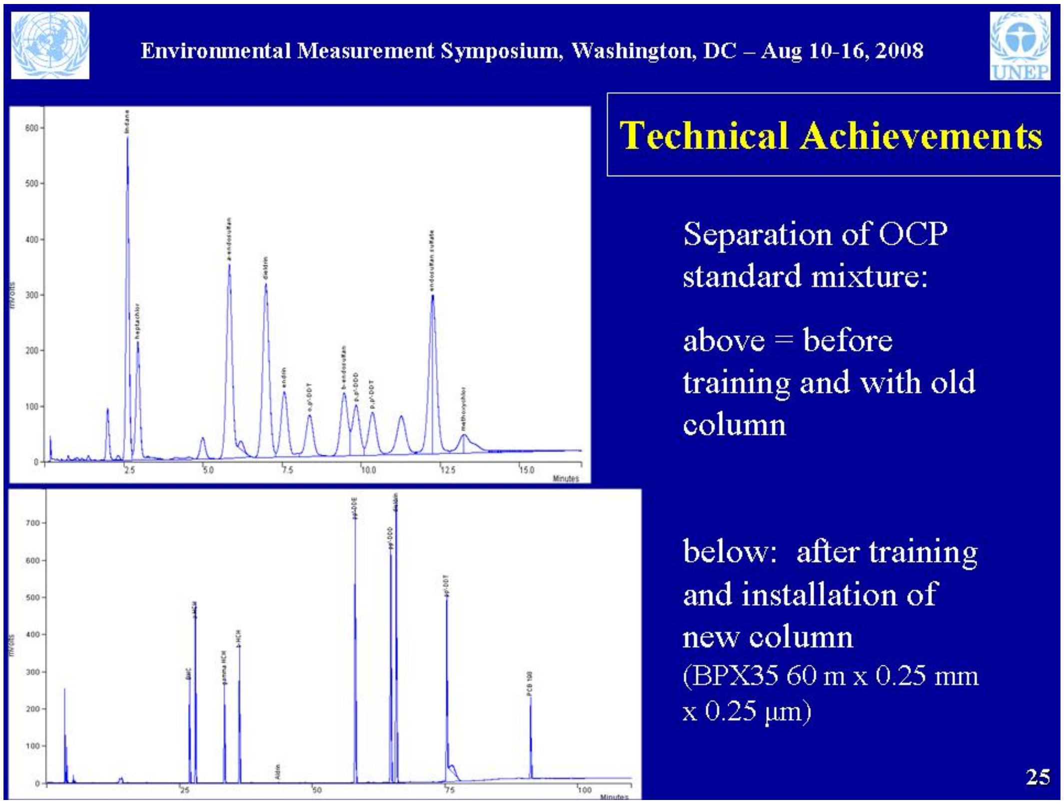
Results of Training Sessions

- Useful contacts established between the local staff and the trainers, and among local staff members
- The understanding of QA/QC aspects and analytical methods has been improved by the training sessions; especially on the following issues:
  - Planning and time spent on QA/QC;
  - Use of autosamplers;
  - Use of better glassware (*e.g.*, coolers, columns) ;
  - Better GC columns and carrier gas (He);
  - Use of internal standards, LRMs, CRMs;
  - Multi-level calibration curve;
  - For standards, use of w/w basis instead of w/v;
  - Co-factor analysis (fat, TOC).



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Technical Achievements

Separation of OCP standard mixture:

above = before training and with old column

below: after training and installation of new column (BPX35 60 m x 0.25 mm x 0.25 µm)

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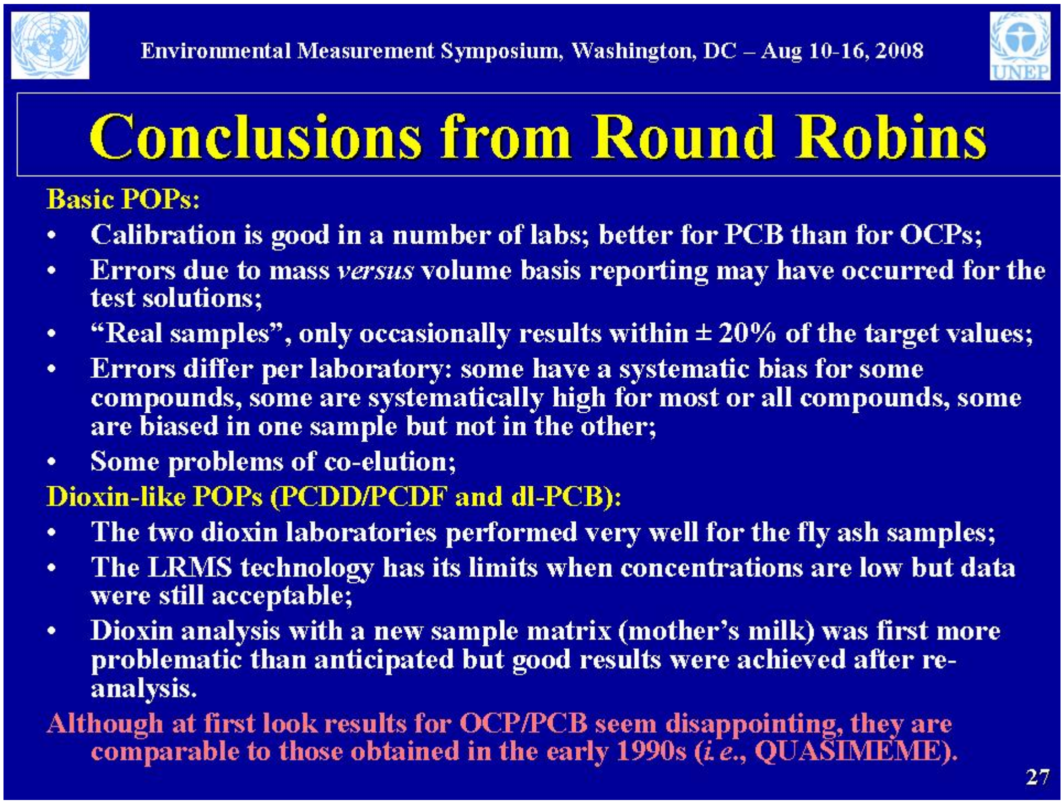
Two Intercalibration Studies

1. **Basic POPs in abiotic and biotic matrices:**
   Institute for Environmental Studies of VU University Amsterdam (Dr. Jacob de Boer);
   7 Laboratories from 5 countries covering 4 UN regions;
   (a) Analyses of national samples and (b) interlaboratory study with a test solution, a sediment sample, and a herring tissue sample.
2. **Dioxin-like POPs in flyash/soil and mothers' milk:**
   MTM Centre of Örebro University (Dr. Bert van Bavel, Dr. Gunilla Lindström):
   Two dioxin laboratories, China (HRMS) and Vietnam (LRMS) participated.



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Conclusions from Round Robins

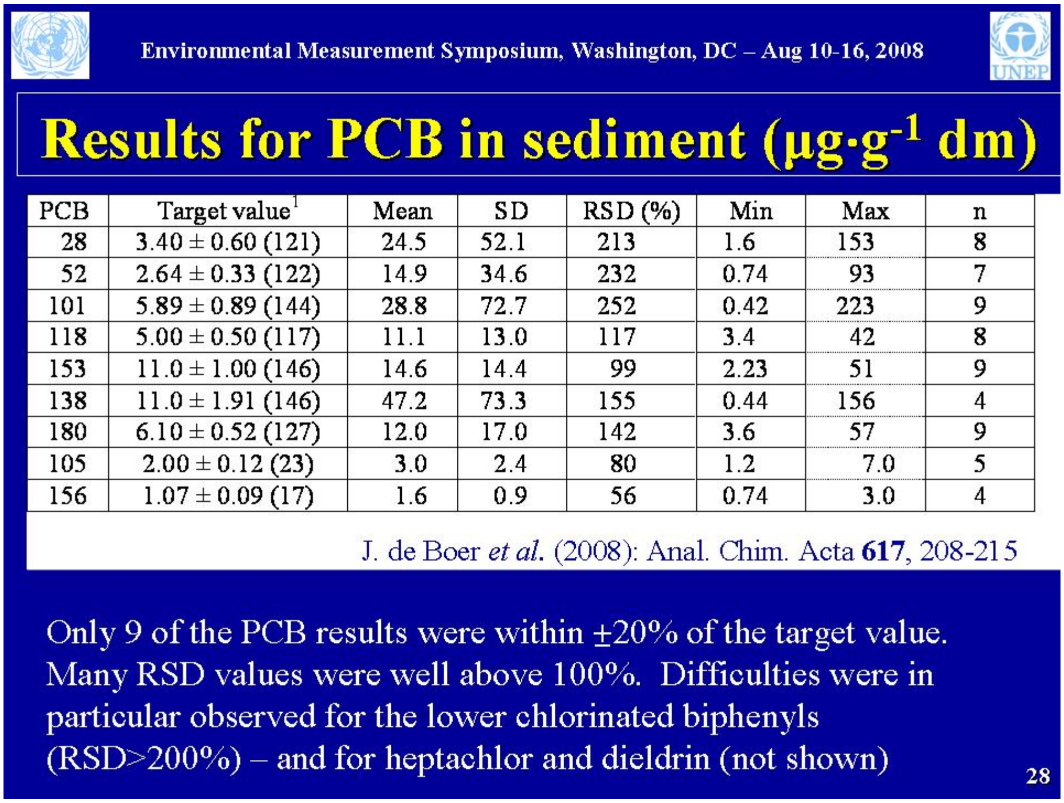
Basic POPs:

- Calibration is good in a number of labs; better for PCB than for OCPs;
- Errors due to mass *versus* volume basis reporting may have occurred for the test solutions;
- "Real samples", only occasionally results within ± 20% of the target values;
- Errors differ per laboratory: some have a systematic bias for some compounds, some are systematically high for most or all compounds, some are biased in one sample but not in the other;
- Some problems of co-elution;

Dioxin-like POPs (PCDD/PCDF and dl-PCB):

- The two dioxin laboratories performed very well for the fly ash samples;
- The LRMS technology has its limits when concentrations are low but data were still acceptable;
- Dioxin analysis with a new sample matrix (mother's milk) was first more problematic than anticipated but good results were achieved after re-analysis.

Although at first look results for OCP/PCB seem disappointing, they are comparable to those obtained in the early 1990s (*i.e.*, QUASIMEME).



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Results for PCB in sediment ($\mu g \cdot g^{-1}$ dm)

PCB	Target value[1]	Mean	SD	RSD (%)	Min	Max	n
28	3.40 ± 0.60 (121)	24.5	52.1	213	1.6	153	8
52	2.64 ± 0.33 (122)	14.9	34.6	232	0.74	93	7
101	5.89 ± 0.89 (144)	28.8	72.7	252	0.42	223	9
118	5.00 ± 0.50 (117)	11.1	13.0	117	3.4	42	8
153	11.0 ± 1.00 (146)	14.6	14.4	99	2.23	51	9
138	11.0 ± 1.91 (146)	47.2	73.3	155	0.44	156	4
180	6.10 ± 0.52 (127)	12.0	17.0	142	3.6	57	9
105	2.00 ± 0.12 (23)	3.0	2.4	80	1.2	7.0	5
156	1.07 ± 0.09 (17)	1.6	0.9	56	0.74	3.0	4

J. de Boer *et al.* (2008): Anal. Chim. Acta **617**, 208-215

Only 9 of the PCB results were within ±20% of the target value. Many RSD values were well above 100%. Difficulties were in particular observed for the lower chlorinated biphenyls (RSD>200%) – and for heptachlor and dieldrin (not shown)

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Results for Herring (µg kg^{-1} wwt)

PCB/OCP	Target value[a]	Mean	St. dev.	Rel. St. Dev. (%)	Min.	Max.	n
PCB 28	0.41 ± 0.04 (6)	2.24	1.53	68	0.34	4.25	7
PCB 52	1.00 ± 0.04 (8)	1.93	1.27	66	0.72	4.41	7
PCB 101	2.12 ± 0.06 (8)	4.21	3.19	76	1.70	10.99	8
PCB 118	1.78 ± 0.07 (9)	4.45	4.23	95	0.17	12.64	8
PCB 153	4.62 ± 0.10 (11)	7.03	4.34	62	2.6	14.60	8
PCB 138	2.97 ± 0.11 (6)	8.24	4.09	50	3.37	13.37	4
PCB 180	0.795 ± 0.027 (7)	1.66	0.97	58	0.41	3.11	6
PCB 105	0.63 ± 0.06 (6)	1.57	0.57	38	0.99	2.13	3
PCB 156	0.19 ± 0.09 (6)	0.84	0.58	69	0.24	1.40	3
p,p'-DDE	5.9	4.50	2.12	47	0.58	6.40	6
p,p'-DDD	<0.1	1.24	1.21	98	0.09	3.16	5
o,p' DDT	<0.1	1.34					1
p,p'-DDT	<0.1	4.00	3.53	88	0.70	9.96	5
HCB	1.5	2.96	3.33	83	0.66	9.63	6
Heptachlor	<0.1	1.12	1.44	129	0.14	2.78	3
Aldrin	<0.1	0.48					1
Dieldrin	5.6	2.13	1.56	73	0.33	4.55	6
Endrin	1.2	1.81	1.97	109	0.42	3.20	2

J. de Boer *et al.* (2008): Anal. Chim. Acta **617**, 208-215

Results for herring were better than for sediment (most RSD<100%);
indicating that sensitivity was not the major problem (sediment concentrations were higher).

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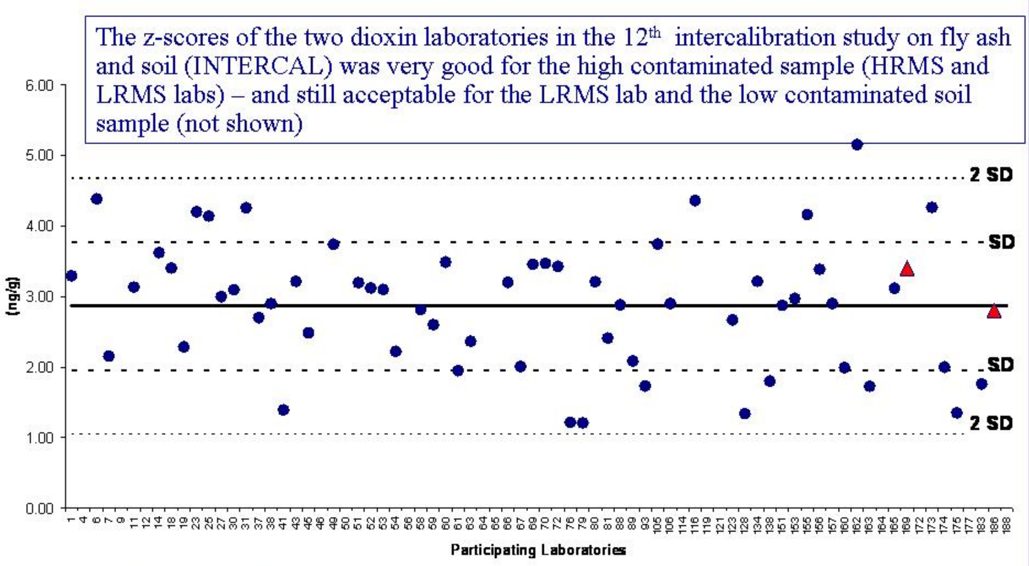
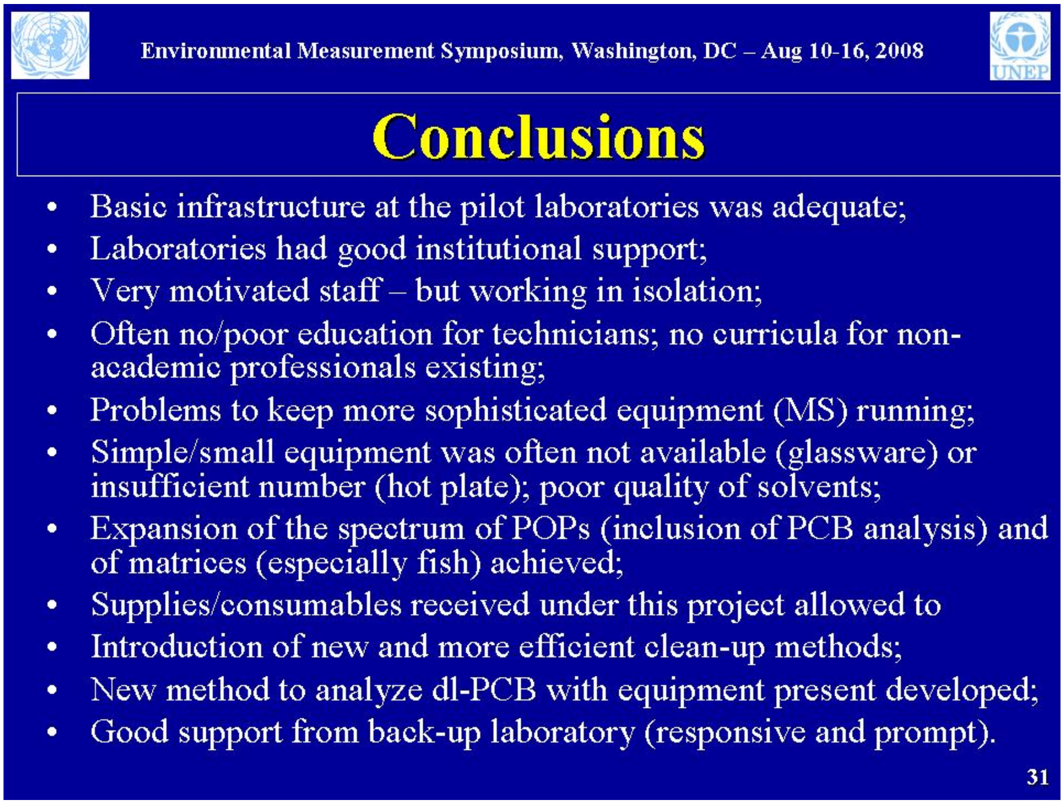
TEQ Ash A (RSD 32%, n =62)



The z-scores of the two dioxin laboratories in the 12th intercalibration study on fly ash and soil (INTERCAL) was very good for the high contaminated sample (HRMS and LRMS labs) – and still acceptable for the LRMS lab and the low contaminated soil sample (not shown)

UNEP pilot labs ▲

Courtesy: B. van Bavel, 12th INTERCAL



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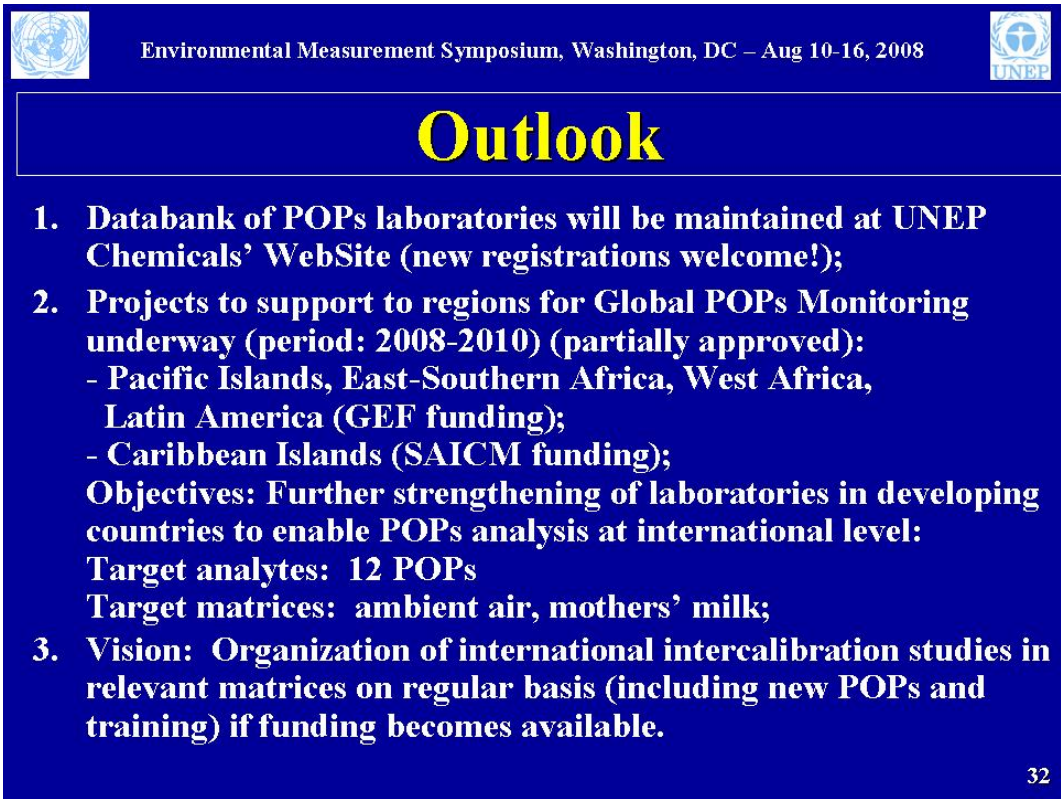
Conclusions

- Basic infrastructure at the pilot laboratories was adequate;
- Laboratories had good institutional support;
- Very motivated staff – but working in isolation;
- Often no/poor education for technicians; no curricula for non-academic professionals existing;
- Problems to keep more sophisticated equipment (MS) running;
- Simple/small equipment was often not available (glassware) or insufficient number (hot plate); poor quality of solvents;
- Expansion of the spectrum of POPs (inclusion of PCB analysis) and of matrices (especially fish) achieved;
- Supplies/consumables received under this project allowed to
- Introduction of new and more efficient clean-up methods;
- New method to analyze dl-PCB with equipment present developed;
- Good support from back-up laboratory (responsive and prompt).



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Outlook

1. **Databank of POPs laboratories will be maintained at UNEP Chemicals' WebSite (new registrations welcome!);**
2. **Projects to support to regions for Global POPs Monitoring underway (period: 2008-2010) (partially approved):**
   - Pacific Islands, East-Southern Africa, West Africa, Latin America (GEF funding);
   - Caribbean Islands (SAICM funding);
   Objectives: Further strengthening of laboratories in developing countries to enable POPs analysis at international level:
   Target analytes: 12 POPs
   Target matrices: ambient air, mothers' milk;
3. **Vision: Organization of international intercalibration studies in relevant matrices on regular basis (including new POPs and training) if funding becomes available.**

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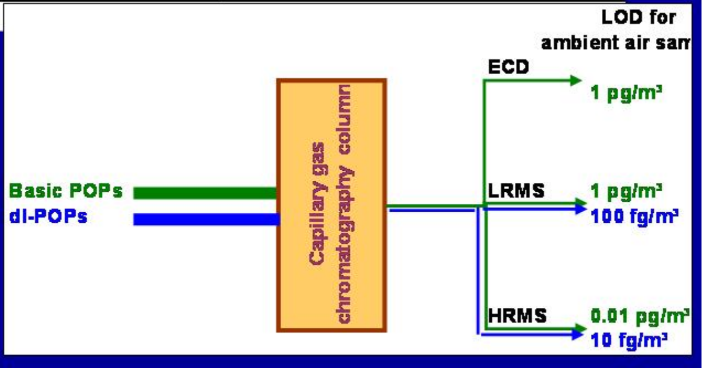
Type	Basic Elements	Sampling Time, Volume, Infrastructure
High-volume active samplers	Glass-fiber/quartz filters combined with PUF plugs; XAD or carbon filters/disks added	Time: 1-2 days; up to one week; Volume: 600-1,500 m³/d; High demands on infrastructure
Passive samplers	PUF disks, XAD-based resin, LDPE with triolein; in stainless steel chambers	Time: several months to year; Volume: 0.5-4 m³/d; Low demands on infrastructure
~~Bulk deposition samplers~~	~~Frisbee-type (plastics), Bergerhoff (glass)~~	~~Time: 14-30 days~~ ~~Surface-based measurements;~~ ~~Low demands on infrastructure~~

Ambient Air Monitoring: Active and Passive Samplers



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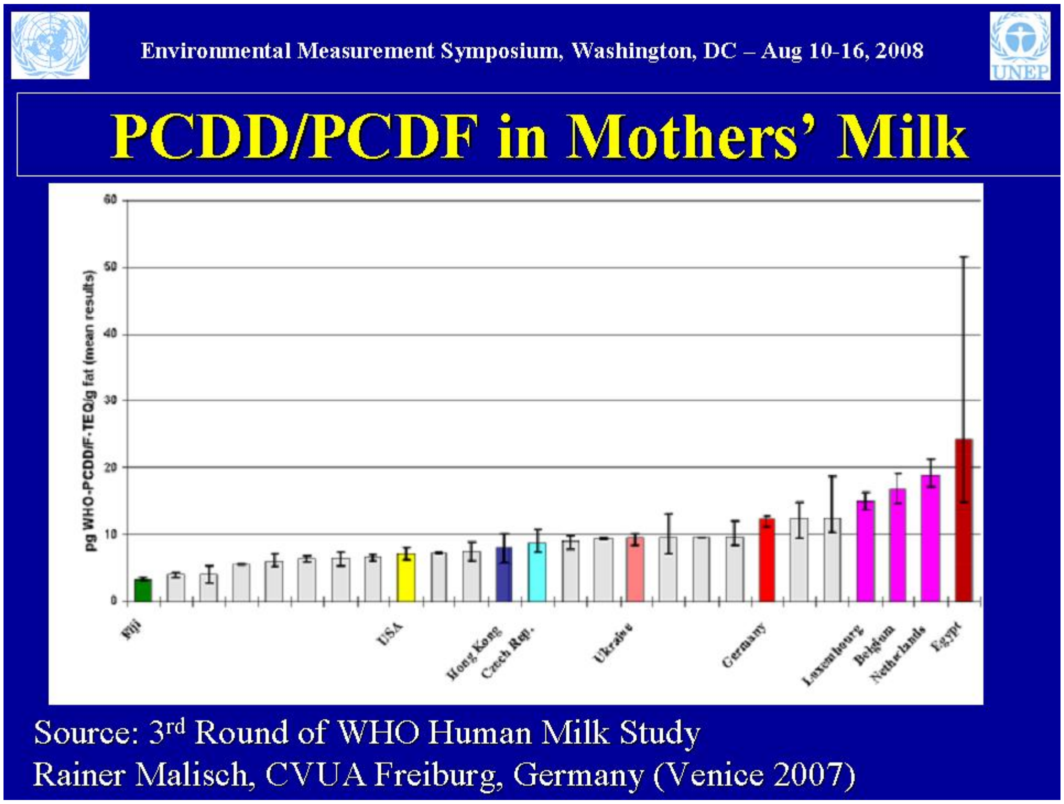
High Volume Air Sampler at Maun, Botswana (courtesy: Frank Wania, U Toronto)

PUF disk sampler operated under GAPS Network at Jeju Island in South Korea (courtesy: Tom Harner, Environment Canada)



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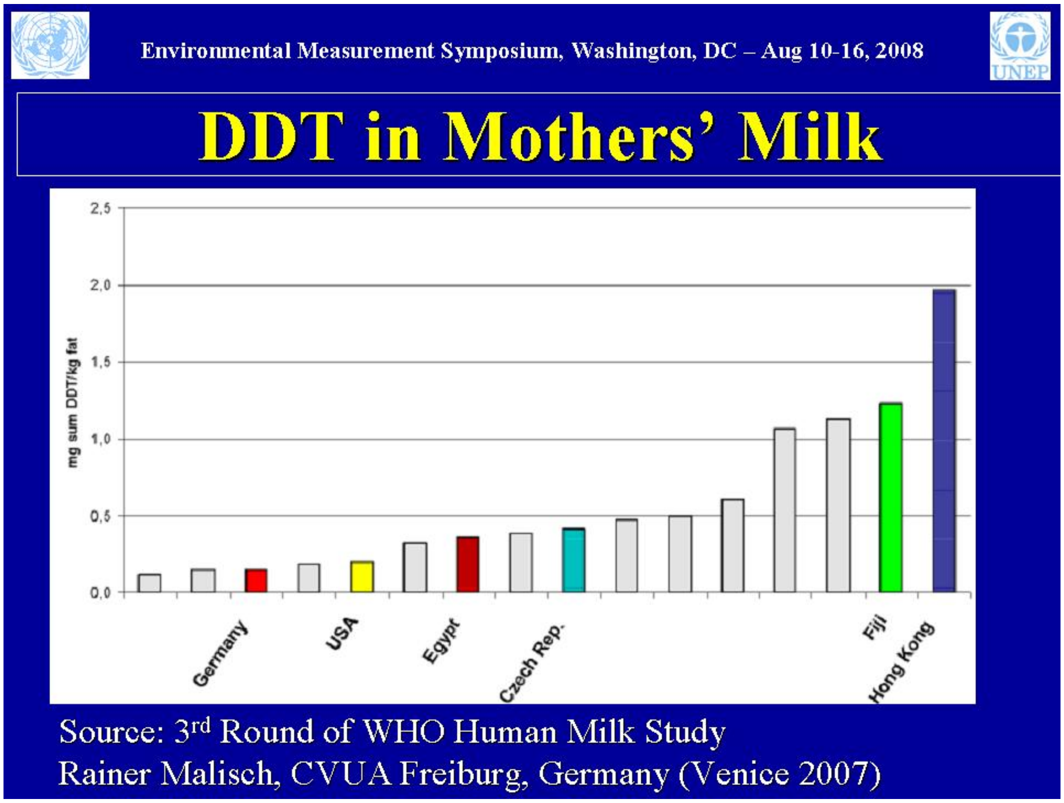
PCDD/PCDF in Mothers' Milk

Source: 3rd Round of WHO Human Milk Study
Rainer Malisch, CVUA Freiburg, Germany (Venice 2007)



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DDT in Mothers' Milk

Source: 3rd Round of WHO Human Milk Study
Rainer Malisch, CVUA Freiburg, Germany (Venice 2007)

Acknowledgment

- The POPs Laboratory Project was financed by the Global Environment Facility (GEF) with co-financing by governments of Canada, Germany, and Japan.
- Thanks to Osmany Perreira and Douglas Rofes for programming of the databank, Rogerio Fenner for input of data; all laboratories for submitting information;
- Private sector for support with spares/consumables, and analytical standards;
- Drs. Gunilla Lindström, Bert van Bavel (MTM Örebro), and Jacob de Boer (IVMVU Amsterdam) and their staff for technical backstopping.

Thank you.

2008 NEMC Proceedings

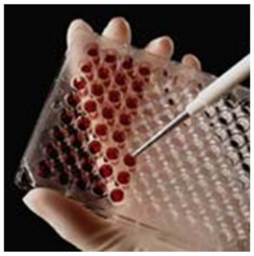
EPA PANEL

EPA’s Directions in Environmental Measurement

Dr. Pai-Yei Whung
Chief Scientist
Office of the Science Advisor

Environmental Measurement Symposium
August 13, 2008

Office of the Science Advisor (OSA)



- Science Policy Council
- Risk Assessment Forum
- Human Studies Review Board
- Human Subjects Research Review
- Environmental Technology

- Forum on Environmental Measurements
- Environmental Laboratory Advisory Board
- Council for Regulatory Environmental Modeling
- Global Earth Observations System of Systems

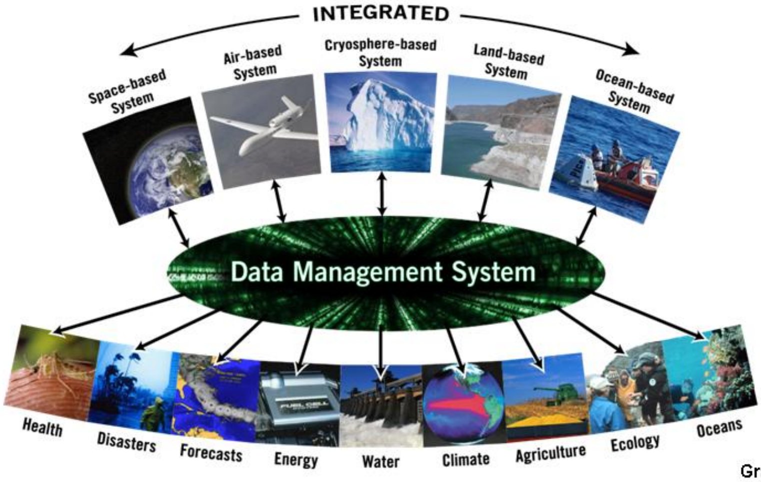
Measurement Science

Environmental Technology
ETOP
Opportunities Portal

RISK
ASSESSMENT
FORUM

CREM

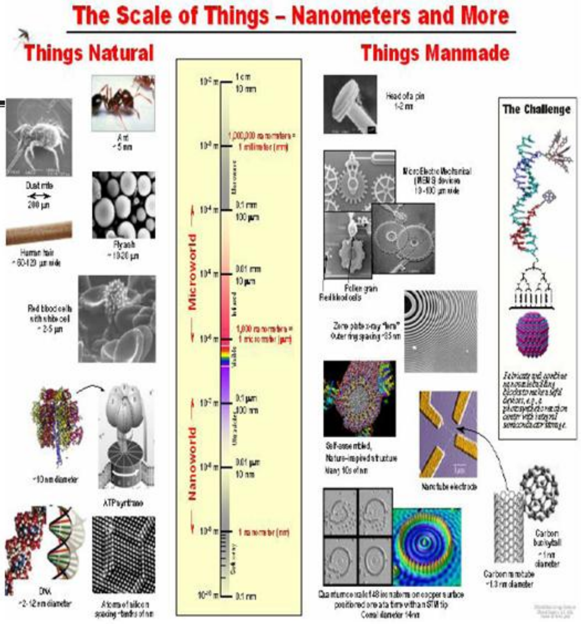
Global Earth Observation System of Systems



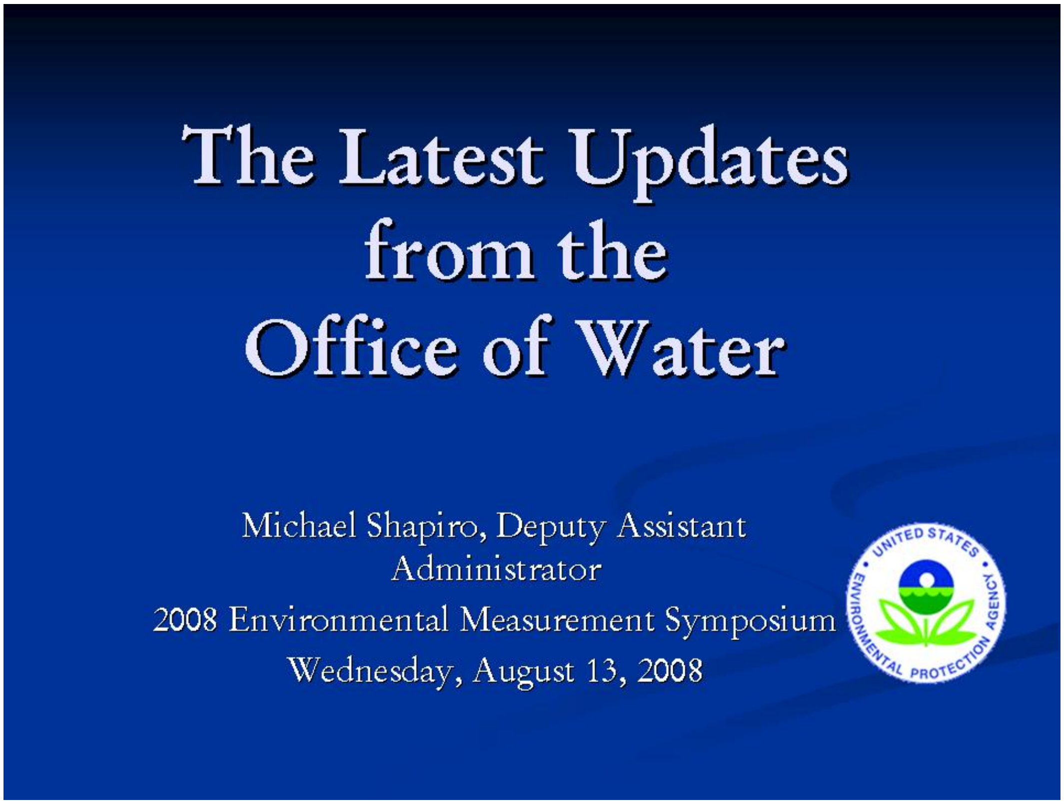
Graphic: NOAA

Emerging Needs

- Climate Change
- Biofuels
- Nanotechnology



Environmental Landscape

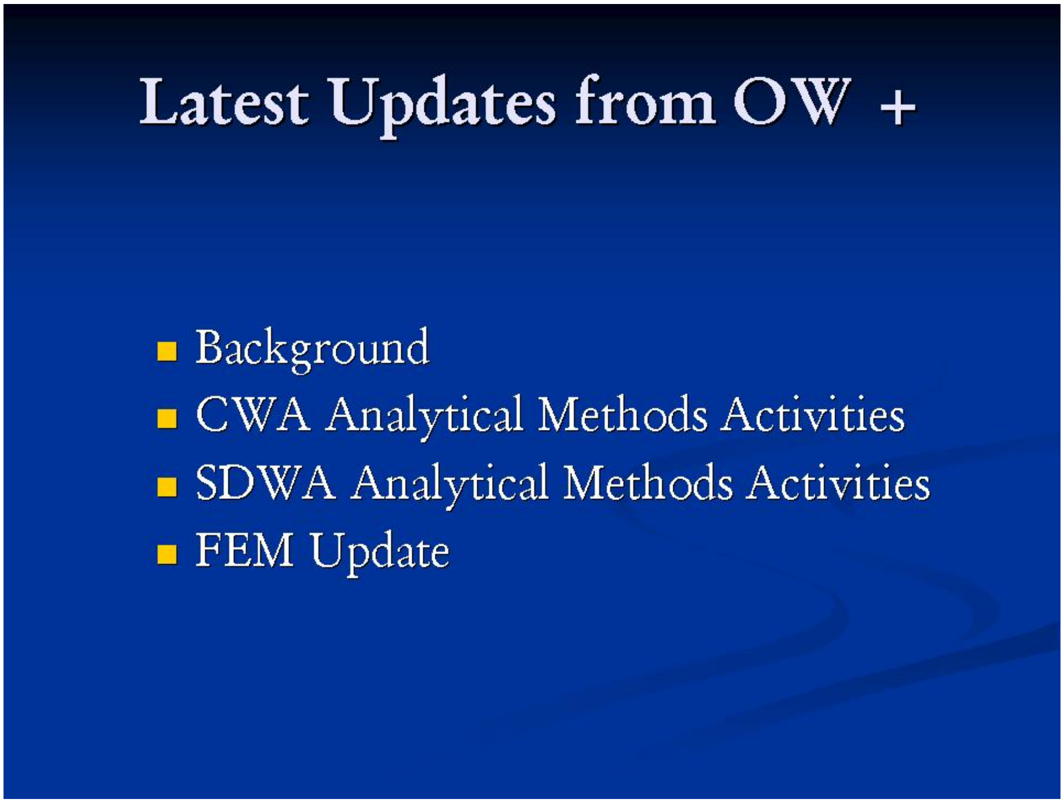


The Latest Updates from the Office of Water

Michael Shapiro, Deputy Assistant Administrator

2008 Environmental Measurement Symposium

Wednesday, August 13, 2008



Latest Updates from OW +

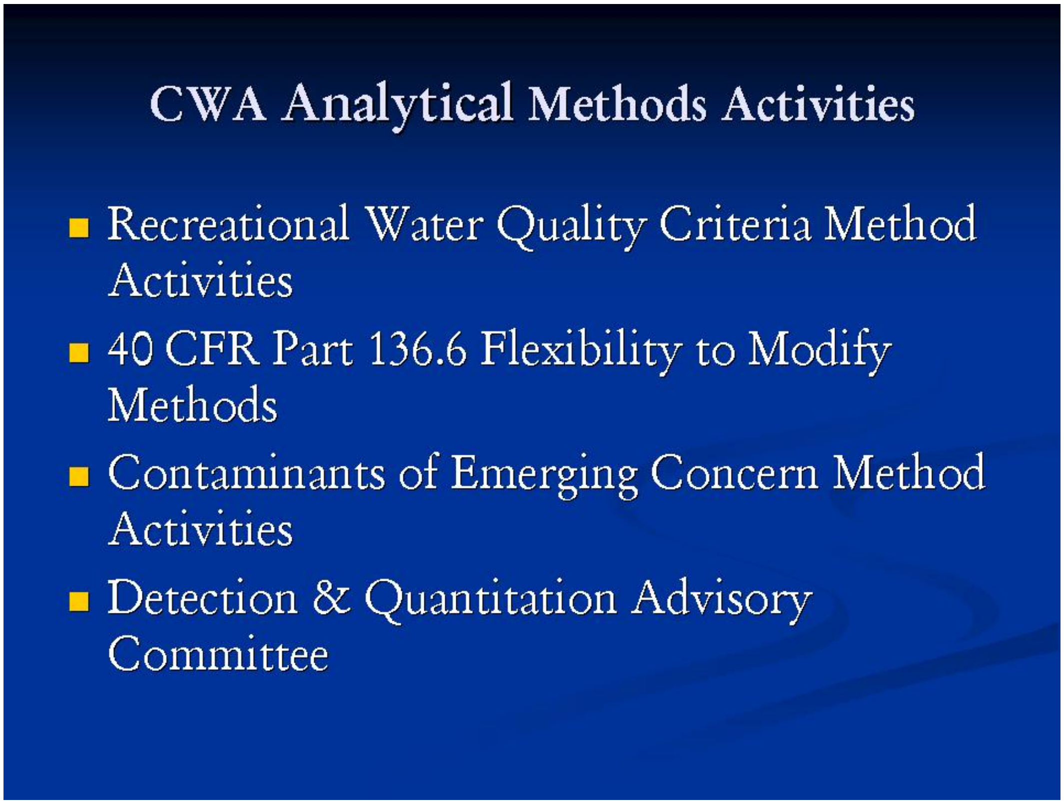
- Background
- CWA Analytical Methods Activities
- SDWA Analytical Methods Activities
- FEM Update

Background: Drivers for Office of Water Methods Work

- Support compliance monitoring
  - Permit conditions, NPDES/CWA
  - Water Quality Standards, CWA
  - Regulated drinking water contaminants, SDWA
- Support occurrence data gathering for assessment, problem identification, regulatory development
  - UCMR data supports regulatory determinations for chemical and microbial contaminants, SDWA
  - Effluent Guideline development, CWA
  - Assess surface water conditions, CWA

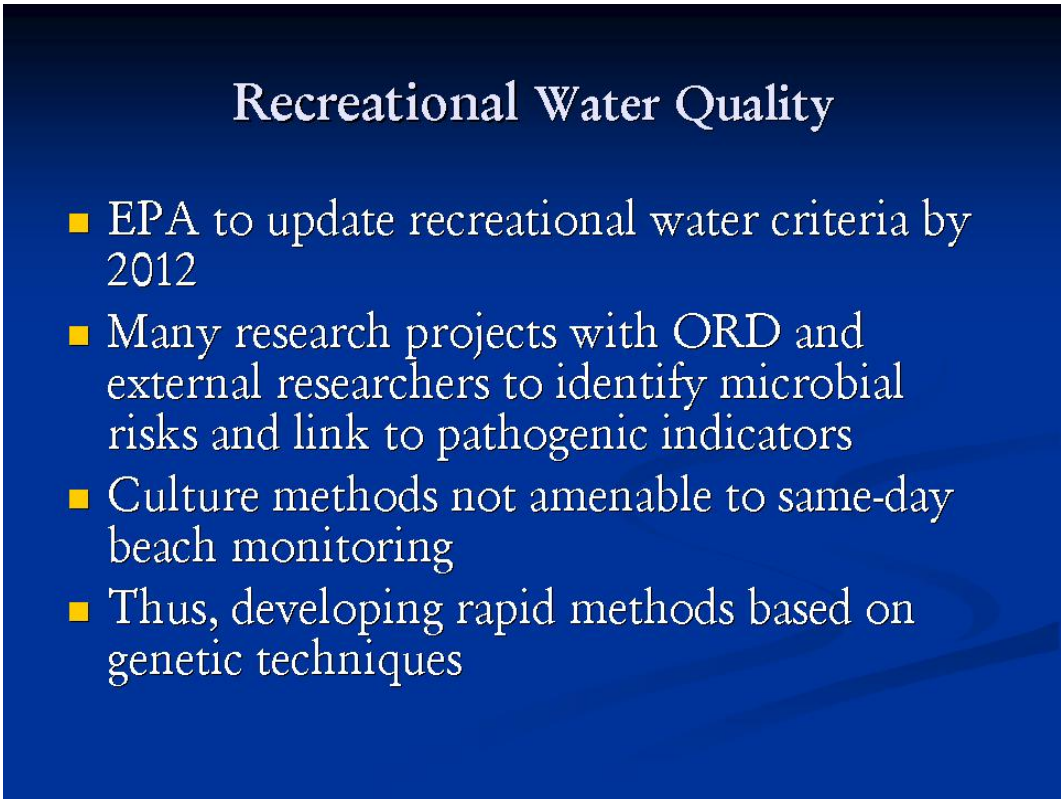
Background: Office of Water Method Development Goals

- Keep pace with technologies for the analysis of both currently regulated and emerging contaminants.
- Maximize flexibility in analytical methods while maintaining data quality data needed to make regulatory decisions and to monitor compliance
- Consider cost of analysis and use technologies familiar/available to laboratories whenever practical.



CWA Analytical Methods Activities

- Recreational Water Quality Criteria Method Activities
- 40 CFR Part 136.6 Flexibility to Modify Methods
- Contaminants of Emerging Concern Method Activities
- Detection & Quantitation Advisory Committee

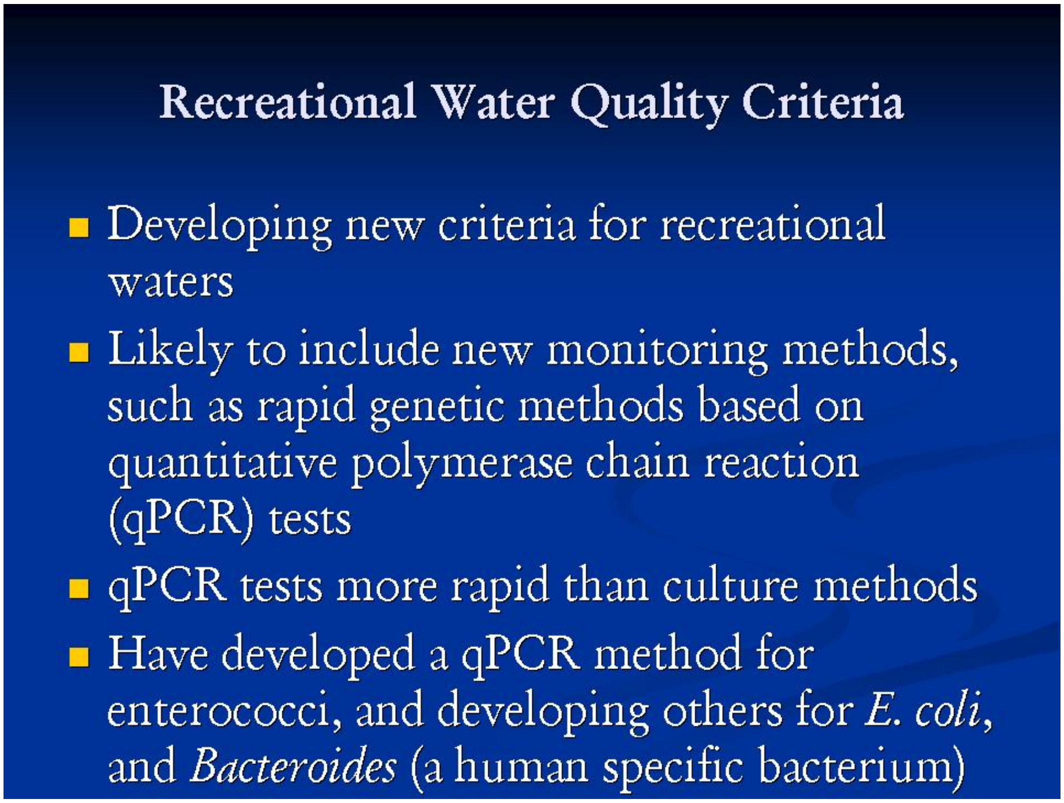


Recreational Water Quality

- EPA to update recreational water criteria by 2012
- Many research projects with ORD and external researchers to identify microbial risks and link to pathogenic indicators
- Culture methods not amenable to same-day beach monitoring
- Thus, developing rapid methods based on genetic techniques

Recreational Water Quality

- Not practical to look for all pathogens (too many)
- We currently use bacterial indicators of fecal contamination
- In recreational waters, these are enterococci and *Escherichia coli* (*E. coli*)
- EPA epidemiological studies positively correlated these indicator organisms with human fecal contamination in beach waters



Recreational Water Quality Criteria

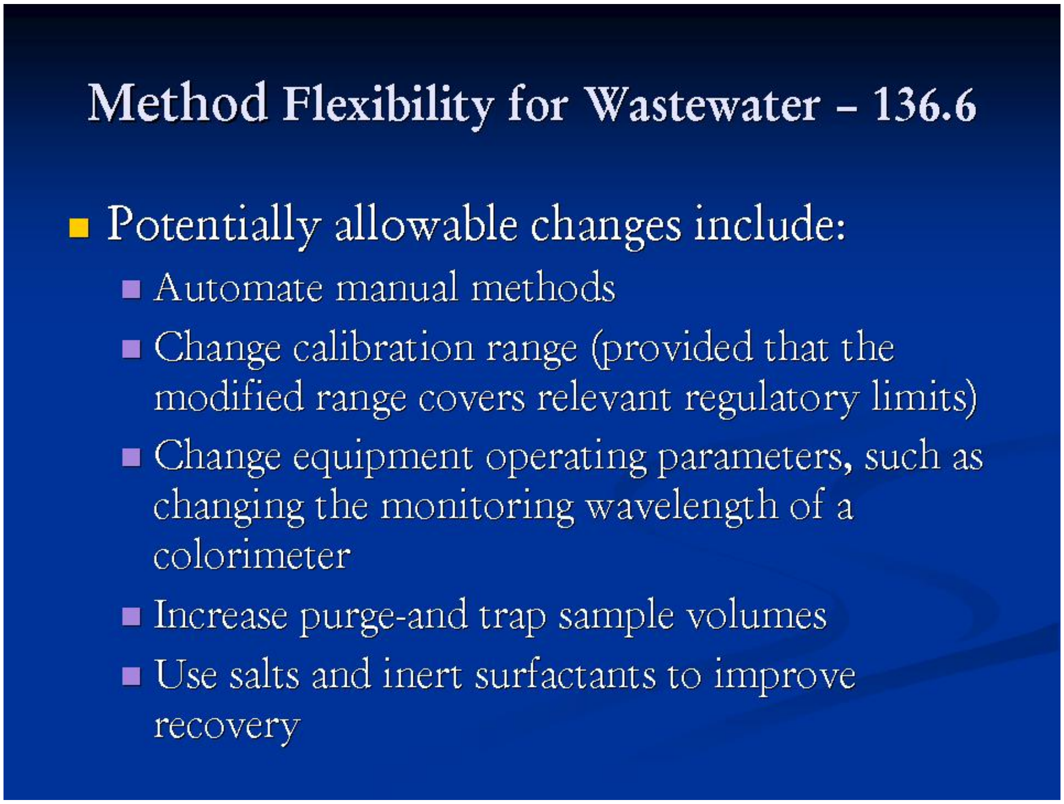
- Developing new criteria for recreational waters
- Likely to include new monitoring methods, such as rapid genetic methods based on quantitative polymerase chain reaction (qPCR) tests
- qPCR tests more rapid than culture methods
- Have developed a qPCR method for enterococci, and developing others for *E. coli*, and *Bacteroides* (a human specific bacterium)

Flexibility to Modify CWA Methods – 136.6

- Many approved methods provide analysts the flexibility to modify, without prior approval, of the modification.
- In 2007, EPA added 40 CFR Part 136.6 to describe additional (and to clarify existing) flexibility to modify any Part 136 chemical method without prior review.

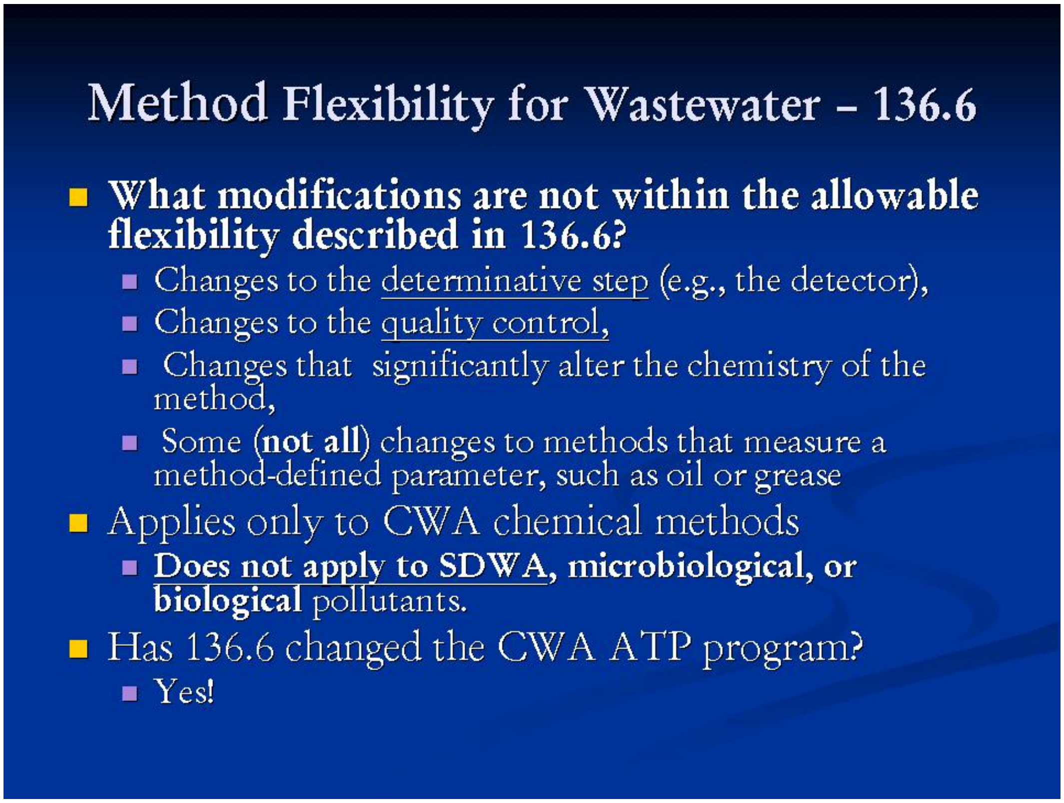
Method Flexibility for Wastewater – 136.6

- 40 CFR Part 136.6
  - May modify methods to overcome matrix problems, automate methods, or otherwise improve method efficiency or accuracy without unnecessary delay.
  - Modifications acceptable for compliance use, if the modification is documented to work, i.e., using your sample method performance should be comparable to that of the unmodified method.



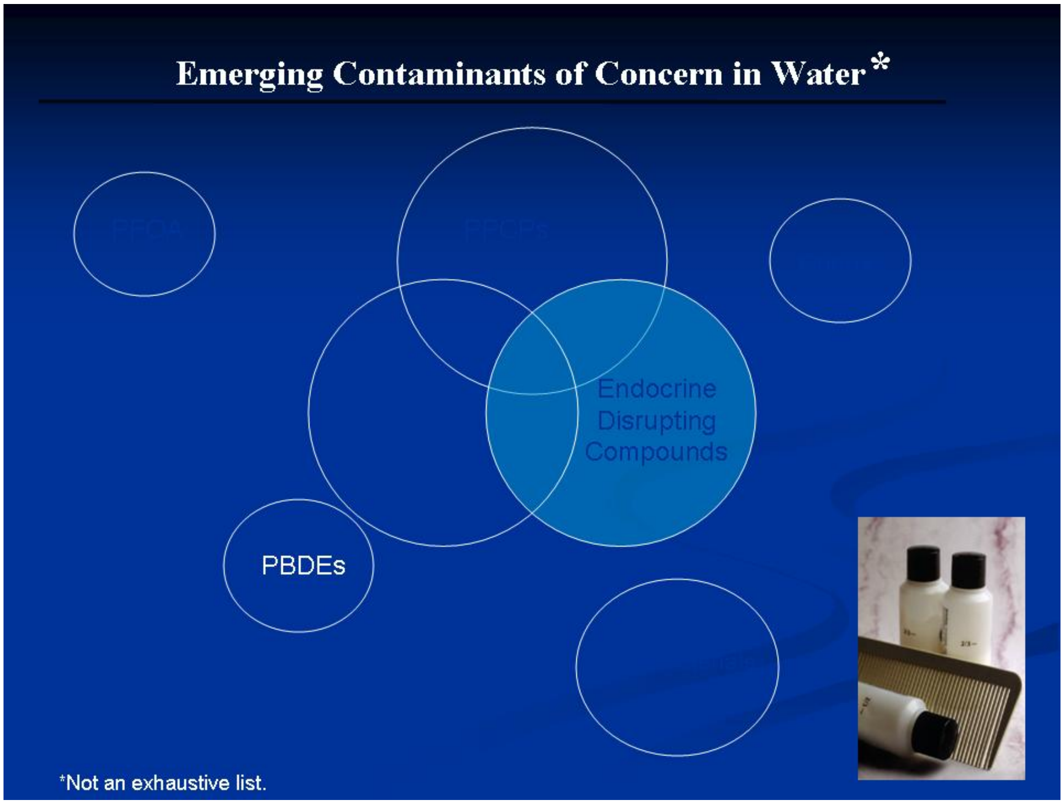
Method Flexibility for Wastewater – 136.6

- Potentially allowable changes include:
  - Automate manual methods
  - Change calibration range (provided that the modified range covers relevant regulatory limits)
  - Change equipment operating parameters, such as changing the monitoring wavelength of a colorimeter
  - Increase purge-and trap sample volumes
  - Use salts and inert surfactants to improve recovery



Method Flexibility for Wastewater – 136.6

- **What modifications are not within the allowable flexibility described in 136.6?**
  - Changes to the determinative step (e.g., the detector),
  - Changes to the quality control,
  - Changes that significantly alter the chemistry of the method,
  - Some (**not all**) changes to methods that measure a method-defined parameter, such as oil or grease
- Applies only to CWA chemical methods
  - **Does not apply to SDWA, microbiological, or biological** pollutants.
- Has 136.6 changed the CWA ATP program?
  - Yes!



Emerging Contaminants of Concern in Water*

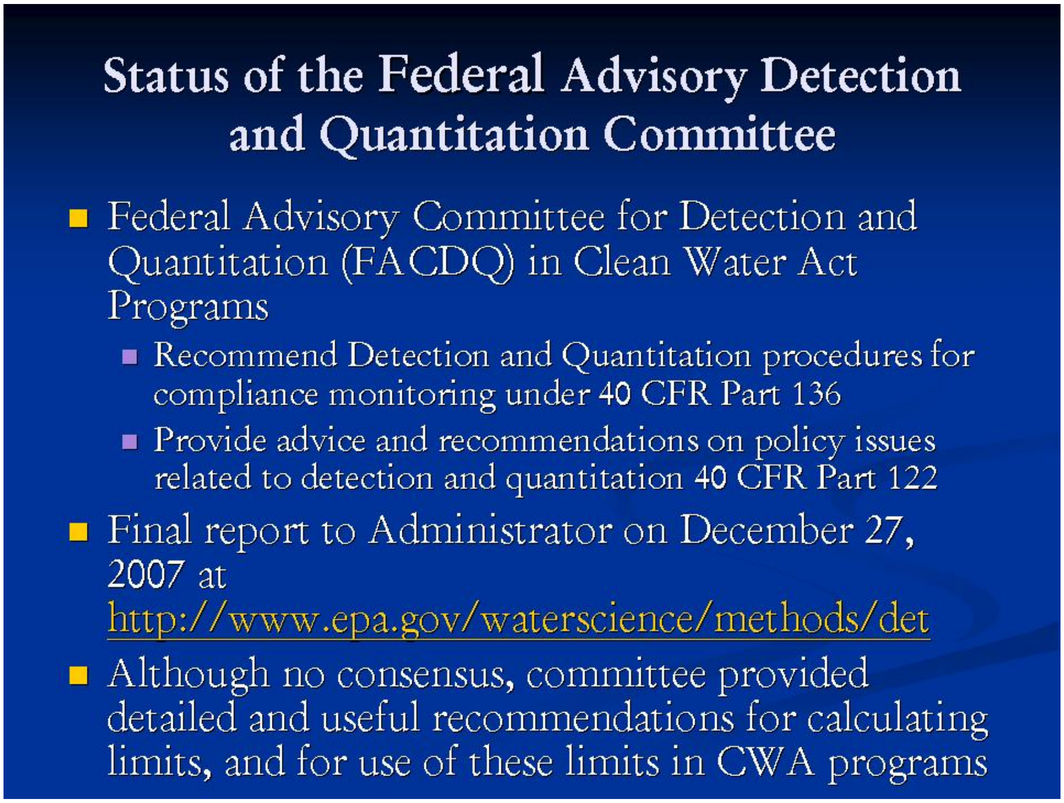
PFOA

PPCPs

Endocrine
Disrupting
Compounds

PBDEs

*Not an exhaustive list.



Status of the Federal Advisory Detection and Quantitation Committee

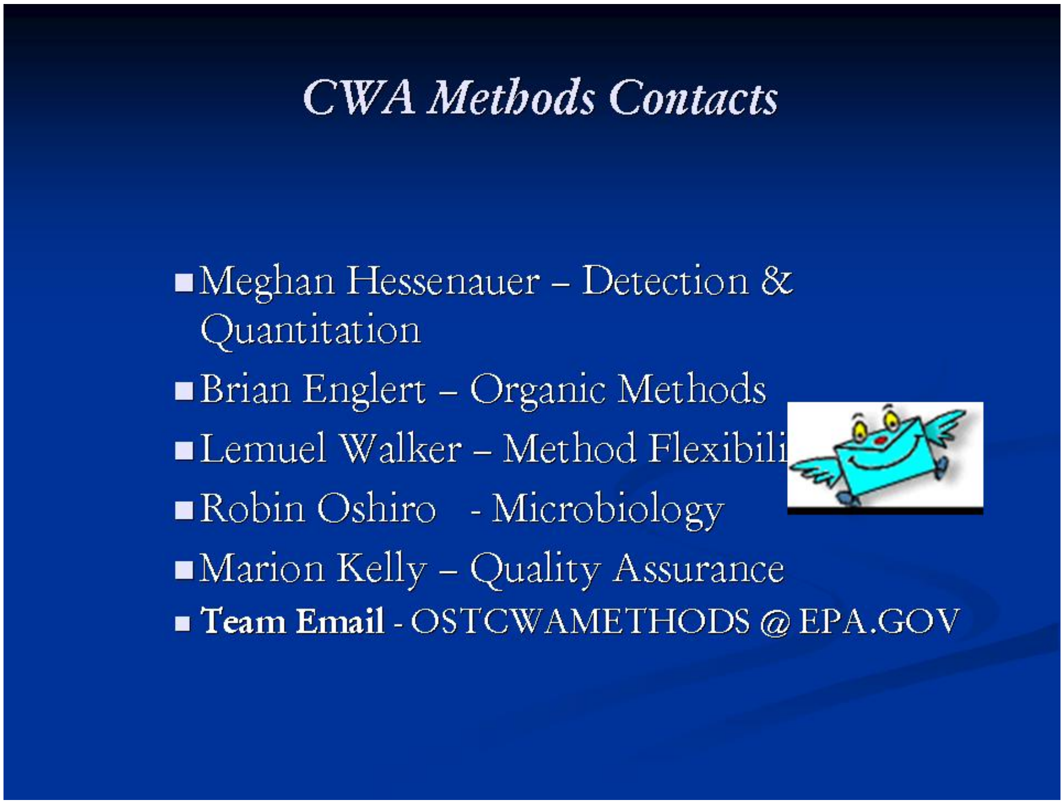
- Federal Advisory Committee for Detection and Quantitation (FACDQ) in Clean Water Act Programs
  - Recommend Detection and Quantitation procedures for compliance monitoring under 40 CFR Part 136
  - Provide advice and recommendations on policy issues related to detection and quantitation 40 CFR Part 122
- Final report to Administrator on December 27, 2007 at http://www.epa.gov/waterscience/methods/det
- Although no consensus, committee provided detailed and useful recommendations for calculating limits, and for use of these limits in CWA programs

Desirable DL/QL Procedure Characteristics

- Incorporate temporal variability
- Reflect routine performance
- Address matrices
- Evaluate the entire test method
- Address blank bias
- Address intermittent blank contamination
- Produce an explicit estimate of bias and precision
- Address false positive and negative rates

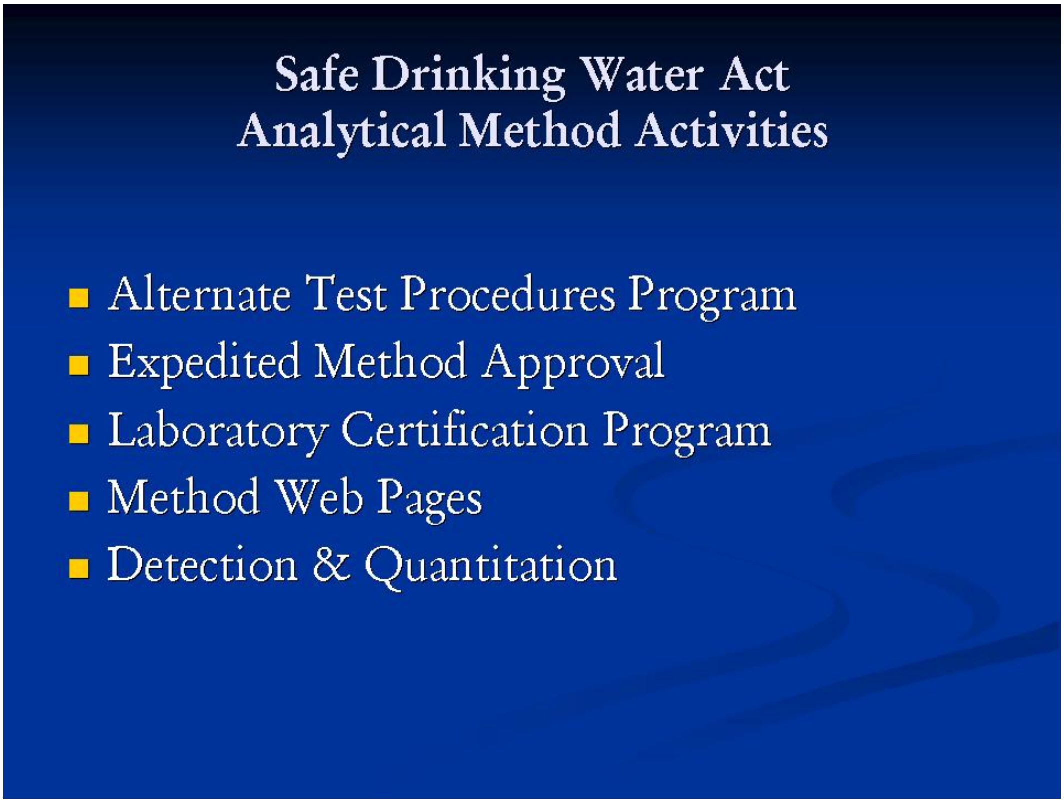
Status and Next Steps

- Collaborated with EPA programs, such as OGWDW and OSW to
  - Expand the scope of the FACDQ DL-QL procedure
    - Now also describes how a method developer may establish best performance in setting benchmark limits
  - Design the study plan for the next Lab Pilot of this procedure
- Preparing the study plan formal peer review of this Lab Pilot
  - Evaluate the DL-QL procedure in a several labs using chemical methods for metal and organic CWA pollutants
- Conduct the Lab Pilot in 2009



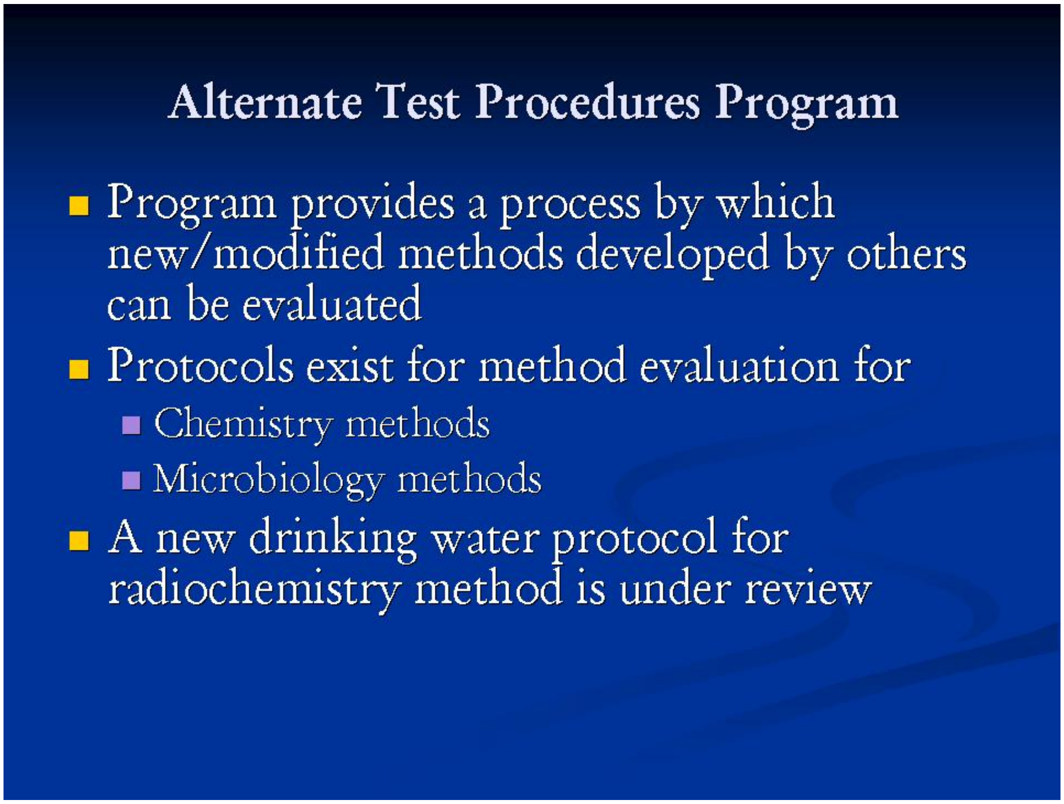
CWA Methods Contacts

- Meghan Hessenauer – Detection & Quantitation
- Brian Englert – Organic Methods
- Lemuel Walker – Method Flexibili
- Robin Oshiro - Microbiology
- Marion Kelly – Quality Assurance
- **Team Email** - OSTCWAMETHODS @ EPA.GOV



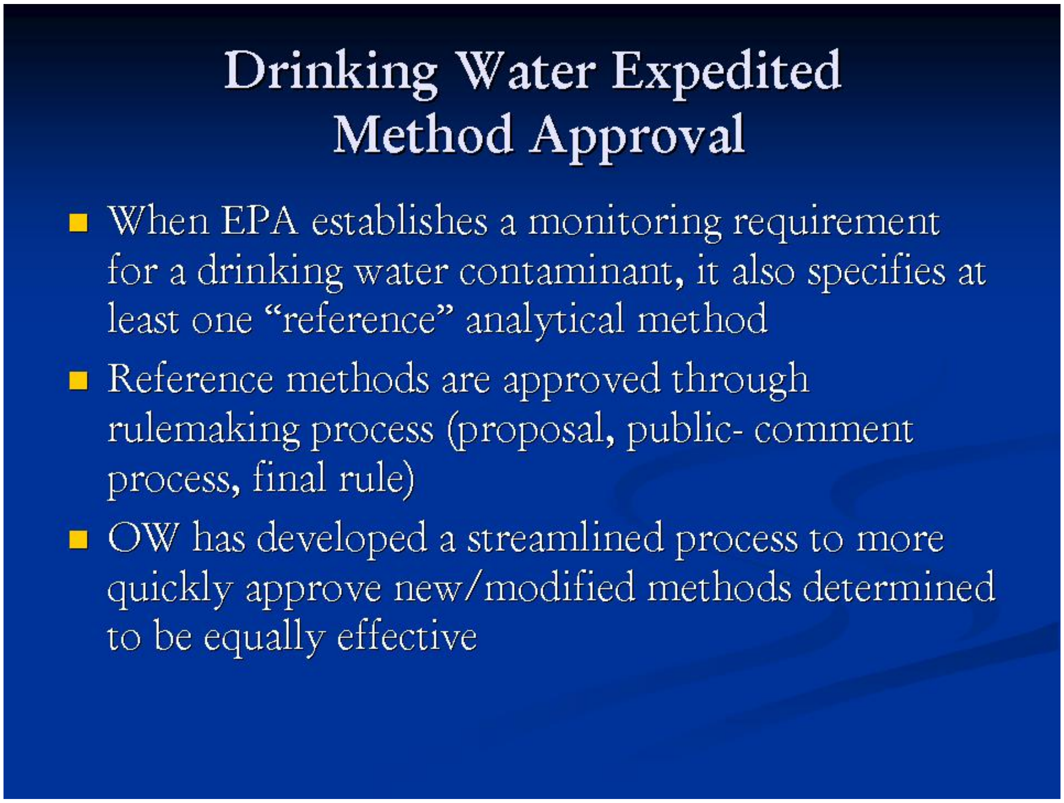
Safe Drinking Water Act Analytical Method Activities

- Alternate Test Procedures Program
- Expedited Method Approval
- Laboratory Certification Program
- Method Web Pages
- Detection & Quantitation



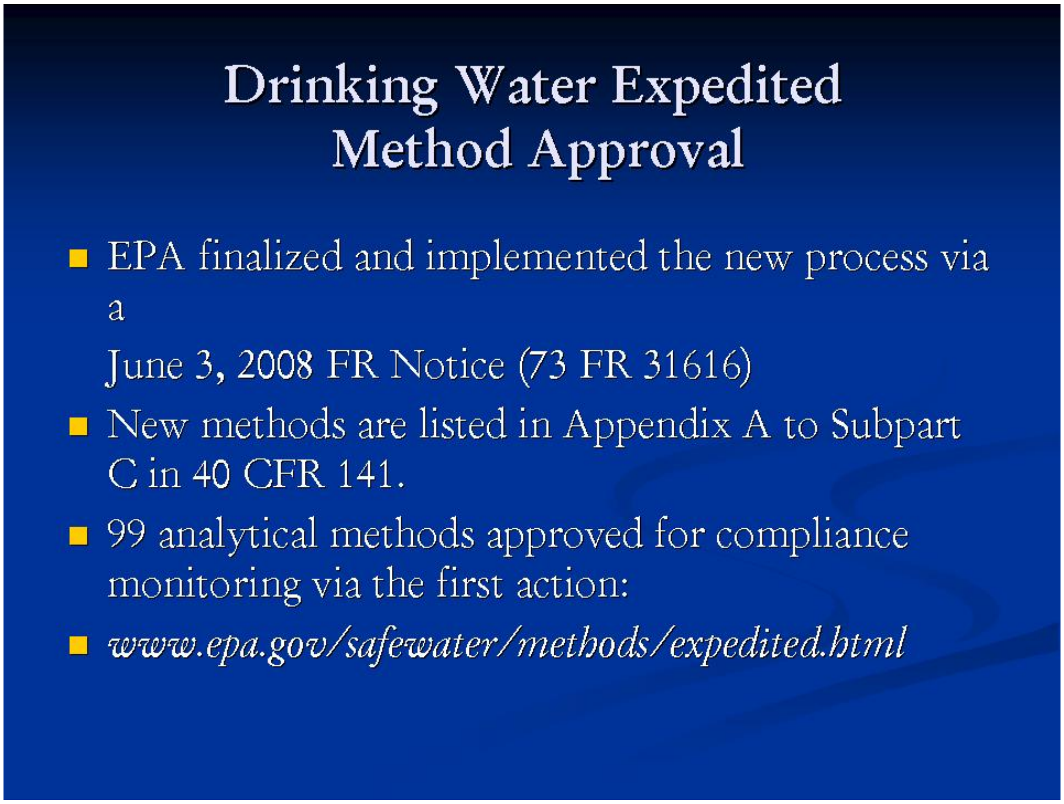
Alternate Test Procedures Program

- Program provides a process by which new/modified methods developed by others can be evaluated
- Protocols exist for method evaluation for
  - Chemistry methods
  - Microbiology methods
- A new drinking water protocol for radiochemistry method is under review



Drinking Water Expedited Method Approval

- When EPA establishes a monitoring requirement for a drinking water contaminant, it also specifies at least one “reference” analytical method
- Reference methods are approved through rulemaking process (proposal, public- comment process, final rule)
- OW has developed a streamlined process to more quickly approve new/modified methods determined to be equally effective



Drinking Water Expedited Method Approval

- EPA finalized and implemented the new process via a

  June 3, 2008 FR Notice (73 FR 31616)
- New methods are listed in Appendix A to Subpart C in 40 CFR 141.
- 99 analytical methods approved for compliance monitoring via the first action:
- *www.epa.gov/safewater/methods/expedited.html*

Drinking Water Laboratory Certification Program

- Published ***Supplement 1*** to the Fifth Edition of the ***Manual for the Certification of Laboratories Analyzing Drinking Water*** in June 2008.
  - Quality Control Program requirement (as specified in EPA Methods). The *Supplement* further emphasizes this requirement and encourages programs based on ISO 9001 (achieves greater consistency with TNI approach).
  - Includes additional emphasis on fraud and ethics issues (training, prevention, detection, policy) for laboratories and laboratory certification officers

Drinking Water Methods Web Pages

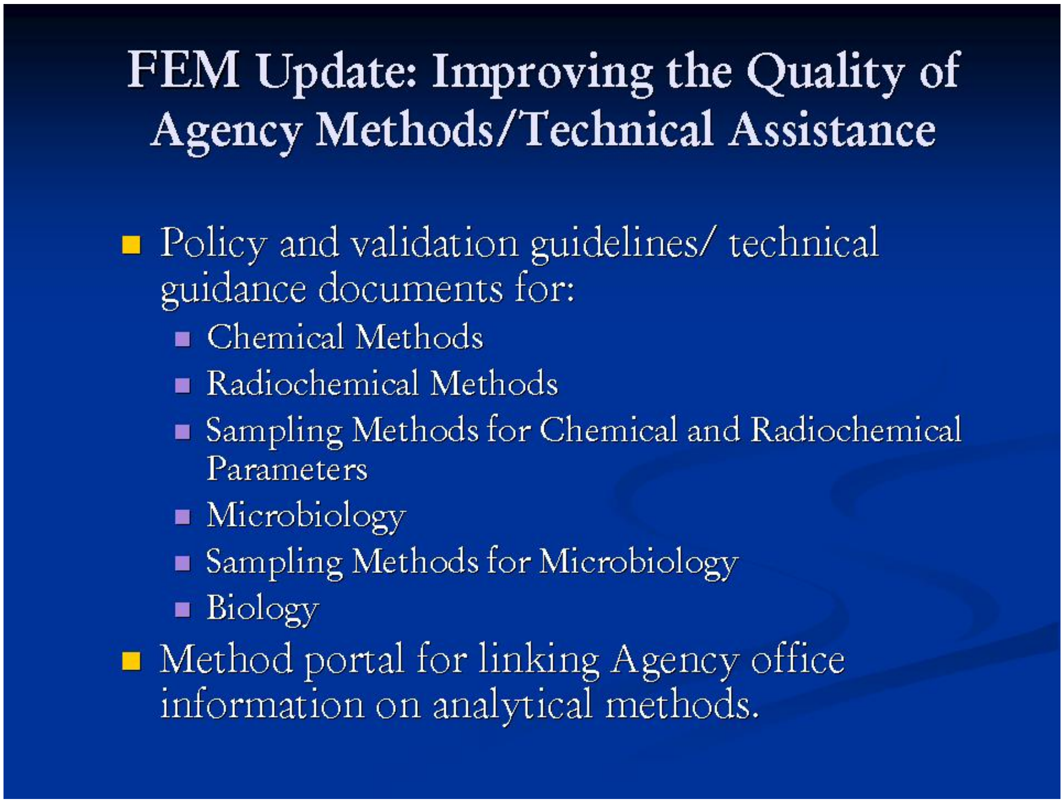
- *www.epa.gov/safewater/methods*
- Extensive updates in June 2008 to:
  - consolidate all drinking water methods information
  - update list of approved methods
  - more clearly identify methods applicable to each SDWA-based regulation

Drinking Water Methods: Detection/Quantitation

- "Lowest-Concentration Minimum Reporting Level" (LCMRL) concept developed by OGWDW's Technical Support Center for use in the ongoing Unregulated Contaminant Monitoring program (UCMR2)
- More details on the revised procedure are being presented at the NEMC

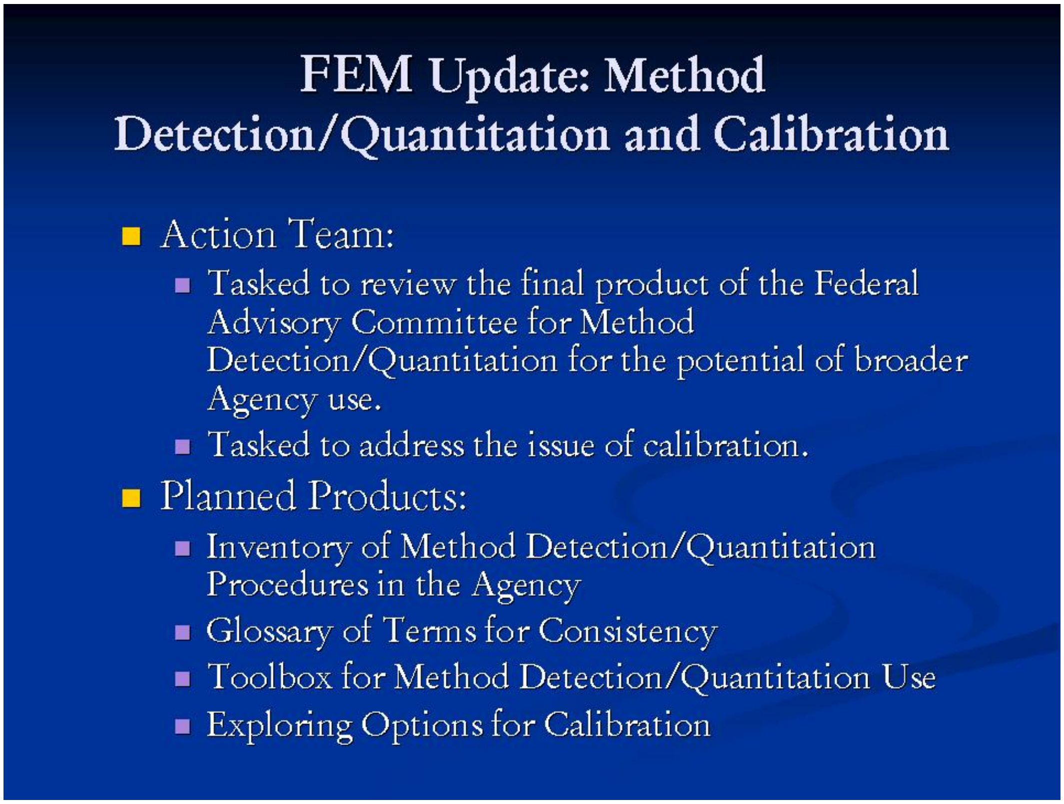
SDWA Methods Contacts

- David Munch – Chemistry Methods
- Sandhya Parshionikar – Microbiological Methods
- Steve Wendelken – Drinking Water ATP Program
- Patricia Fair – Expedited Method Approval
- Judy Brisbin, Jennifer Best, Michella Karapondo – Laboratory Certification Program
- Greg Carroll – Director, Technical Support Center



FEM Update: Improving the Quality of Agency Methods/Technical Assistance

- Policy and validation guidelines/ technical guidance documents for:
  - Chemical Methods
  - Radiochemical Methods
  - Sampling Methods for Chemical and Radiochemical Parameters
  - Microbiology
  - Sampling Methods for Microbiology
  - Biology
- Method portal for linking Agency office information on analytical methods.



FEM Update: Method Detection/Quantitation and Calibration

- Action Team:
  - Tasked to review the final product of the Federal Advisory Committee for Method Detection/Quantitation for the potential of broader Agency use.
  - Tasked to address the issue of calibration.
- Planned Products:
  - Inventory of Method Detection/Quantitation Procedures in the Agency
  - Glossary of Terms for Consistency
  - Toolbox for Method Detection/Quantitation Use
  - Exploring Options for Calibration

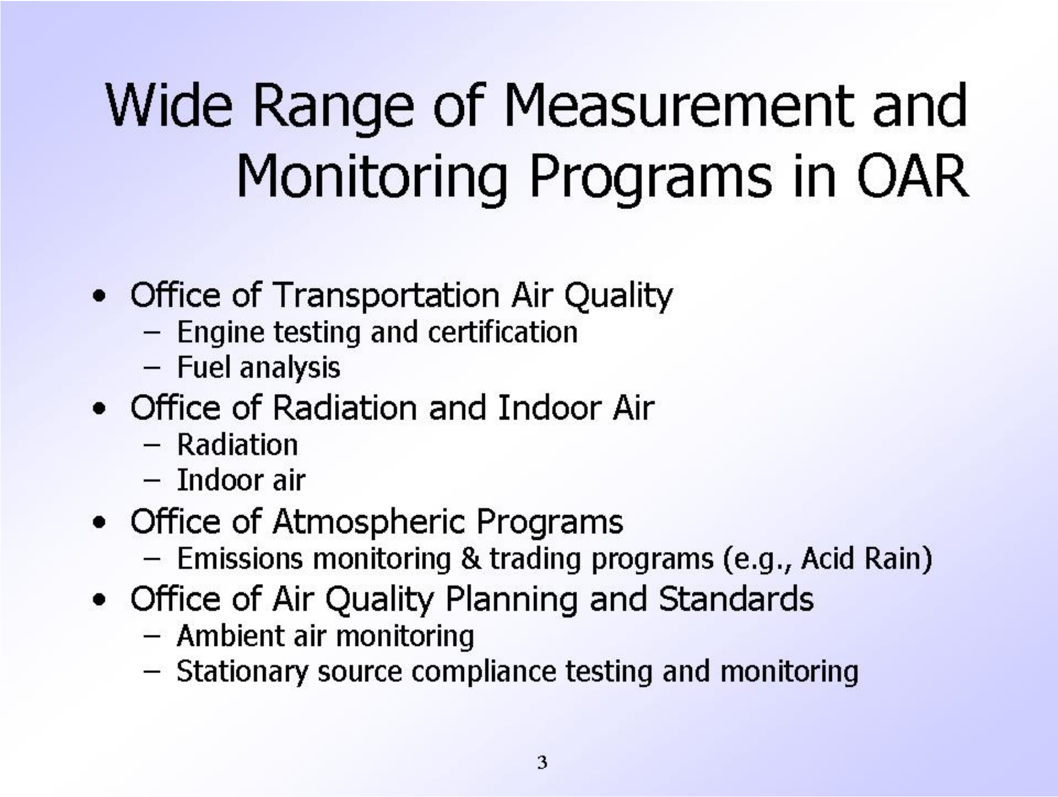
Office of Air and Radiation Measurement, Monitoring and the Performance Approach

Robert D. Brenner
Director, Office of Policy Analysis and Review
EPA Office of Air and Radiation

National Environmental Monitoring Conference
Washington, DC
August 13, 2008

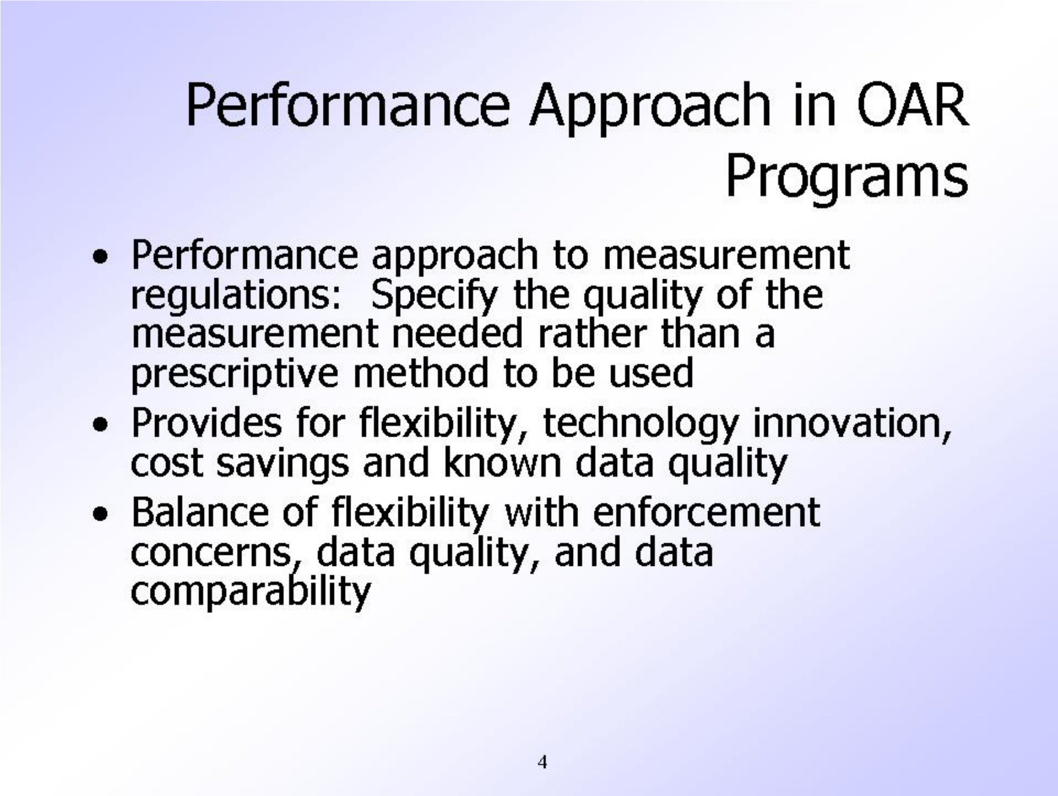
Topics

- Measurement and Monitoring Programs in Office of Air & Radiation (OAR)
- OAR Use of Performance Approach
- Importance of Monitoring in Air Programs
- New Initiatives in Measurement and Monitoring



Wide Range of Measurement and Monitoring Programs in OAR

- Office of Transportation Air Quality
  - Engine testing and certification
  - Fuel analysis
- Office of Radiation and Indoor Air
  - Radiation
  - Indoor air
- Office of Atmospheric Programs
  - Emissions monitoring & trading programs (e.g., Acid Rain)
- Office of Air Quality Planning and Standards
  - Ambient air monitoring
  - Stationary source compliance testing and monitoring



Performance Approach in OAR Programs

- Performance approach to measurement regulations: Specify the quality of the measurement needed rather than a prescriptive method to be used
- Provides for flexibility, technology innovation, cost savings and known data quality
- Balance of flexibility with enforcement concerns, data quality, and data comparability

Performance Approach in Facility Monitoring

- Office of Air Quality Planning & Standards and Office of Atmospheric Programs use performance criteria in regulations for continuous monitoring of stationary sources
- Sulfur dioxide and nitrogen oxide monitoring as well as recent mercury monitoring requirements all rely on the performance approach
- Performance approach relatively straightforward for gaseous pollutants, but much more challenging for particulate matter measurements

Performance Approach in Vehicle Emissions Durability Program

- Car makers must demonstrate that their vehicles will meet emission standards over vehicle lifetime to gain annual certification
- Prior test protocols were very prescriptive
- New rules affecting model year 2008 vehicles allow for manufacturer-specified driving-based and laboratory-based testing of vehicles and their emission control components

Performance Approach in Radiation Testing

- ORIA led the development of the Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) Manual
  - MARLAP Manual provides a performance approach for all types of radiochemical measurements
- 40 CFR Part 61 regulates radionuclide emissions from stationary sources citing Reference Methods 111, 114 and 115

Monitoring Key in Air Programs for Over Three Decades

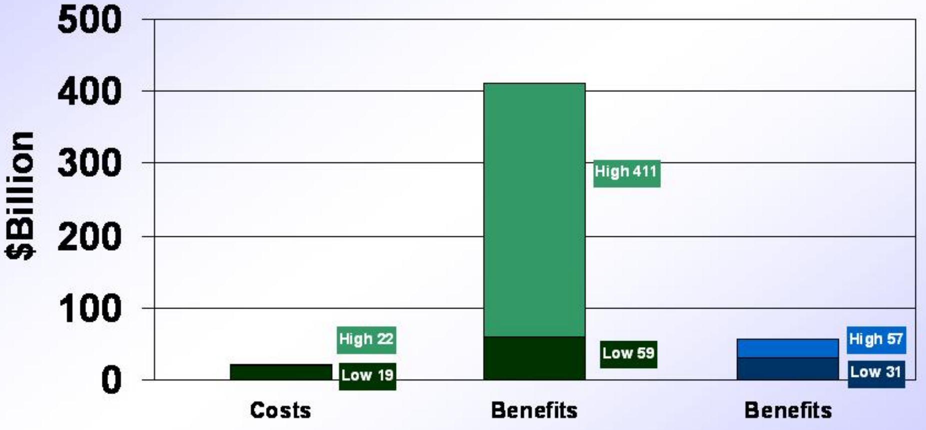
- Ambient air monitoring program directly assesses progress toward clean air for criteria and toxic pollutants
  - Track trends, compare to health outcomes, and evaluate benefits of control programs
  - Supports regulation development
  - Helps answer basic research questions

Monitoring Key in Air Programs for Over Three Decades

- Facility monitoring provides real-time feedback to source operators, encourages attention to processes and controls and yields better compliance and lower emissions
  - Acid Rain Monitoring Program
  - Clean Air Interstate Rule
  - Compliance Assurance Monitoring
- Strong facility monitoring requirements and resulting data yield innovation opportunities
  - More robust mercury monitoring systems
  - Creative and useful program impact assessments
  - Design of more effective programs

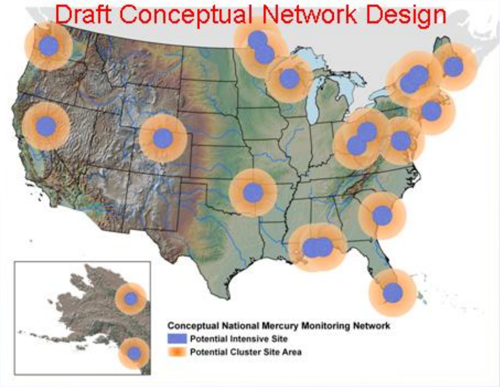
Annual Costs and Benefits of Air Program Compared with Benefits of All Other U.S. Government Regulations Combined

(from Draft 2007 Report to Congress on the Cost and Benefits of Federal Regulations (OMB))



MercNet – Monitoring mercury in air, water, land, fish and wildlife

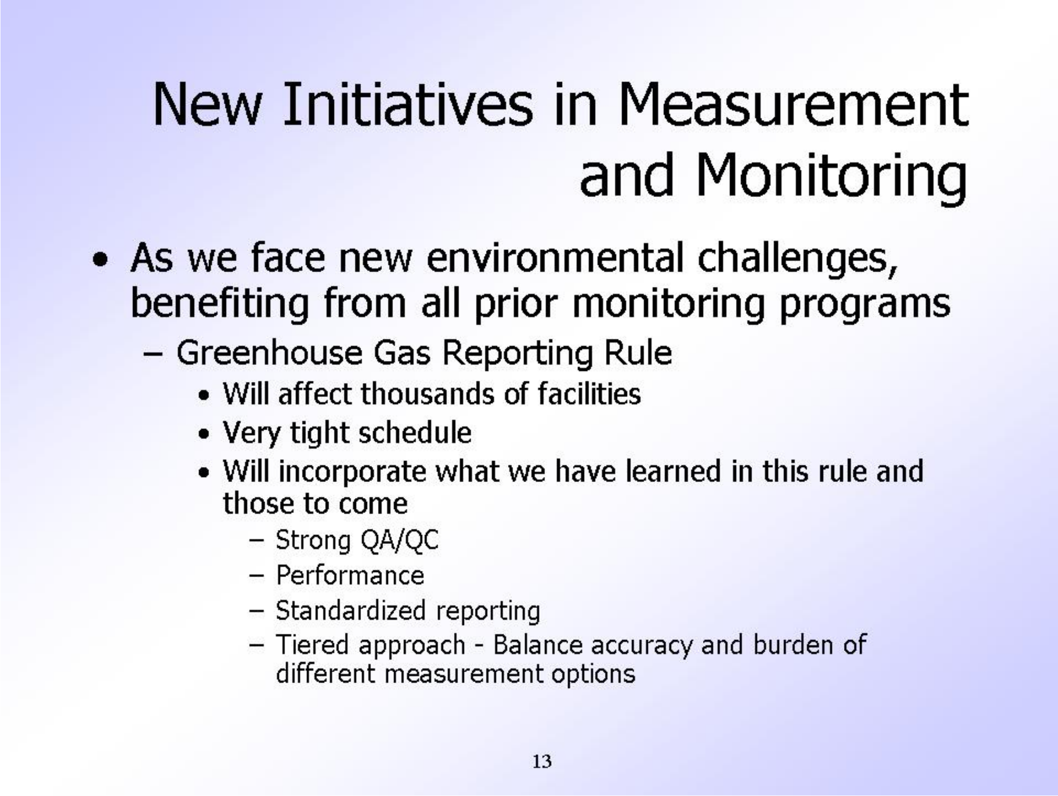
- **Mercury Monitoring Goal** (Established by scientists from federal, state, academic, and private organizations at a 2008 National Mercury Monitoring Workshop)
  - Systematically monitor, assess, and report on indicators of nationwide changes in atmospheric mercury deposition and concentrations of mercury in land, water, and biota in coastal and freshwater ecosystems in response to changing mercury emissions over time
- **Proposed Monitoring Network Design Elements**
  - Combination of "intensive sites" and "cluster sites"
  - 10-20 intensive sites, accompanied by about 20 cluster sites for each intensive site
  - National distribution of sites to understand the sources, consequences, and changes in U.S. mercury pollution
  - Sites operate for 10-40 years to quantify the range of responses expected for many ecosystem types
  - Build on existing monitoring efforts, where possible, to maximize information, benefits and coordination with existing resources


Draft Conceptual Network Design

Based on Mason et al. 2005 & Harris et al. 2007

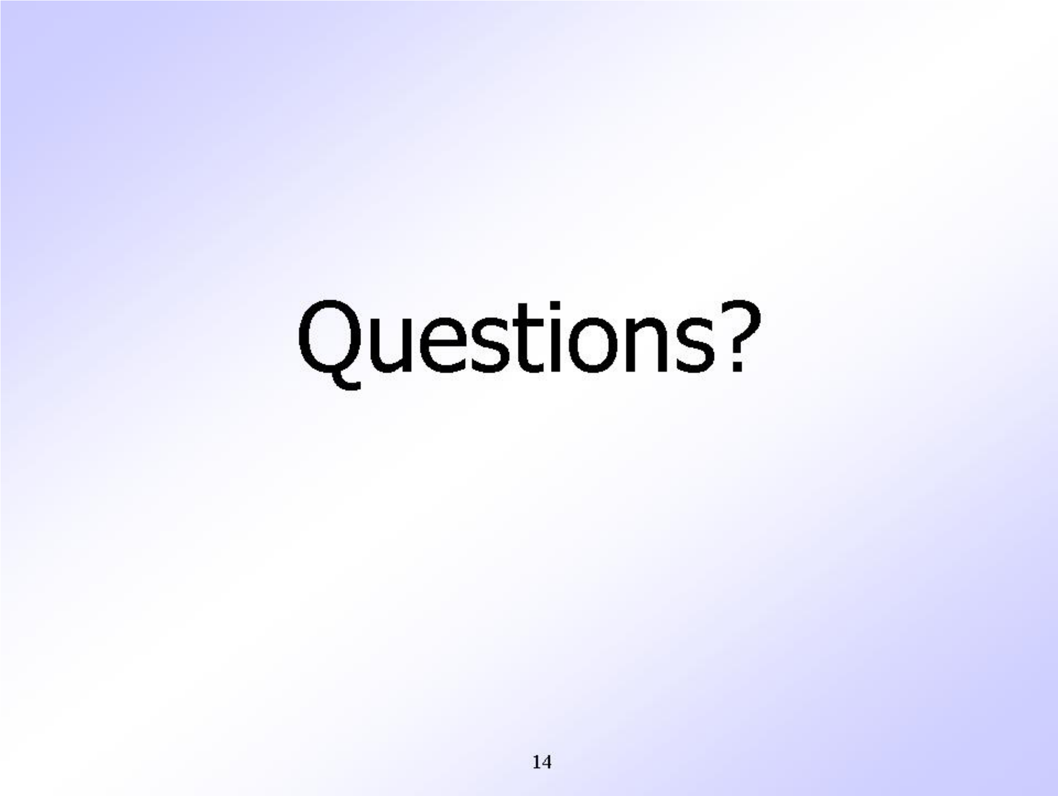
New Initiatives in Measurement and Monitoring

- Predictive Emissions Monitoring Systems or PEMS
  - Software that develops a numerical relationship between unit's operating parameters and emissions
  - For sources (e.g., gas/oil-fired boilers, heaters, and turbines) that can use PEMS, it's significantly less expensive than a NOx CEMS
  - Several PEMS recently approved as monitoring alternatives under Acid Rain and NOx Budget Programs
  - PEMS performance specification to be promulgated under 40 CFR Part 60 by the end of the year

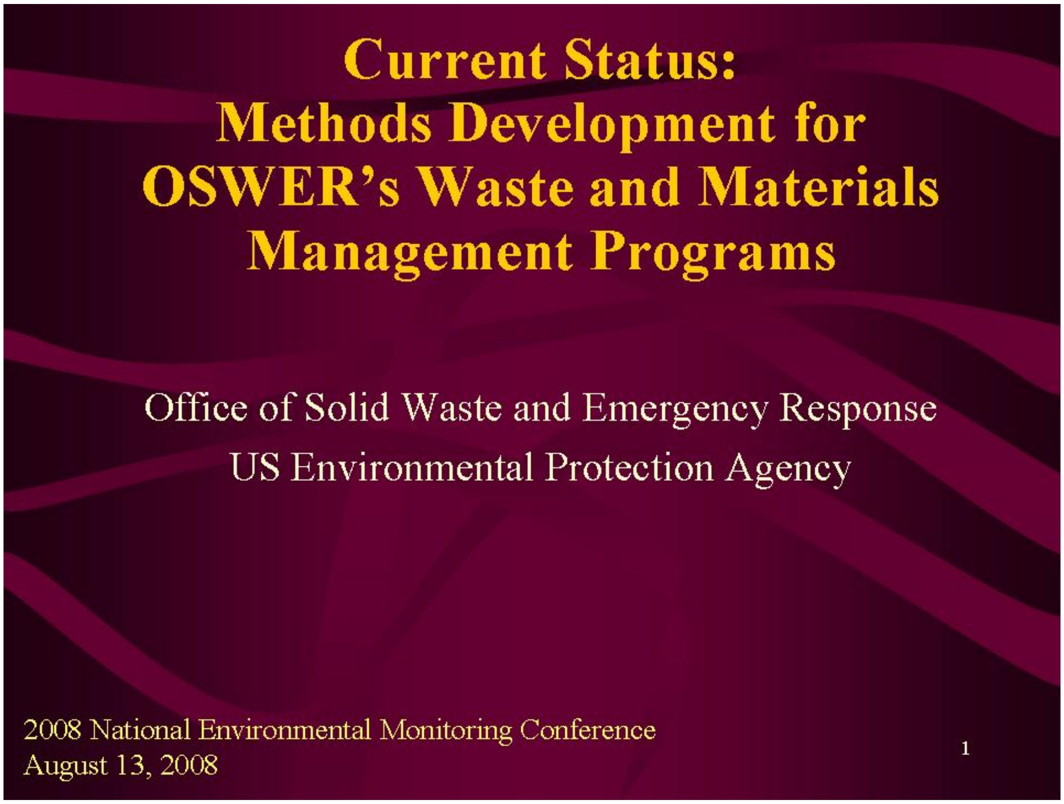


New Initiatives in Measurement and Monitoring

- As we face new environmental challenges, benefiting from all prior monitoring programs
  - Greenhouse Gas Reporting Rule
    - Will affect thousands of facilities
    - Very tight schedule
    - Will incorporate what we have learned in this rule and those to come
      - Strong QA/QC
      - Performance
      - Standardized reporting
      - Tiered approach - Balance accuracy and burden of different measurement options



Questions?



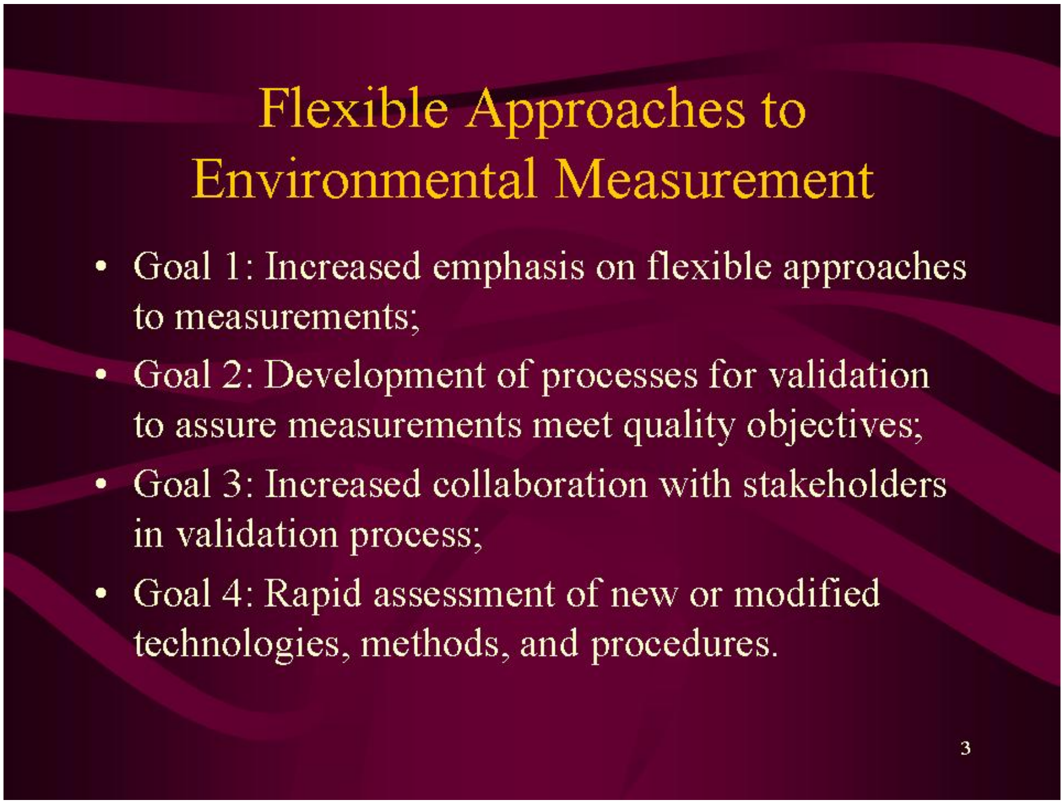
Current Status: Methods Development for OSWER's Waste and Materials Management Programs

Office of Solid Waste and Emergency Response
US Environmental Protection Agency

2008 National Environmental Monitoring Conference
August 13, 2008

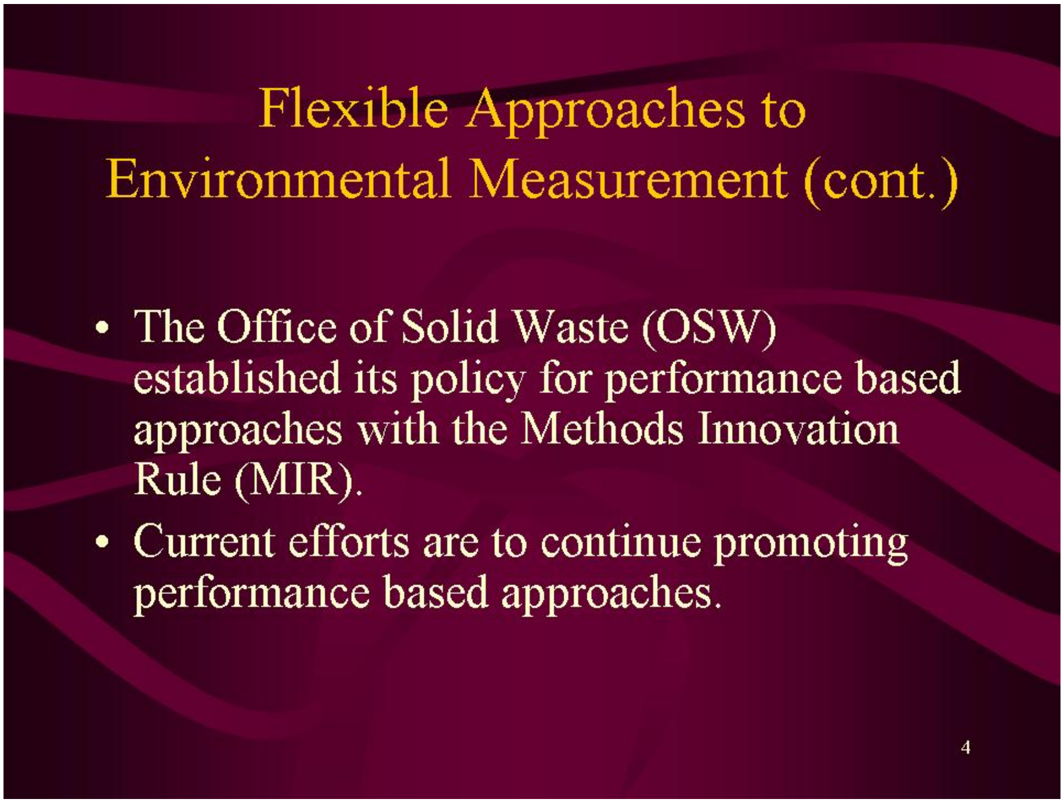
Major Topics to Be Covered

- Performance Based Approaches
- SW-846: Updates and Revisions
- Leach Test Framework: Support for Materials Management and Sustainability Programs
- New Methods and Standard Reference Materials: Support for Risk-Based Cleanups
- Overview of Significant Related Activities



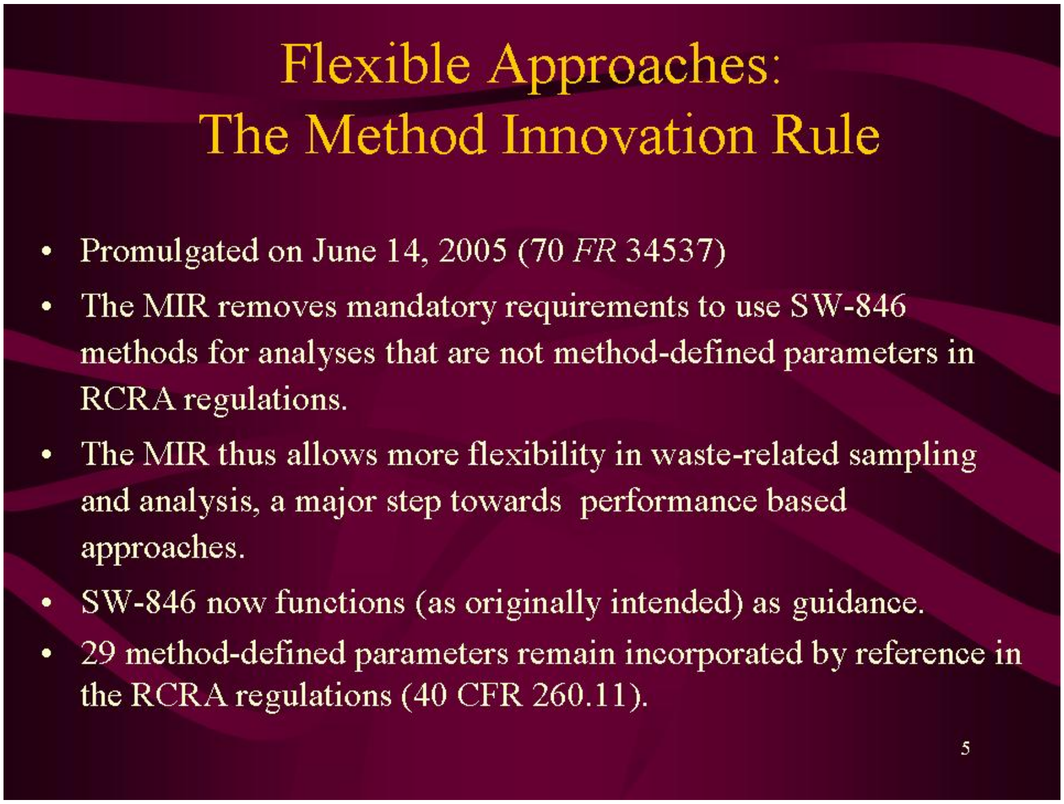
Flexible Approaches to Environmental Measurement

- Goal 1: Increased emphasis on flexible approaches to measurements;
- Goal 2: Development of processes for validation to assure measurements meet quality objectives;
- Goal 3: Increased collaboration with stakeholders in validation process;
- Goal 4: Rapid assessment of new or modified technologies, methods, and procedures.



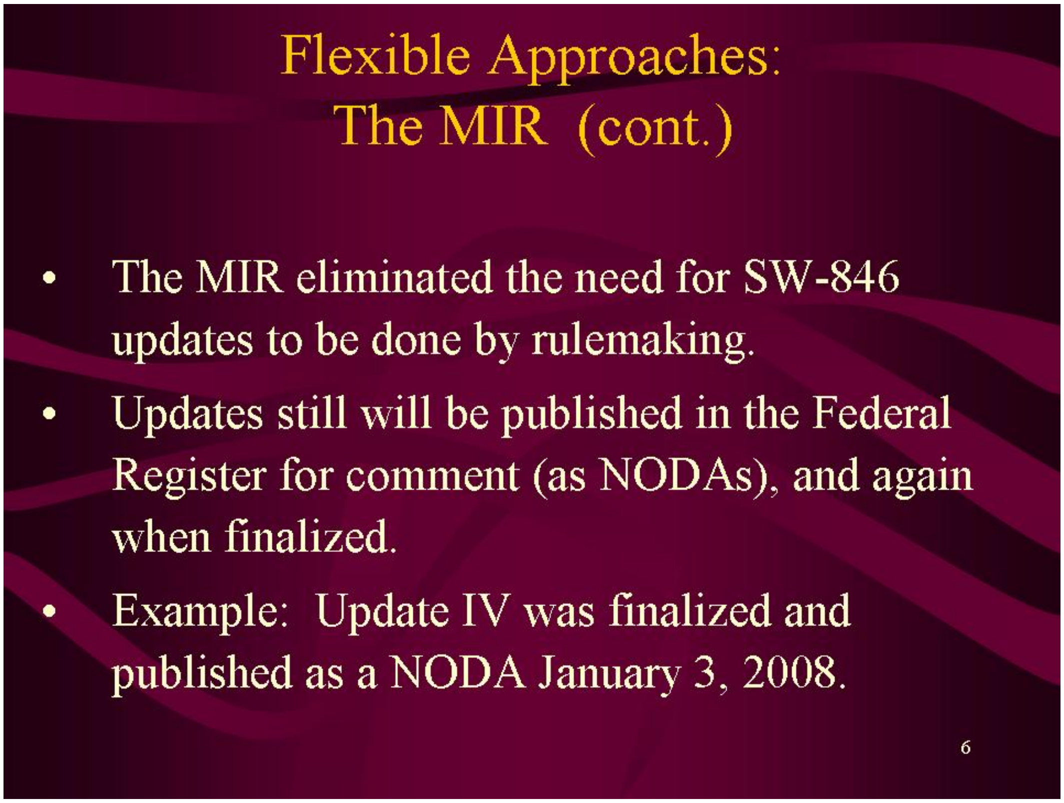
Flexible Approaches to Environmental Measurement (cont.)

- The Office of Solid Waste (OSW) established its policy for performance based approaches with the Methods Innovation Rule (MIR).
- Current efforts are to continue promoting performance based approaches.



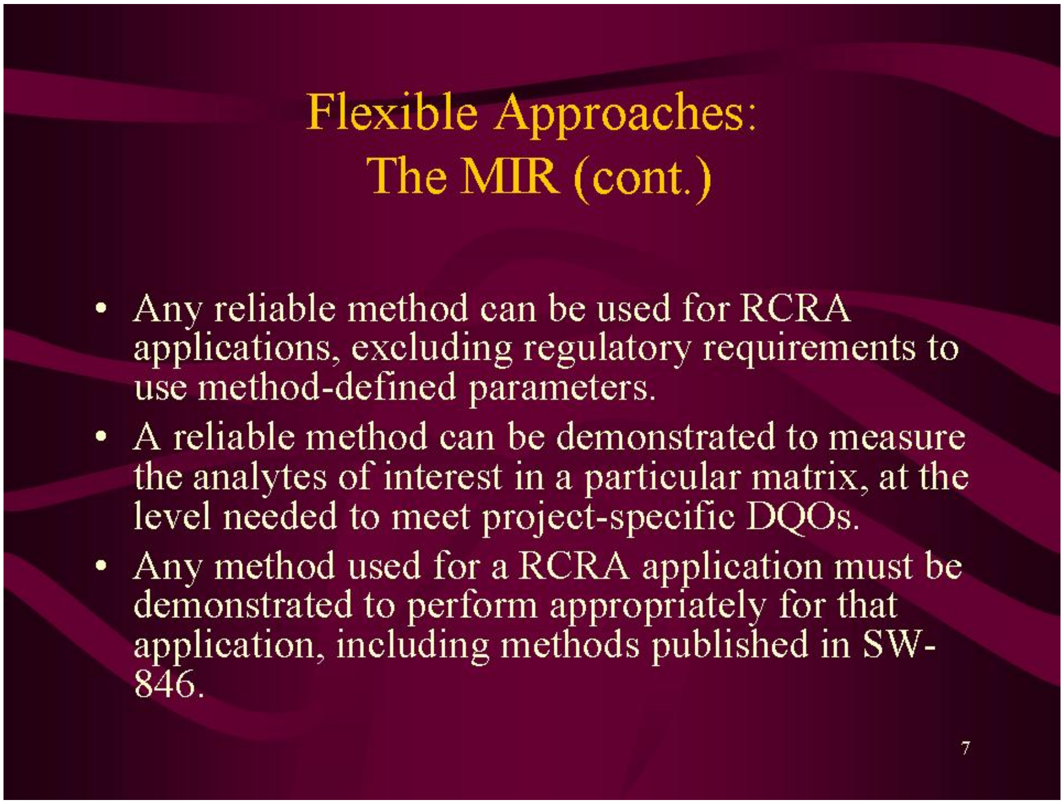
Flexible Approaches:
The Method Innovation Rule

- Promulgated on June 14, 2005 (70 *FR* 34537)
- The MIR removes mandatory requirements to use SW-846 methods for analyses that are not method-defined parameters in RCRA regulations.
- The MIR thus allows more flexibility in waste-related sampling and analysis, a major step towards performance based approaches.
- SW-846 now functions (as originally intended) as guidance.
- 29 method-defined parameters remain incorporated by reference in the RCRA regulations (40 CFR 260.11).



Flexible Approaches:
The MIR (cont.)

- The MIR eliminated the need for SW-846 updates to be done by rulemaking.
- Updates still will be published in the Federal Register for comment (as NODAs), and again when finalized.
- Example: Update IV was finalized and published as a NODA January 3, 2008.

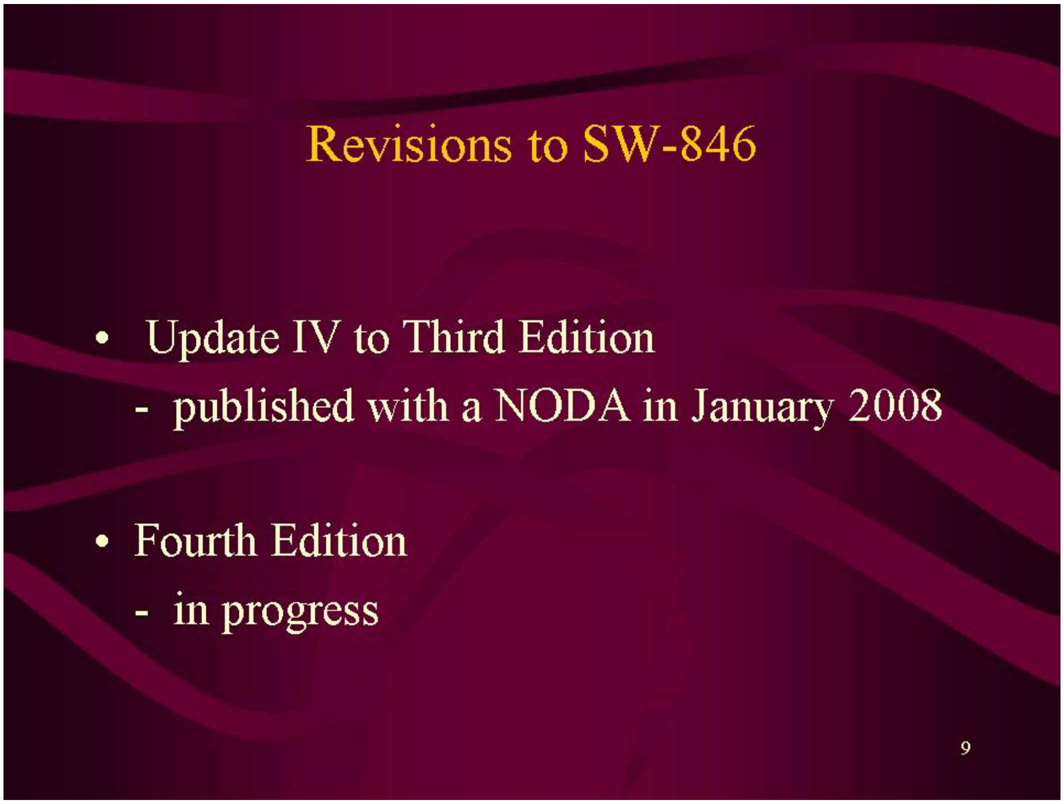


Flexible Approaches:
The MIR (cont.)

- Any reliable method can be used for RCRA applications, excluding regulatory requirements to use method-defined parameters.
- A reliable method can be demonstrated to measure the analytes of interest in a particular matrix, at the level needed to meet project-specific DQOs.
- Any method used for a RCRA application must be demonstrated to perform appropriately for that application, including methods published in SW-846.

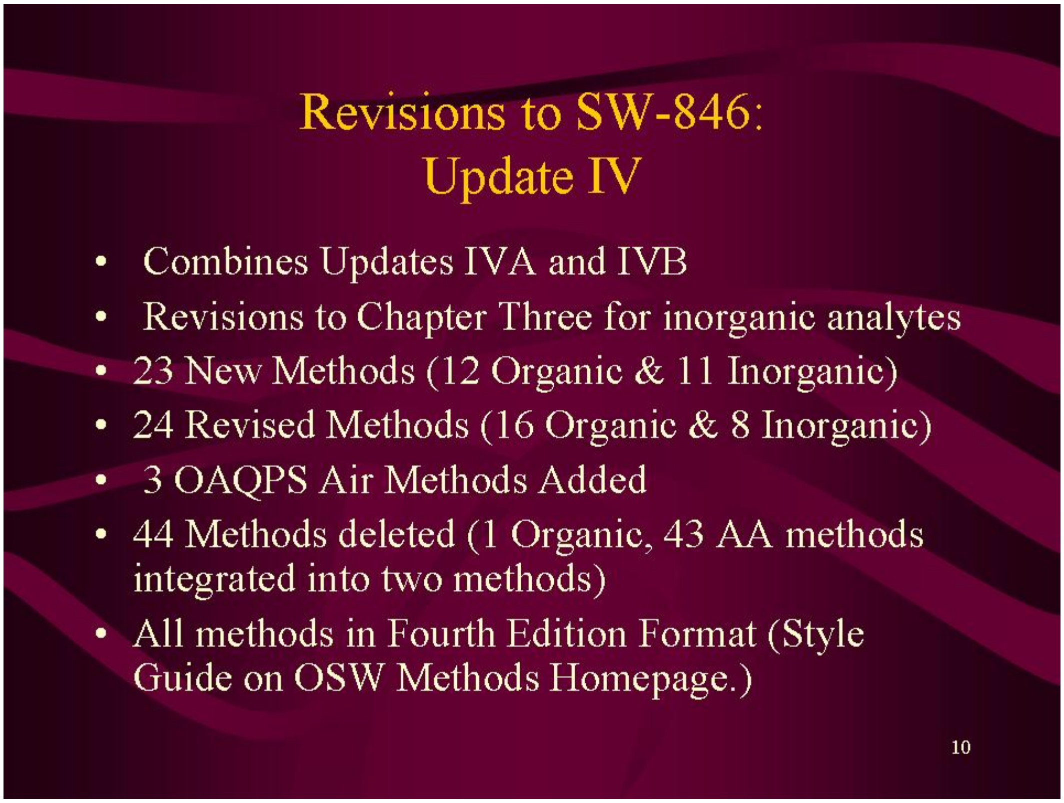
Flexible Approaches:
Examples of Collaboration

- Region 10 is leading the development of two new SW-846 methods for arsenic speciation.
- OSW is collaborating with ORD-LV and Region 4 for the development of one new SW-846 method for toxaphene and toxaphene congeners.



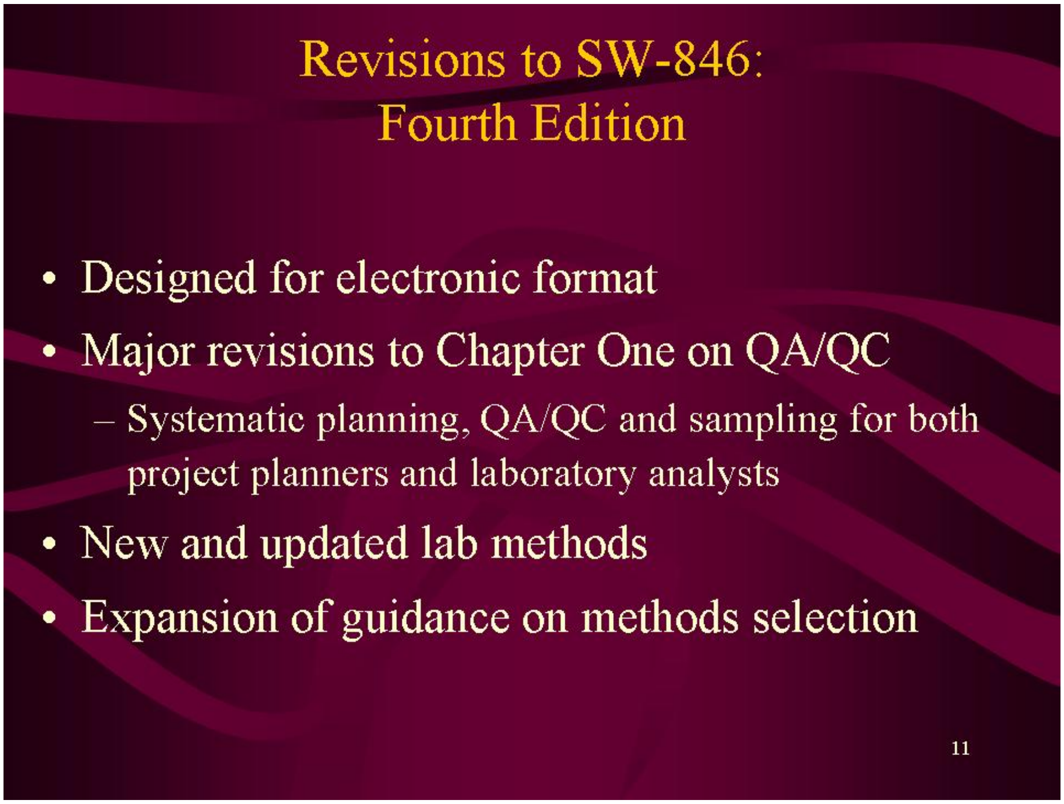
Revisions to SW-846

- Update IV to Third Edition
  - published with a NODA in January 2008
- Fourth Edition
  - in progress



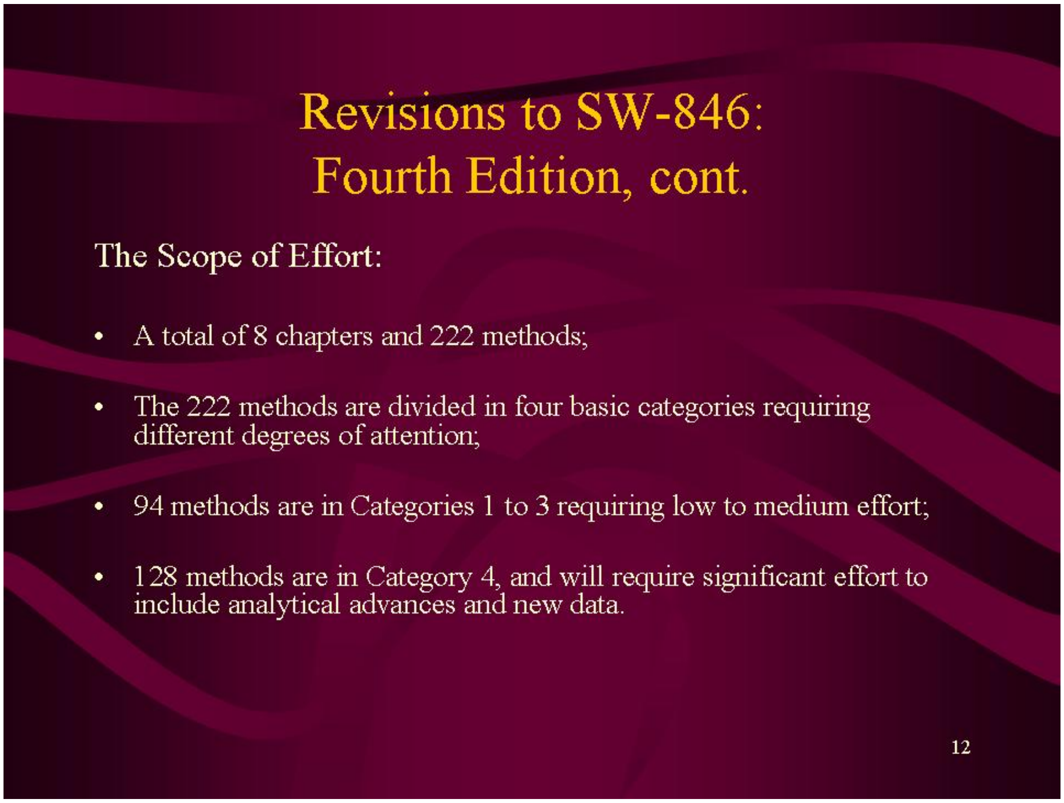
Revisions to SW-846:
Update IV

- Combines Updates IVA and IVB
- Revisions to Chapter Three for inorganic analytes
- 23 New Methods (12 Organic & 11 Inorganic)
- 24 Revised Methods (16 Organic & 8 Inorganic)
- 3 OAQPS Air Methods Added
- 44 Methods deleted (1 Organic, 43 AA methods integrated into two methods)
- All methods in Fourth Edition Format (Style Guide on OSW Methods Homepage.)



Revisions to SW-846:
Fourth Edition

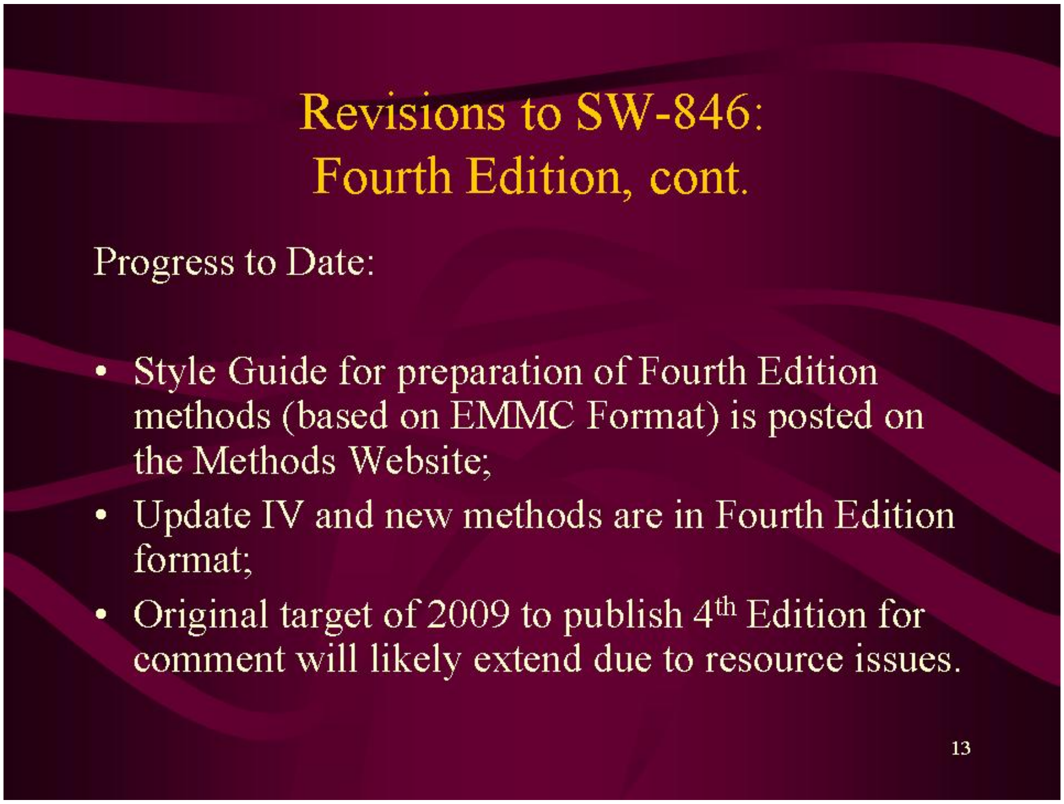
- Designed for electronic format
- Major revisions to Chapter One on QA/QC
  - Systematic planning, QA/QC and sampling for both project planners and laboratory analysts
- New and updated lab methods
- Expansion of guidance on methods selection



Revisions to SW-846:
Fourth Edition, cont.

The Scope of Effort:

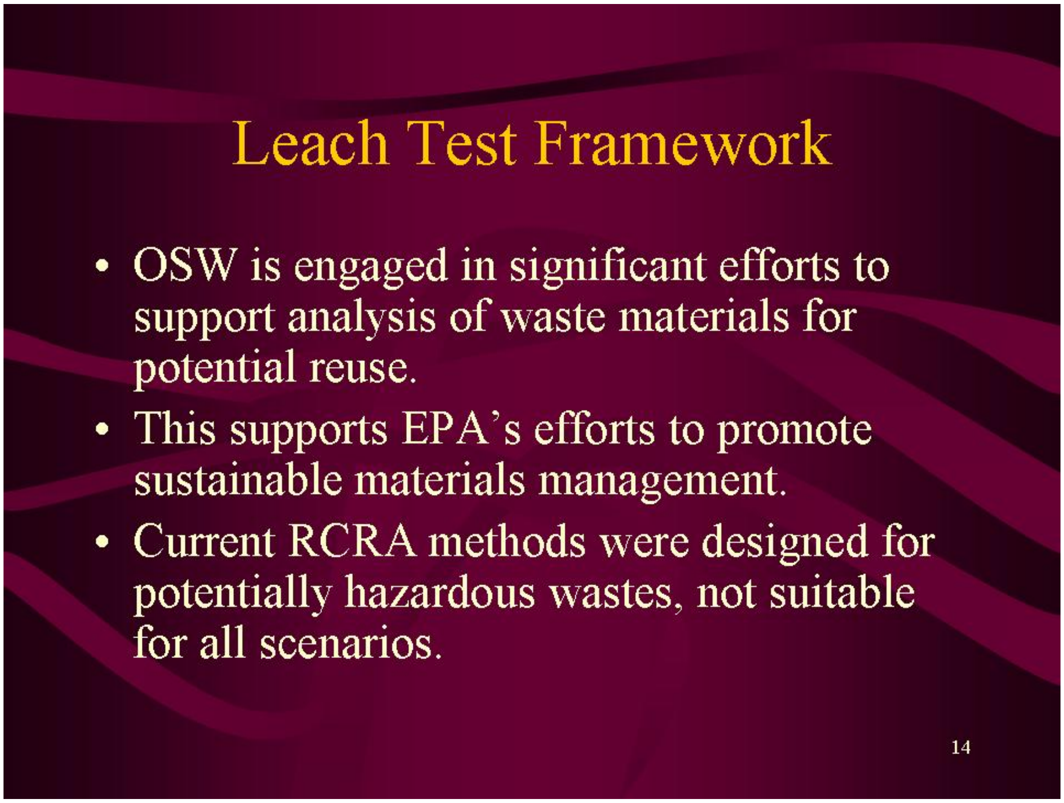
- A total of 8 chapters and 222 methods;
- The 222 methods are divided in four basic categories requiring different degrees of attention;
- 94 methods are in Categories 1 to 3 requiring low to medium effort;
- 128 methods are in Category 4, and will require significant effort to include analytical advances and new data.



Revisions to SW-846: Fourth Edition, cont.

Progress to Date:

- Style Guide for preparation of Fourth Edition methods (based on EMMC Format) is posted on the Methods Website;
- Update IV and new methods are in Fourth Edition format;
- Original target of 2009 to publish 4th Edition for comment will likely extend due to resource issues.



Leach Test Framework

- OSW is engaged in significant efforts to support analysis of waste materials for potential reuse.
- This supports EPA’s efforts to promote sustainable materials management.
- Current RCRA methods were designed for potentially hazardous wastes, not suitable for all scenarios.

Leach Test Framework: Background

- Toxicity Characteristic Leaching Procedure (TCLP) was developed to conservatively estimate leaching of hazardous constituents from a waste when it is co-disposed with municipal solid wastes (MSW).
- TCLP helps to define what is a hazardous waste.
- However, TCLP is often used when not required, or not necessarily the most appropriate test.

Leach Test Framework, cont.

- OSW has been reviewing new tests to use where TCLP is not required and MSW co-disposal conditions are not plausible, such as:
  - Industrial D Guidance
  - Industrial Materials Recycling
  - Delisting
- Key components of Framework:
  - More accurate over a range of conditions that affect leaching
  - Flexibility to apply to a range of waste types

Leach Test Framework, cont.

- A set of leach testing methods developed by Kosson, et al. (2002) address many EPA program needs:
  - Better accuracy is expected because tests consider factors known to significantly affect leaching of metals:
    - pH (solubility of many metal salts change with pH)
    - L/S ratio (or infiltration rate)
    - Form of waste (granular, compacted, or monolithic)
  - Tiered and flexible
    - Can screen using conservative and less expensive tests
    - Can test both worst-case and more realistic cases
  - Framework includes equilibrium-based tests, column test, diffusion-limited leach test

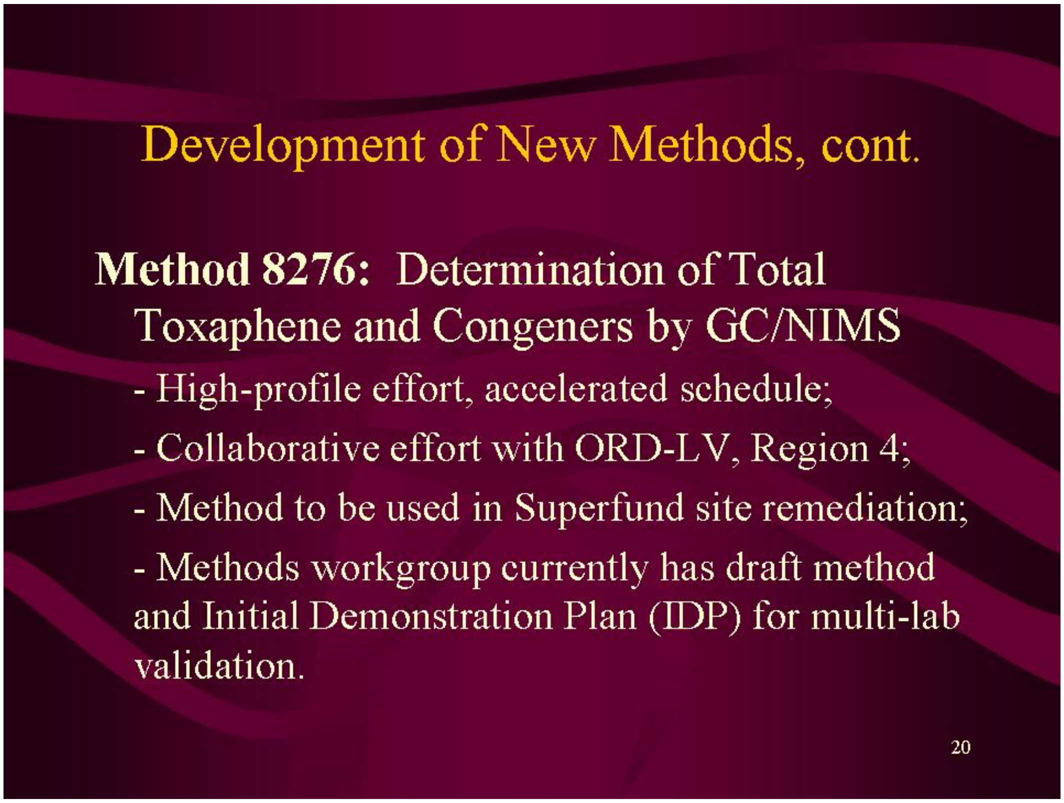
Leach Test Framework, cont.

- The testing framework supports environmental decision-making by:
  - Producing results that are more representative of actual field conditions than any single-point leach test;
  - Producing results that, combined with site conditions data, can be used in groundwater transport modeling.
- Methods in the leach test framework will be published in SW-846.
- Separate guidance will be developed to explain the relationship of various tests and how to interpret the results.

Development of New Methods

A major focus of new methods and reference standards is to support risk-based decision making in OSWER's cleanup programs, e.g.:

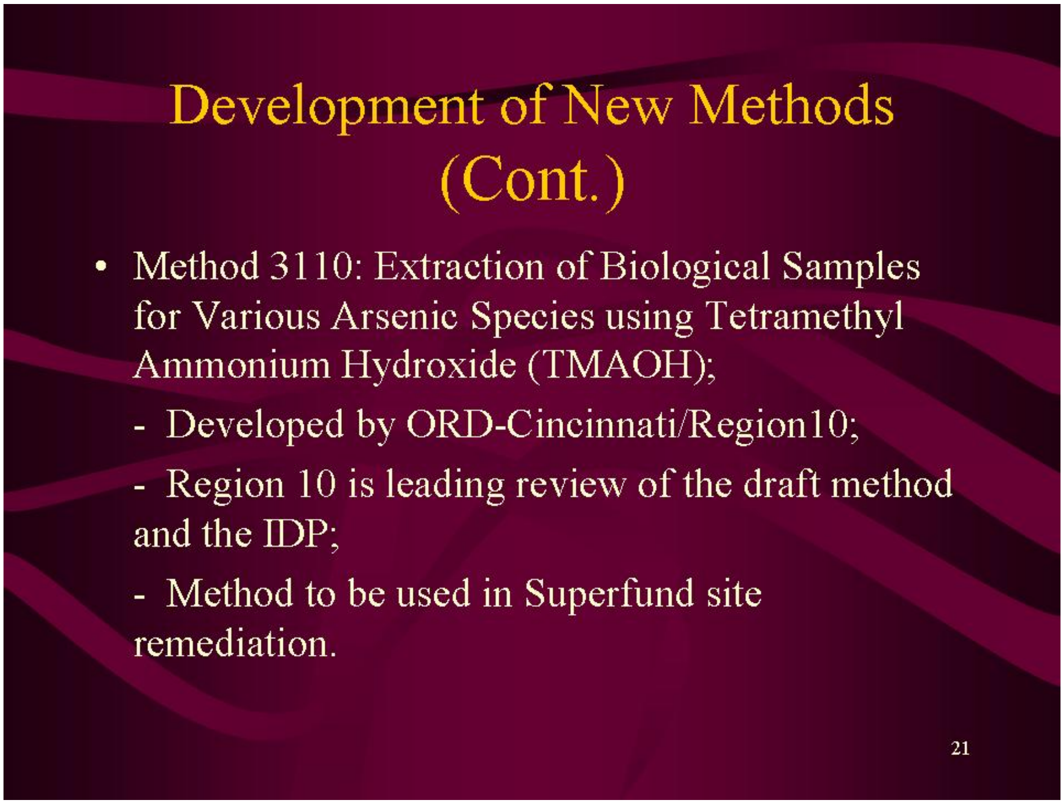
- Toxaphene and congeners
- Arsenic species
- Reference standards (an interagency effort)



Development of New Methods, cont.

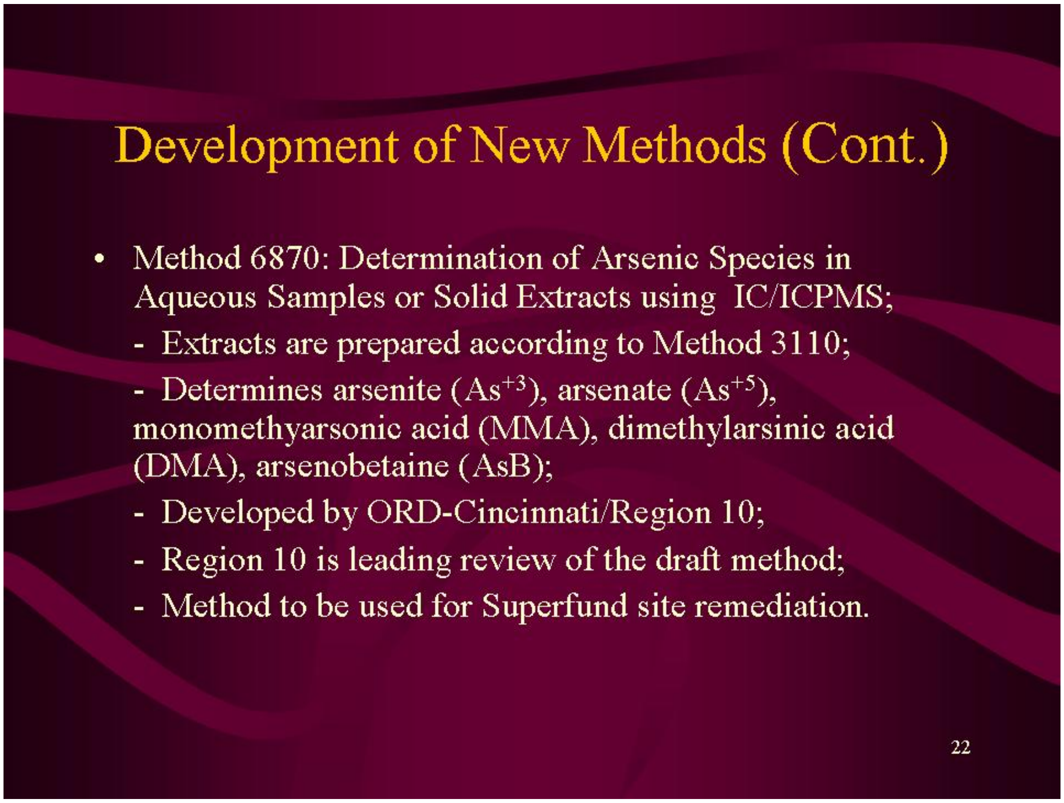
Method 8276: Determination of Total Toxaphene and Congeners by GC/NIMS

- High-profile effort, accelerated schedule;
- Collaborative effort with ORD-LV, Region 4;
- Method to be used in Superfund site remediation;
- Methods workgroup currently has draft method and Initial Demonstration Plan (IDP) for multi-lab validation.



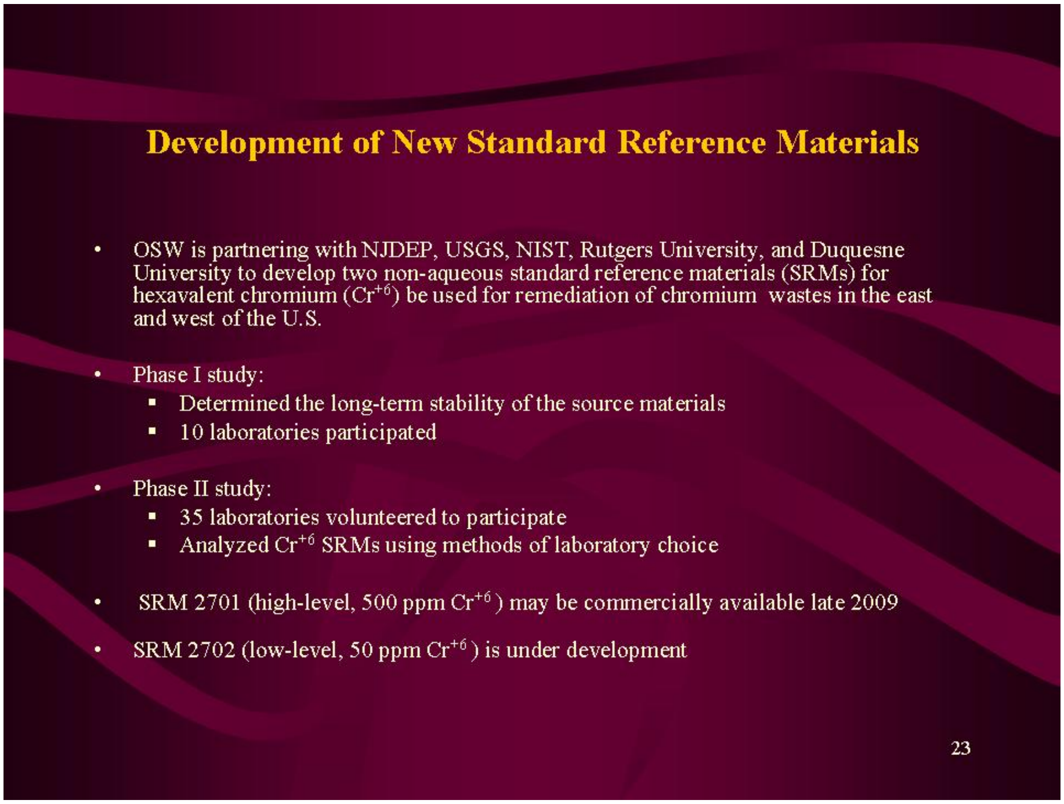
Development of New Methods (Cont.)

- Method 3110: Extraction of Biological Samples for Various Arsenic Species using Tetramethyl Ammonium Hydroxide (TMAOH);
  - Developed by ORD-Cincinnati/Region10;
  - Region 10 is leading review of the draft method and the IDP;
  - Method to be used in Superfund site remediation.



Development of New Methods (Cont.)

- Method 6870: Determination of Arsenic Species in Aqueous Samples or Solid Extracts using IC/ICPMS;
  - Extracts are prepared according to Method 3110;
  - Determines arsenite (As^{+3}), arsenate (As^{+5}), monomethyarsonic acid (MMA), dimethylarsinic acid (DMA), arsenobetaine (AsB);
  - Developed by ORD-Cincinnati/Region 10;
  - Region 10 is leading review of the draft method;
  - Method to be used for Superfund site remediation.



Development of New Standard Reference Materials

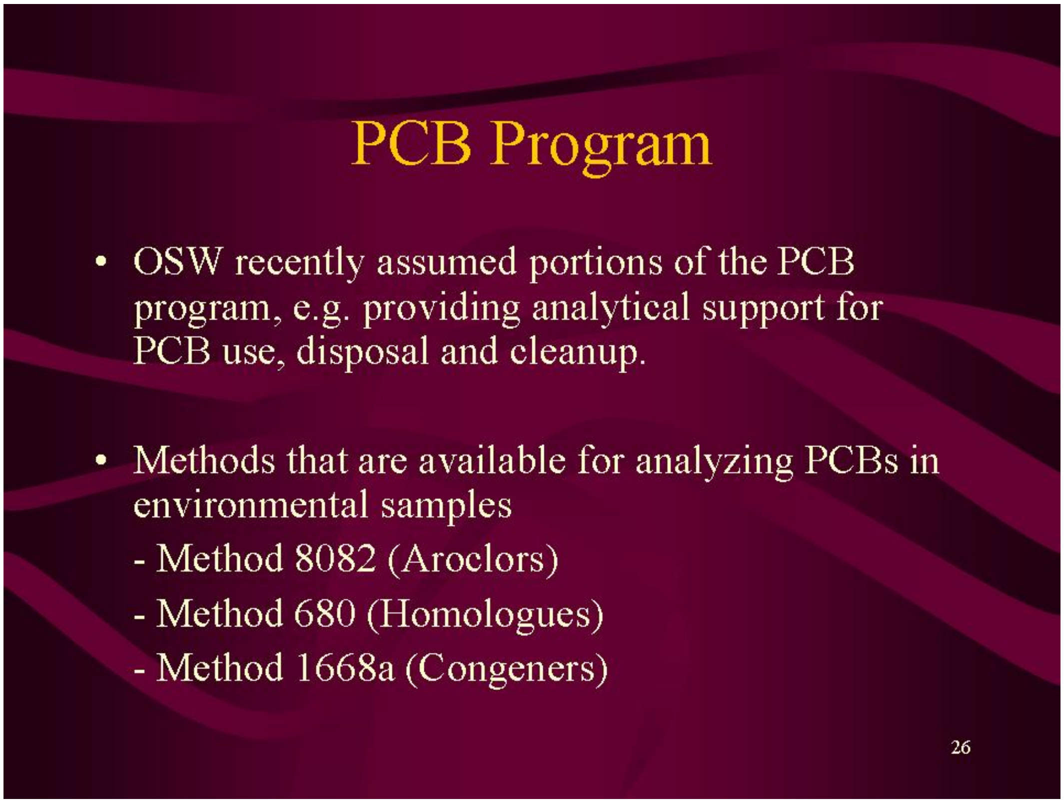
- OSW is partnering with NJDEP, USGS, NIST, Rutgers University, and Duquesne University to develop two non-aqueous standard reference materials (SRMs) for hexavalent chromium (Cr^{+6}) be used for remediation of chromium wastes in the east and west of the U.S.
- Phase I study:
  - Determined the long-term stability of the source materials
  - 10 laboratories participated
- Phase II study:
  - 35 laboratories volunteered to participate
  - Analyzed Cr^{+6} SRMs using methods of laboratory choice
- SRM 2701 (high-level, 500 ppm Cr^{+6}) may be commercially available late 2009
- SRM 2702 (low-level, 50 ppm Cr^{+6}) is under development

Overview of Related Program Activities

- Superfund Contract Lab Program Developments
- PCB Methods
- Project Collaborations on Environmental Monitoring Topics

Superfund Contract Lab Program Developments

- CLP is putting into place new inorganic lab analytical services contracts
- New contracts include Staged Electronic Data Deliverable format requirements
- SEDD is expected to provide cost savings and streamline review
- Sessions and training take place this week



PCB Program

- OSW recently assumed portions of the PCB program, e.g. providing analytical support for PCB use, disposal and cleanup.
- Methods that are available for analyzing PCBs in environmental samples
  - Method 8082 (Aroclors)
  - Method 680 (Homologues)
  - Method 1668a (Congeners)

Project Collaborations

- Detection, quantitation and calibration (Agency FEM Action Team);
- Hexavalent chromium holding time study (Duquesne University);
- Reducing use of non-fever mercury-filled thermometers in test methods and RCRA regulations (OPPT);
- Sampling strategy to detect mercury contamination during biomedical building renovation and demolition (NIH);
- Development of Standard Reference Materials for Speciated Metals (NJDEP, NIST, USGS, Duquesne University and Rutgers University)

Resources for Additional Information

- OSW Methods Homepage: *www.epa.gov/SW-846*
- Methods Information Communication Exchange (MICE)
    - Phone No.: (703)-676-4690
    - E-mail: *mice@cpmx.saic.com*

The Latest from the Office of Prevention, Pesticides, and Toxic Substances

Environmental Measurement Symposium
August 13, 2008
Betsy Grim

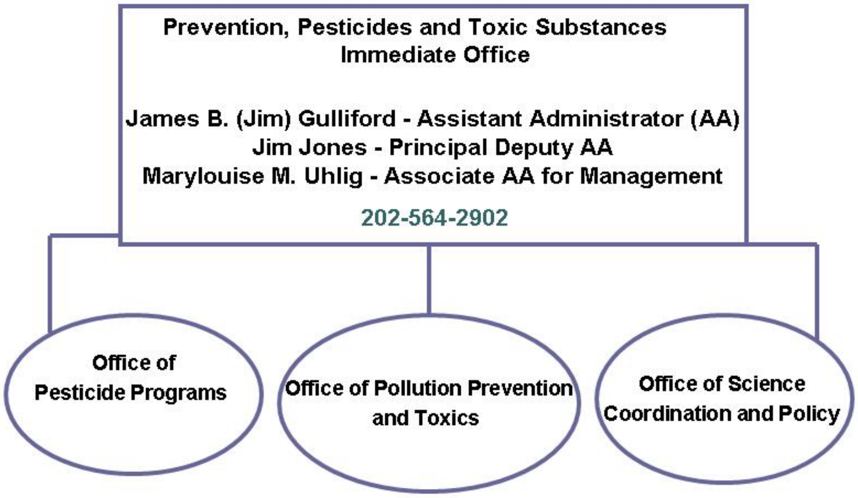
What is OPPTS?

The Office of Prevention, Pesticides, and Toxic Substances within EPA

About OPPTS

- OPPTS plays an important role in protecting public health and the environment from potential risk of pesticides and toxic chemicals
- OPPTS promotes pollution prevention through innovative partnerships and collaboration
- OPPTS evaluates pesticides to safeguard all Americans, ensures pesticides used on food are safe, and will not adversely impact the environment
- OPPTS provides chemical management by promoting the use of safer chemicals, processes, and technologies

OPPTS Organizational Structure



Prevention, Pesticides and Toxic Substances
Immediate Office

James B. (Jim) Gulliford - Assistant Administrator (AA)
Jim Jones - Principal Deputy AA
Marylouise M. Uhlig - Associate AA for Management
202-564-2902

Office of Pesticide Programs

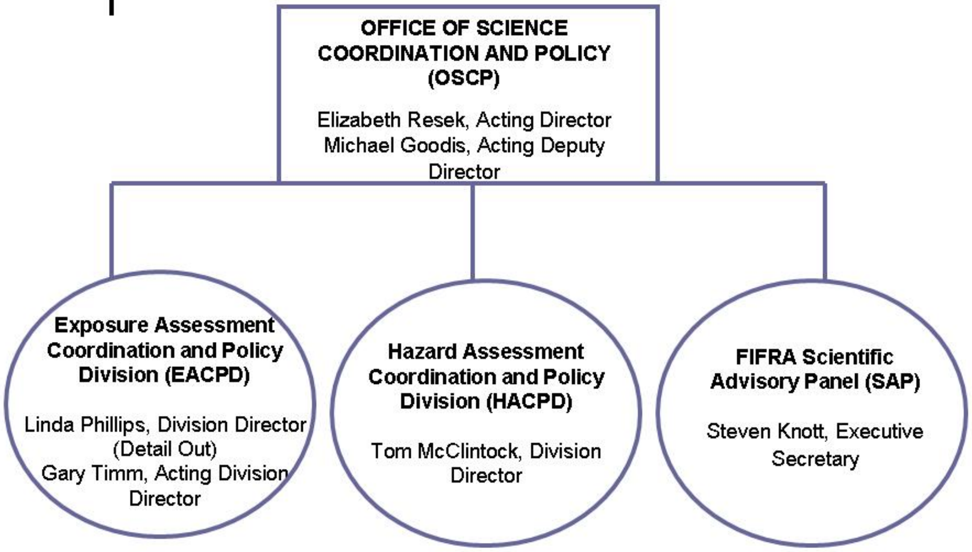
Office of Pollution Prevention and Toxics

Office of Science Coordination and Policy

What is OSCP?

The Office of Science Coordination and Policy

OSCP



OFFICE OF SCIENCE COORDINATION AND POLICY (OSCP)

Elizabeth Resek, Acting Director
Michael Goodis, Acting Deputy Director

Exposure Assessment Coordination and Policy Division (EACPD)

Linda Phillips, Division Director (Detail Out)
Gary Timm, Acting Division Director

Hazard Assessment Coordination and Policy Division (HACPD)

Tom McClintock, Division Director

FIFRA Scientific Advisory Panel (SAP)

Steven Knott, Executive Secretary

Office of Science Coordination and Policy

Responsibilities:

Provides coordination, leadership, peer review and synthesis of science and science policy within OPPTS

- Implements the Endocrine Disruptor Screening Program
- Manages the FIFRA Scientific Advisory Panel that serves as the primary external scientific peer review mechanism for OPPTS
- Coordinates emerging exposure and hazard assessment topics (e.g., Biotechnology)

Endocrine Disruptor Screening Program (EDSP)

Background:

- **Food Quality Protection Act (FQPA)**

Requires EPA to: Develop a screening program using validated assays to identify pesticides that may have estrogenic effects in humans.

Authorizes EPA to include:
Other endocrine effects, as designated by the EPA Administrator (e.g., androgen and thyroid; endocrine effects in species other than humans).
Other non-pesticide chemicals that:

- **Have "an effect cumulative to that of a pesticide," and**
- **To which a substantial human population may be exposed.**

- **Safe Drinking Water Act (SDWA) Amendments**

Allow EPA to require testing of chemical substances found in sources of drinking water, if a substantial human population may be exposed.

Endocrine Disruptor Screening Program (EDSP)

EPA plans to issue first Test Orders for Tier 1 Screening under the EDSP in August 2008

- Validation has been completed for 10 of 11 Tier 1 Battery assays; validation of remaining Tier 1 assay to be completed early 2009.
- Issuance of Test Orders expected upon OMB review of the Information Collection Request and the Policy and Procedures for screening.
- Endocrine Disruptor Screening and Testing Committee EDSTAC

What is OPPT?

The Office of Pollution Prevention and Toxics

OPPT-The Facts

General Budget: $96. 2 Mill.

$423.7 Mill. (FTE)

Key Tasks:

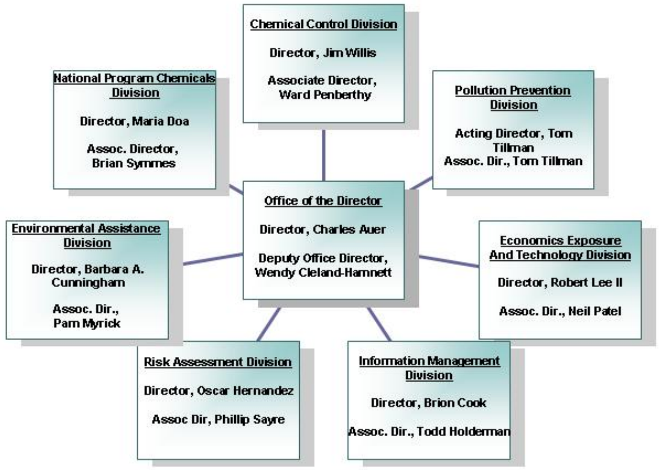
-Manages programs under the Toxic Substances Control Act and the Pollution Prevention Act of 1990

-Evaluates new and existing chemicals and their risks

-Finds ways to prevent or reduce pollution before it gets into the environment

-Manages a variety of environmental stewardship programs encouraging companies to reduce and prevent pollution

OPPT



Chemical Control Division
Director, Jim Willis
Associate Director, Ward Penberthy

National Program Chemicals Division
Director, Maria Doa
Assoc. Director, Brian Symmes

Pollution Prevention Division
Acting Director, Tom Tillman
Assoc. Dir., Tom Tillman

Office of the Director
Director, Charles Auer
Deputy Office Director, Wendy Cleland-Hamnett

Environmental Assistance Division
Director, Barbara A. Cunningham
Assoc. Dir., Pam Myrick

Economics Exposure And Technology Division
Director, Robert Lee II
Assoc. Dir., Neil Patel

Risk Assessment Division
Director, Oscar Hernandez
Assoc Dir, Phillip Sayre

Information Management Division
Director, Brion Cook
Assoc. Dir., Todd Holderman

OPPT Recent Hot Topics

- Lead in Toy Jewelry
- Lead based paint
- Mercury (Rebecca Woods-August 14th)
- Asbestos
- New efforts on chemicals management (ChAMP)

Visit http://www.epa.gov/oppt/ to read more about these topics

What is OPP?

The Office of Pesticide Programs

Pesticide Factoids

- **OPP is one of the largest programs at USEPA HQ**
    - About 900 staff in nine divisions - highly technical (includes toxicologists, chemists, economists)
- **About 85 FTEs across 10 Regional Offices work with states and tribes to implement the pesticide program**

OPP-The Facts

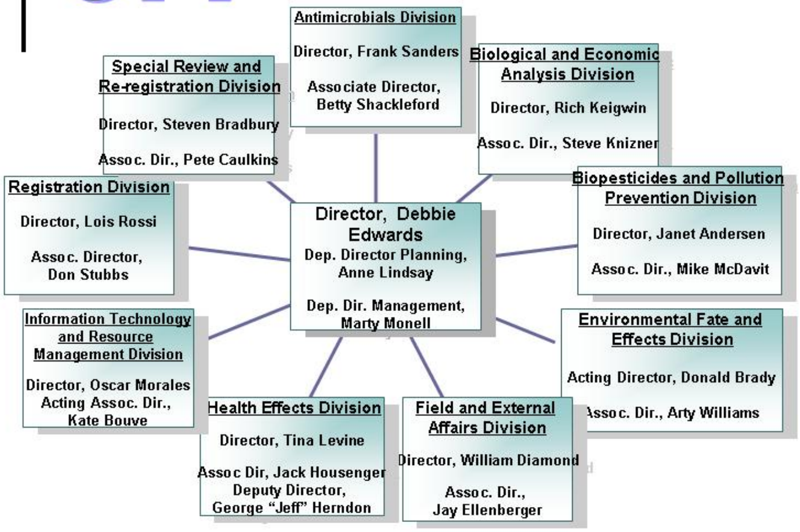
General Budget: $133.2 Mill.
$868.6 Mill.(FTE)

Key Tasks:

-OPP, works with ten regional offices and other EPA program offices on a wide range of pesticide issues and topics, such as:

Evaluating potential new pesticides and uses
-Providing for local needs and emergency situations
-Reviewing safety of older pesticides
-Enforcing pesticide requirements
-Regulating products of biotechnology
-Speeding the review of safer pesticides

OPP



Director, Debbie Edwards
Dep. Director Planning, Anne Lindsay
Dep. Dir. Management, Marty Monell

Antimicrobials Division
Director, Frank Sanders
Associate Director, Betty Shackleford

Biological and Economic Analysis Division
Director, Rich Keigwin
Assoc. Dir., Steve Knizner

Biopesticides and Pollution Prevention Division
Director, Janet Andersen
Assoc. Dir., Mike McDavit

Environmental Fate and Effects Division
Acting Director, Donald Brady
Assoc. Dir., Arty Williams

Field and External Affairs Division
Director, William Diamond
Assoc. Dir., Jay Ellenberger

Health Effects Division
Director, Tina Levine
Assoc Dir, Jack Housenger
Deputy Director, George “Jeff” Herndon

Information Technology and Resource Management Division
Director, Oscar Morales
Acting Assoc. Dir., Kate Bouve

Registration Division
Director, Lois Rossi
Assoc. Director, Don Stubbs

Special Review and Re-registration Division
Director, Steven Bradbury
Assoc. Dir., Pete Caulkins

OPP Recent Hot Topics

- Nanotechnology
- Endangered Species
- Rodenticide

Visit http://www.epa.gov/pesticides/ to learn more about these topics

The OPP Laboratories

Environmental Science Center
Fort Meade, Maryland

- Microarray Research
- Microbiology Laboratory Branch
- Analytical Chemistry Branch

Stennis Space Center
Bay St. Louis, MS

- Environmental Chemistry Branch

OPP's Laboratories

- Microarray Research- Integrates recent advances in the field of genomics into current microbiological techniques for testing the effectiveness of antimicrobial agents
- Microbiology Laboratory Branch –Develops new antimicrobial methods and conducts efficacy testing
- Environmental Chemistry Branch-Reviews and develops new methods for pesticides and persistent organic pollutants
- Analytical Chemistry Branch-Validates tolerance analytical enforcement methods, develops new analytical methods and operates the EPA National Pesticide Standard Repository

The Latest!

- Demonstrating Flexible Approaches to Environmental Measurement
- Demonstrating Laboratory Competency
- Homeland Security
- USEPA National Pesticide Standard Repository
- Environmental Management System

The Latest!

Demonstrating Flexible Approaches to Environmental Measurement-The Evolution of the Performance Approach-attend 4:15 session today to learn more.

Demonstrating Flexible Approaches to Environmental Measurement

- OPP has adopted and fully supports the performance approach for submission of methods by registrants.
- Registrants develop methods
- OPP sets criteria (precision, accuracy etc.)
- OPP Guidelines provide framework

Demonstrating Flexible Approaches to Environmental Measurement

Analytical Chemistry Branch and State partners conduct formulation chemistry analyses to support the Antimicrobial Testing Program

OECA has required analysis using the exact method submitted by registrant

OECA and OPP have agreed to use established methods for Quaternary Ammonium active ingredients.

Working on Phenols and other major active ingredients

(increased emphasis on flexibility in choosing sampling and analytical approaches)

Demonstrating Flexible Approaches to Environmental Measurement

- Participating in international inter-calibration programs for dioxins/furans (increased collaboration)
- Evaluating “universal” quantitative methods – efficacy of antimicrobials (rapid assessment of new methods)

Challenges

- **OPP will have to continue to work with the registrants to update their methods using new technology when applicable**

The Latest!

- Demonstrating Laboratory Competency

Demonstrating Laboratory Competency

- FEM Laboratory Competency Policy
  Assuring the Competency of Environmental Protection Agency Laboratories issued February 23, 2004
    - Agency Laboratories required to:
    - **I.** Maintain a **documented Quality System** that complies with the EPA Quality System EPA Order 5360.1 (now CIO 2105.0) and contains specific components
    - **II.** Participate in **inter-laboratory comparisons**
    - **III.** In the absence of accreditation programs, **periodic assessment by qualified independent assessors** at least once every three years
    - **IV.** Seek **accreditation** where appropriate accreditation programs are available

Advantages of Accreditation

- Complies with Agency's Laboratory Competency Policy
- ERLN strongly prefers accreditation
- Provides National and International credentials
- Provides recognition of scope of laboratory's qualifications and activities
- Removes barriers to participation in projects that require accreditation
- Increases credibility of laboratory's products

Current Activities

- **Workgroup meeting every two weeks**
- **Each lab addressing own GAP findings**
- **Labs will exchange information at meetings**
- **Have assembled several sources of information: NEIC, PDP, FDA, Each lab's documents**
- **Will classify findings:**
  - **1. Deficiencies that a single lab can address by itself,**
  - **2. Deficiencies where all labs need to have the same response,**
  - **3. Deficiencies that need to be elevated such as a change in Division or Office QMP and the level of management review**

Examples of responses to Gap analysis findings

- **Upgrade Quality Management Plans**
- **Upgrade or Develop Processes and Procedures for:**
  - **Internal audits**
  - **Corrective actions**
  - **Training**
  - **Demonstration of Laboratory and Staff Capability**
  - **Measurement Uncertainty**
  - **Customer Service/Client Review**
  - **Management Review**
  - **Electronic Data Management**

Next Steps, Questions and Issues

- **Approach**
  - Accreditation
  - Assessment
  - Accreditation and Assessment.
- **Scope of Accreditation**
  - Comprehensive scope of Accreditation
  - Limited accreditation scope with OEI consensus
- **Accreditation Body Selection**

- **Establish a schedule**

The Latest!

- Homeland Security

Homeland Security

- All OPP labs members of OSWER/OEM ERLN

  Provide laboratory surge capacity in event of emergency

- OPP Member of EPA Homeland Security Lab Response Workgroup

  Standard Analytical Methods (SAM) Workgroup

  LC/MS/MS CWA degradation products method trial

- Microbiology Laboratory

  efficacy test method development

  SAP endorsement of quantitative efficacy test method for *Bacillus anthracis*

The Latest!

- US EPA National Pesticide Standard Repository

US EPA National Pesticide Standard Repository

- NPSR processed over 6000 orders of analytical grade standards to be used by state, federal, or tribal labs for enforcement in 2005.
- An email order form is available www.epa.gov/oppbead1/labs/analyticalchem_lab.htm.
- A return receipt is sent so that those ordering online from NPSR can be assured their requests are received.

EPA National Pesticide Standard Repository

- What types of pesticide standards are in the Repository?
  - Technical grade active ingredient (TGAI)
  - Analytical grade
    - Active ingredient
    - Metabolites listed in the tolerance expression
    - Degradates of “toxicological significance”
    - Any standard required for the enforcement method, but not commercially available

EPA National Pesticide Standard Repository

What types of chemicals are kept in the Repository?

- Pesticides with the following:
  - Current U.S. registration and tolerance
  - Import tolerance but no U.S. registration
  - Prior U.S. registration and/or tolerance (limited availability)
- NO industrial chemicals
- NO reagents that are commercially available
- NO inerts

EPA National Pesticide Standard Repository

How do we get standards?

- OPPTS Test Guidelines 830.1900—Registrants must submit standards as a condition of registration
  - ~5 g analytical grade of each active ingredient
  - ~200 g technical grade of each active ingredient
- When specifically requested by EPA
  - ~200 g proposed product
  - Inerts, impurities, degradation products, or metabolites

EPA National Pesticide Standard Repository

Who is eligible to receive standards?

- Federal labs enforcing pesticide laws (FIFRA, FFDCA, etc.)
- State labs enforcing pesticide laws

The Latest!

EMS

- Environmental Management Systems
- Linked to the Environmental Science Center Policy Statement

ISO 14001 EMS Registration Awarded - October 23, 2002

ESC WAS THE FIRST OF 32 REPORTING EPA FACILITIES!

SOME EMS Results Include:

- Reduced paper copies by 24%.
- Increased paper recycling by 5%.
- Reduced electricity consumption 17%.
- Reduced water consumption by 37%.
- Trained other federal agencies to develop their own EMS.
- created a CHEMICAL ADOPTION PROGRAM for local schools.
- Using 100% recycled-content process chlorine-free copy paper.

EMS website:

Environmental Science Center

Serving Delaware, District of Columbia, Maryland, Pennsylvania, Virginia, and West Virginia

You are here: EPA Home » Environmental Science Center » EMS

Environmental Management System

Man preventing woman from placing paper in t... to full size view

The EPA's Environmental Science Center (ESC) at Ft. Meade, Md. is a state-of-the-art laboratory facility occupied by employees who care deeply about the environment. To help ensure that the ESC is a good neighbor in the Ft. Meade community, we have implemented an Environmental Management System (EMS) that conforms to ISO 14001:2004, the international standard for EMS. We believe that an EMS, such as the one prescribed in ISO14001, provides a management scheme that facilitates incorporating environmental consideration into our daily business activities. The EMS approach fosters compliance with legal requirements, as well as thinking beyond compliance to improve environmental performance for impacts to the environment that are not regulated. Our EMS Policy was written to show how committed the ESC facility is to this approach and to making EMS a part of our daily business.

EMS Tool Box

- About PDF Files
- EMS Manual-PDF [20pp, 134k]
- Aspects
- Procedures
- Tip Sheets/Other Info.
- EMS Refresher Course (PDF) [53pp, 1M]
- Accomplishments
- Presentations
- Mid-Atlantic EMS

We would like to share our EMS tools and products with any interested organization that is implementing an EMS or looking for new ideas. This web page is a collection of documents/links that may prove helpful to others working on their own EMS. Please browse this page for relevant information and contact us if you

ESC Staff only: SUGGESTIONS

If you have any suggestions or comments, please submit them to

Artwork Contest held on Earth Day 2006 for an EMS LOGO. Team Member Signs were made using the LOGO and placed outside each member's cubicle for recognition.

EMS Team Member

Golden Groundhog – created our EMS mascot

Golden Groundhog Awards:
given by the EMS Team to good
environmental stewards at ESC

Golden Groundhog Day:
February 12, 2004 to Promote EMS Awareness

ECO TIP of the month award: parking spot of choice
and free wind-up flashlight for winner!

An on-line
EMS REFRESHER TRAINING
was created with help from the University
of Maryland Capstone Project.

Environmental Management System

Environmental
Science Center
An ISO 14001:2004
certified facility

Refresher Training
(updated 2007)

Created rain garden.

EARTH DAY 2006

QUESTIONS?

2008 NEMC Proceedings

POSTERS

Development and Validation of Biothreat Sample Analyses Methods in Support of the Environmental Response Laboratory Network (ERLN)

Sanjiv Shah
USEPA Homeland Security
26 W. Martin Luther King Dr.
Cincinnati, OH 45268
513-569-7776
shah.sanjiv@epa.gov

ABSTRACT

One of the missions of the Response Capability Enhancement Team (RCE) within the EPA's National Homeland Security Research Center (NHSRC) is to provide technical support to the Environmental Response Laboratory Network (ERLN) by developing standard analytical methods for environmental samples that may contain terrorism agents of a biological, chemical, or radiological nature. These methods are intended to be used during the restoration of an area following a terrorist act or other incident. A compendium of Standardized Analytical Methods (SAM) has been published and is updated on an annual basis. Standard Analytical Procedure (SAP) documents that specify sample preparation and analyses requirements for individual biothreat agents are being prepared and proceeding toward verification. Similarly, Sample Collection Procedure (SCP) documents for biothreat agents to include spores, vegetative pathogens, viruses, and biological toxins have been prepared. Efforts are underway for a single laboratory verification and multi laboratory validation of these methods. New and improved methods for sample collection, concentration, processing, and analysis are also being developed. The current status of these efforts will be presented and the framework for collaborative work with other internal/external organizations will be discussed.

Rapid Analyte Verification and Quantitation Using Deconvoluted Full Scan GC/MS Data

Mike Szelewski
Agilent Technologies
2850 Centerville Rd.
Wilmington, DE 19808
302-633-8275
mike_szelewski@agilent.com

ABSTRACT

Verification and quantitation of analytes at low concentration levels in environmental samples is challenging due to matrix interferences. Large numbers of false positives and false negatives are reported due to incorrect ion ratios when comparing data from a few ions. The concentration of the identified compounds is subject to errors when integrating areas of peaks unresolved from the background.

Deconvolution of full scan data allows the analyst to compare "clean spectra" to that found in mass spectral libraries. The deconvolution is accomplished using AMDIS (Automated Mass spectral Deconvolution and Identification Software). AMDIS also provides deconvoluted EICs (Extracted Ion Currents) which are used directly for quantitation in the MSD Chemstation software. A significant time savings is realized in data reduction. The analyst is presented with a spectral match that is matrix free, and peak integration is less subjective with improved peak resolution. The AMDIS process of deconvolution and identification takes less than two minutes and is more reproducible than manual interpretation, which could take twenty minutes. The AMDIS and Chemstation processes have been seamlessly combined to eliminate the need for analyst expertise in multiple software packages.

Libraries for of all single component analytes found in USEPA Methods 8270 and 525 have been built specifically for use within AMDIS. Each library entry also includes a precisely locked retention time that will differentiate among compounds of similar spectra.

Data from spiked sample extracts containing large interferences will be presented. The number and quality of identified analytes using deconvoluted full spectrum data will be compared to the that found using traditional techniques with matrix interference.

The Science of Solid Phase Extraction

Don Shelly
UCT
MCIDC Plaza Bldg 10
Lewistown, PA 17044
717-247-0896
dshelly@unitedchem.com

ABSTRACT

Solid phase extraction (SPE) is often over looked as a method of choice for extracting both priority pollutants and emerging pollutants. This is in part due to the misconception that SPE is a type of liquid chromatography. Sometimes referred to as digital or start-stop chromatography, SPE does not employ theoretical plates.

Early SPE consisted of what we now refer to as dispersive SPE. Sorbent was added to centrifuge tubes and aqueous sample was added to the tubes and shaken. The sorbent was then filtered out of the tubes, washed, and the analytes of interest eluted off for analysis. Later, sorbent was added to plastic syringe barrels and held in place with frits. The frits acted to hold the sorbent in the tube during shipping and did double duty as a prefilter. Now, SPE has evolved further to include both polymeric and silica based sorbents with silica remaining the dominant type. Also, many different functionalities are now available including, reverse phase, normal phase, ion exchange and mixed modal copolymeric.

SPE can be fun and easy when a few basic principles are understood. These principles include sorbent selection, carbon hydrogen interaction, polarity, positive and negative attraction, pH and pKa.

Techniques for Reducing Purge-and-Trap (P&T) Cycle Times in VOC Analysis

Moklesur Rahman
OI Analytical
151 Graham Road
College Station, TX 77842
979-690-1711 ext. 236
mrahman@oico.com

ABSTRACT

The USEPA Office of Ground Water and Drinking Water Technical Support Center has recently reported on its efforts to update and revise Method 524.2 for Determination of Volatile Organic Compounds (VOCs) in Drinking Water. VOCs in soils, surface water, groundwater, and drinking water are usually extracted by purge-and-trap (P&T) methods and analyzed by gas chromatography-mass spectrometry (GC-MS). One of the toughest challenges of the P&T method is decreasing the total cycle time of the instrumentation. The longest sample-processing step prescribed in USEPA methods is the 11-minute purge step. By maintaining a sample temperature of 40 °C the purge time can be reduced to 7 minutes without compromising analytical performance.

The steps in the P&T cycle consist of sample transfer, internal standard addition, purging the sample, desorbtion, and reconditioning the trap. The typical cycle can take as long as 25–30 minutes including the P&T, autosampler, and GC cycles. Shortening the cycle time without compromising method performance is a high priority for production laboratories needing to optimize productivity and profitability.

This poster will confirm the purge time in a VOC analysis can be reduced from 11 to 7 minutes when sample temperature is maintained at 40 °C. A study of relative purge efficiencies was conducted using 11- and 7-minute purge times with sample at 40 °C. The results were compared to purge efficiencies using the traditional 11-minute purge and ambient temperature. The experimental results indicate heating the sample during the purge step is essential if purge time is to be reduced. A sample temperature of 40 to 45 ° C improves purge efficiencies and reduces purge time. Full method performance is shown. These findings are consistent with results reported by the USEPA indicating a 7-minute purge time is sufficient for VOC methods.

Benzene in Soft Drinks by Teledyne Tekmar HT3 Headspace Sampler

Teri Dattilio
Teledyne/Tekmar
4736 Socialville Foster Road
Mason, OH 45040
513-229-7215
teri_dattilio@teledyne.com

ABSTRACT

This poster presents a method that demonstrates the ability of the HT3 Headspace autosampler to analyze Benzene in Soft drinks at low detection levels. Benzene, a known carcinogen, is naturally occurring (for example, in crude oil) but is also manufactured for use in the industrial sector. Benzene is released to the air from various sources, including industrial emissions, fires, vehicle exhaust and tobacco smoke. Because benzene readily evaporates into the air from gasoline, gas stations are also a source of benzene to the air.

In the early 1990s, it was found that benzene could be formed in soft drinks containing certain food preservatives and nutrient additives. It was shown that benzoate salts, used as an anti-microbial agent in certain soft drink products, could react with ascorbic acid (vitamin C) to form benzene, especially in the presence of light and elevated temperatures. Ascorbic acid may be either naturally present from a fruit juice ingredient in the soft drink or added as an antioxidant food additive. In November 2005 the FDA received private laboratory results reporting low levels of benzene in a small number of soft drinks that contained benzoate salts and ascorbic acid. FDA has no regulatory limits for benzene in beverages other than bottled water, for which FDA uses the US Environmental Protection Agency (EPA) maximum contaminant level (MCL) of 5 ppb for drinking water, as a quality standard. This method could be used by quality control to determine whether soft drinks contain benzene above the U.S.E.P.A. MCL of 5 ug/L (ppb) or the Uk s MCL of 1 ug/L (ppb). Soda beverages were analyzed using a Teledyne Tekmar HT3 with a Gas Chromatograph equipped with a Mass Spectrometer. A 5-point calibration curve for Benzene was first analyzed in water.

Rapid Dual GC Column Analysis of Pesticides

Jason Thomas
Retek Corporation
110 Benner Circle
Bellefonte, PA 16827
814-353-1300
jason.thomas@restek.com

ABSTRACT

Analysis of organ chlorine pesticides, PCB's, and peony-acid herbicides has become a routine assay in the environmental sector. Although production and application of many of these agents were phased out decades ago, they can still be found in the environment. Environmental laboratories are expected to rapidly analyze extracts of samples with very complicated sample matrices without sacrificing target compound identification. These tests require a gas chromatographic stationary phase with proper selectivity and high thermal stability. There is also a constant desire for ever faster and faster analysis times, to help increase sample throughput and thereby increase laboratory productivity.

The dual column/ECD configuration allows simultaneous determination and confirmation of monitored compounds, while taking advantage of the greater sensitivity, toward halogenated species, of the ECD with respect to GC/MS. Differing selectivity, and the stable nature of the two stationary phases, enables a wide variety of pesticides to be analyzed with this system. A single instrument can be used for several methods, eliminating the need to change columns or dedicate multiple instruments to specific analyses.

As an alternative to relying on the high efficiency of narrow bore columns for fast separations, a column stationary phase with a specially tuned selectivity for a particular group of compounds can be used to effect a rapid separation. This poster will focus on the use of two such unique stationary phases, in a standard 0.32mm ID, to perform a variety of EPA methods, including 8081, 8151, 8082, and others. In addition to this conventional column configuration, a look at a more modern technique for greatly improved runtimes will be examined for the EPA Method 8081 target compound list.

Analysis of Volatile Organic Compounds in Drinking Water to Meet Newly Enacted Regulations in China

Moklesur Rahman
OI Analytical
151 Graham Road
College Station, TX 77842
979-690-1711 ext. 236
mrahman@oico.com

ABSTRACT

China has enacted 71 new rules to regulate their drinking water quality; included on the list of regulated contaminants are 23 volatile organic compounds (VOCs) plus their isomers, for a total of 29 VOCs. Some of the contaminants on this list are halogen-containing VOCs, and others contain at least one degree of unsaturation. A third group contains both aromatic properties and at least one halogen heteroatom. The unique characteristics of these components make them excellent candidates for extraction by purge-and-trap (P&T) followed by analysis using a tandem photoionization detector (PID) interfaced with an electrolytic conductivity detector (ELCD).

In 1986, the United Stated Environmental Protection Agency (USEPA) introduced Method 502.2 to monitor the levels of aromatic and halogenated VOCs in drinking water using P&T with PID/ELCD tandem selective detectors. Twenty-eight of the 29 compounds in the Chinese drinking water regulation are included in this commonly used method and can be simultaneously analyzed at the required detection limits in a single analysis.

This poster will demonstrate that the USEPA Method 502.2 can easily be adapted to detect and quantify all 29 VOCs listed in the new Chinese Tap water regulation in a single analysis. The equipment configuration and operating conditions of P&T system with a tandem PID/ELCD detector for the analysis of VOCs in the Chinese drinking water regulation will be described. Data including the retention times, calibration information, limits of detection and quantification, and applications will also be presented.

Evaluation of New Moisture Management Techniques for the Analysis of Volatile Organic Compounds Utilizing the StratUm PTC

Teri Dattilio
Teledyne/Tekmar
4736 Socialville Foster Road
Mason, OH 45040
513-229-7215
teri_dattilio@teledyne.com

ABSTRACT

Water interference is a common problem in purge and trap analysis. When purging aqueous samples, water vapor is transferred with the analytes of interest onto the adsorbent trap and subsequently into the gas chromatograph (GC). In GC/MS this water vapor can cause inconsistent vacuum pressures and reduced ionization resulting in loss in stability of response over time. Traditionally, water vapor interference has been controlled by utilizing hydrophobic adsorbents in the trap and passing an inert, moisture free gas through the adsorbent prior to desorption of the trap to the gas chromatograph. Depending on the detector and parameters this "dry purge" may not be completely effective in eliminating the interference. This poster evaluates a revolutionary new way to remove water vapor after the concentration step in purge and trap analysis.

Analysis of Volatile Organic Compounds Using New Products and Novel Techniques

Jason Thomas
Restek Corporation
110 Benner Circle
Bellefonte, PA 16827
814-353-1300
jason.thomas@restek.com

ABSTRACT

In anticipation of the imminent promulgation of a revised method for analysis of volatile organic compounds in drinking water, EPA Method 524.3, it was felt that an evaluation of a new volatiles column, as well as, a few alternative approaches, for use with the newly revised method would be a good way to test the performance of these new materials.

Due to the rigorous constraints emplaced upon a capillary column for use with this method including, but not limited to, excellent inertness, appropriate selectivity, and low bleed, a new column has been designed expressly for VOC analysis. This new column is designed to be extremely inert, possess optimized selectivity, and exhibit ultra-low bleed. This should make it quite amenable for use in analysis of a complex list of low concentration compounds such as that found in EPA Method 524.3.

As the degree of scarcity of helium supplies increases, with a resulting concomitant rise in helium prices, many labs are considering converting to hydrogen carrier and alternative purge gases as a way to defray some of the increasing costs of laboratory operation. The efficacy of using hydrogen as a carrier gas, combined with nitrogen as a purge gas will be explored for the analysis of EPA Method 524.3.

Groundwater Plume, Source and Risk Identification Using Passive Soil Gas

Harry O'Neill; Beacon Environmental Services, 323 Williams St., Bel Air, MD 21014; 410-838-8780; harry.oneill@beacon-usa.com
Joseph E. Odencrantz, Ph.D.; Beacon Environmental Services, Newport Beach, CA; joe.odencrantz@beacon-usa.com

ABSTRACT

The results from sixty passive soil gas (PSG) samplers placed at a grain silo and mixed-use industrial facility with a known chlorinated solvent release aided with the rapid investigation time-schedule. The samplers were used to determine potential sources, plume extent and vapor risks in areas beyond the previous discrete soil sampling and permanent groundwater monitoring well network. Passive soil gas testing involves the installation of sorbents in a one-inch diameter hole, three-foot deep and the samplers are then placed at depth of approximately six inches. The samplers were installed in grid pattern at a spacing of 25 to 50 feet at the site and across the street. The depth to groundwater was approximately ten to twelve feet below grade.

Analyses of the high-resolution PSG samplers were performed following EPA Method 8260 with results based on an initial five-point calibration. Internal standards and surrogates were used, per the method, for accurate quantification of the compounds. This level of QA/QC for the analysis of passive soil gas samples, which is unique to the industry, allows for excellent correlation between PSG measurements and groundwater concentrations.

The installation of numerous monitoring wells in areas where TCE and PCE were discovered using the PSG samplers revealed that there were indeed two separate sources and groundwater plumes at the site which became co-mingled downgradient. In this previously uninvestigated portion of the site, TCE found at 600 ng in a PSG sampler and a permanent groundwater monitoring well was subsequently installed in that vicinity. The groundwater concentration of TCE in that well was 400 ug/L. The PSG sampling network was wide enough to identify the approximately size of this new plume. In a region approximately 75 feet downgradient from the previously discussed location, 294 ng of TCE was discovered in a PSG sampler. The groundwater monitoring well installed at this location revealed 110 ug/L of TCE. Further yet, a cross-gradient PSG sampler revealed 107 ng of TCE and the groundwater monitoring well placed at the location revealed 74 ug/L of TCE. The PSG investigation was completed from start to finish in a two-week period of time. The paper will focus on the importance of PSG sampling for source identification, groundwater investigation and complying with regulatory directives for the assessment of the vapor intrusion pathway for risk assessment purposes.

Transportable Universal Detector System for Chemical and Biological Agents: A New "Biochemimarker" Analysis Platform

Matt Pamuku; Applied Isotope Technologies, Inc., 851 Stella Court, Sunnyvale, CA 94087; 408-472-2333; matt@sidms.com
H.M. 'Skip" Kingston, Mizanur Rahman, Greg Zinn, and Timothy Fahrenholtz; Duquesne University, Pittsburgh, PA

ABSTRACT

After 9/11, there has been significant spending to develop detectors for chemical and biological warfare agents. To date, most detectors that were introduced have been capable of detecting a limited number of fugitive agents, often with high false positives. In other cases, the menu of detectable agents has been severely limited because of the inherent technical limitations, making these detectors only useful for a small class of agents. We report here that an "Integrated Instrument-Method System (IIMS)" is being developed as an automated, universal chemical and biological detector, with funding from the Air Force Surgeon General's modernization program. The IIMS is an automated, advanced detector system that utilizes a miniaturized mass spectrometer detector and proven measurement techniques based on innovative and definitive methods that deliver unprecedented accuracy and analytical flexibility.

After successfully completing the proof-of-concept steps early in April of 2008 under the name of "DEBS (Deployable Environmental Biological Surveillance)," the second phase, focused on system design and fabrication stages of the IIMS program, has recently begun. The data gathered and reported by the self-calibrating IIMS enable rapid analyses of drinking water/surface/air for the purpose of supporting risk assessment and decision making capabilities of field commanders. Timely decision-making capabilities through assessment and minimization of environmental and occupational health risks are essential to protect military personnel during and after deployment. The IIMS is will also be adapted to monitor the nation's drinking water network. Transportable systems will be able to monitor drinking water for chemical and biological agents on a 24/7 basis at multiple spots, providing protective shields at specific locations or the entire network. Versions of the IIMS are developed in parallel to measure biochemimarkers™ to validate primary diagnoses of environmentally caused diseases, help predict onset of cellular, immunological and neurological diseases and support patient staging during chemotherapy. The diagnostic systems will also be used for post-deployment health monitoring of military personnel and their families by measuring metabolic biochemimarkers associated with prior exposure to fugitive biological and chemical agents or environmental pollutants. tm- Biochemimarker is a trademark of Applied Isotope Technologies, Inc.

Standard Analytical Protocol for Metal-Based Compounds Using Inductively Coupled Plasma Methods

Adrienne Greenlee
USEPA Region 7
901 N. Fifth Street
Kansas City, MO 66101
913-551-5017
greenlee.adrienne@epa.gov

ABSTRACT

The U.S. Environmental Protection Agency (USEPA) National Homeland Security Research Center (NHSRC), in collaboration with experts from across the U.S. EPA and other Federal Agencies, initiated an effort to identify analytical methods that could be used for the detection of chemical, biological, and radiological agents originated from a homeland security incident. This effort resulted in the publication of the "Standardized Analytical Methods for Environmental Restoration following Homeland Security Events" document (currently SAM 3.1). In addition, U.S. EPA developed Standard Analytical Protocols (SAPs) to assist in determining the existence and extent of contamination, identification of contaminants, effectiveness of decontamination, and site clearance.

Region 7 is performing the single laboratory method development/validation of the standard analytical protocol (SAP) for metal(loids) compounds listed in SAM 3.1 using inductively coupled plasma (ICP) methods. This SAP is based on U.S. EPA SW-846 Methods 3031 and 3050B, EPA OW Method 200.7 and 200.8, and EPA Air Toxics Methods IO 3.1, 3.4, and 3.5 for sample preparation; and U.S. EPA SW-846 Methods 6010C and 6020A for sample analysis. The main objective of the project is to perform a validation of the sample preparation and analysis for target metal/loid compounds of concern in various matrices, including: drinking water, soils, and air. Instrument detection limits (IDLs) and limits of quantitation (LOQs) were calculated for each target compound. If available, reference Risk-based levels (RBLs) were set as upper limits, and thus, calculated LOQs were to be below this threshold. Acceptable recoveries were achieved in all matrices except for the reference soil matrix due to inherent background levels of the target analytes. Following completion of the validation study and revisions to the SAP, several laboratories will be tasked with verifying the revised SAP.

Lowering Detection Limits in the Analysis of Pesticides and Flame Retardants in Drinking Water by GC/MS

Eric Phillips
Thermo Fisher Scientific
2215 Grand Ave Pkwy
Austin, TX 78728
512-251-1551
eric.phillips@thermofisher.com

ABSTRACT

"The Safe Drinking Water Act", as amended in 1996, required the U.S. Environmental Protection Agency to establish a program for the determination of contaminates that are priorities for future regulation. In response the EPA created a Contaminate Candidate list that was designed around a series of criteria, two of which were endocrine disruptors and those compounds which showed adverse health effects. To address the analysis of these compounds, EPA Method 527, Determination of Selected Pesticides and Flame Retardants in Drinking Water by Solid Phase Extraction and Capillary Chromatography/ Mass Spectrometry (GC/MS), was developed for the analysis of 45 contaminates including pesticides, synthetic pyrethroids, and flame retardants in Drinking water."1 The sample is prepared in ethyl acetate and a splitless injection is made with detection in EI Full Scan with a single quadrupole. A calibration curve is generated from 0.25 ug/L to 10 ug/L. The optimization of the instrument method includes: tuning to meet the tuning criteria for DFTPP. The author has validated EPA Method 527 on the Thermo Fisher Scientific DSQ II by optimizing the injection, separation and detection. This was achieved by utilizing a scan speed of 2300 amu/sec and optimization of the pre-filter voltage in the Advanced Tuning Options of the DSQ II. The breakdown of the more fragile pesticides was minimized by using a surge splitless injection. Elevated column flows of 3 mL/min also improved the target compound transfer efficiency through the analytical column, minimizing loss in the stationary phase by either irreversible absorption or chemical degradation. Quality Control data for the accuracy of the calibration curve was within +/- 30 % at each level and the average Instrument Detection Limit was 0.034 ug/L.

REFERENCES

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Profiling Analysis of the Degradation Products of Alkylphenol Polyethoxylates by LC-MS Using an Acclaim® Surfactant Column with Mass Spectrometric Detection

Richard Jack
Dionex Corporation
500 Mercury Way
Sunnyvale, CA 94085
408-481-4227
richard.jack@dionex.com

ABSTRACT

Alkylphenol polyethoxylates (APEOs) are among the most commonly used non-ionic surfactants consisting of approximately 80% nonylphenol ethoxylates (NPEOs) and nearly 20% octylphenol ethoxylates. APEOs are degraded to shorter chain APEOs (with EO units ≤ 3) and alkylphenols (APs) during sewage treatment, with known estrogenic potency and persistency revealed by numerous studies. To obtain the profile information of APs and APEOs from target samples remains a challenge due to the chromatographic complexity of differentiating APEOs by alkyl chain structures and EO units simultaneously. This study demonstrates the first time a total chromatographic separation for short-chain APEOs and APs with mass spectrometric detection for confirmation and quantification.

LC-MS analysis was performed on a coupled Dionex HPLC system and MSQ Plus mass spectrometer with an electrospray ionization (ESI) interface. Chromatographic separation was achieved on a Dionex Acclaim Surfactant column (150×2.1mm, 5µm) with gradient elution at 0.4 mL/min consisting of 3 mobile phase components: methanol, 100 mM ammonium acetate and water. The mass spectrometer was operated in selected ion monitoring (SIM) mode with polarity switching (positive SIM for APEOs and negative SIM for APs). ESI probe temperature was set at 350 °C with probe voltage at 3.0 kV. Method development was performed in terms of column selection, solvent selection, speed of analysis and MS sensitivity. APEOs (OPEO1, OPEO2, OPEO3, NPEO1, NPEO2, and NPEO3) and APs (OP and NP) were well retained and separated in a single chromatographic run. This method takes advantage of the mixed reverse phase retention and ionic interaction to separate APEOs with different alkyl chains (reverse phase retention) as well as different EO units (ionic interaction). Mass spectrometric detection was applied to ensure selectivity and sensitivity. Chromatographic parameters affecting MS response will be explored and discussed. Linearity was achieved over 2 to 3 orders of magnitude for APs and APEOs with correlation coefficients R2 > 0.99. Detection limits were estimated to be 1 ng injected on column for APEOs and 5 ng injected on column for APs.

This is the first LC-MS method to completely resolve and quantify APEOs and APs in a single short run.

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Modified EPA Method 8261 using Pulsed Vacuum Extraction for Analysis of Volatiles in Water

Link Brown, II
Entech Instruments
155 Harrow Drive
Somerset, NJ 08873
805-527-5939
linkbrown@entechinst.com

ABSTRACT

A modified EPA Method 8261 is presented that utilizes alternating vacuum and pressure during the extraction process to analyze Volatile Organic Compounds in water samples. The vacuum pump is positioned downstream of a cryogenic dehydration trap and a collection trap, as in standard 8261, to allow water removal followed by trapping of volatiles drawn out of the sample under vacuum. To effectively transfer the volatiles extracted into the headspace of the 125 mL sample vial, the vial is backfilled with UHP helium to roughly 2 psig, then drawn down to vacuum again. This process is repeated multiple times until the required sensitivity is reached. Unlike vials with septum based seals, a new septum-less seal allows operation under pressure or vacuum conditions without leaks. Data will be presented showing the improved recovery of 1,4-Dioxane and other polar compounds relative to standard Purge and Trap methods.

Apples, Oranges, and SW-846 – National Functional Guidelines Revision Critique

Stephen Zeiner
Environmental Standards, Inc.
1140 Valley Forge Rd.
Valley Forge, PA 19482-0810
610-935-5577
szeiner@envstd.com

ABSTRACT

In July 2007, the US EPA released a new revision of the Organic National Functional Guidelines for Data Validation. These revised guidelines changed several aspects of the evaluation and usability review of the analytical data generated using organic CLP analytical methods. Some of the additions and changes seem to represent an improvement in the assessment of data usability; however, others do not.

As a firm that provides third-party data validation services, Environmental Standards has reviewed tens of thousands of data sets for thousands of sites over two decades - data analyzed in accordance with 40 CFR Part 136 requirements (100-1600 series), drinking water regulations (500 series), RCRA requirements (SW 846), and CERCLA/SARA requirements (Contract Laboratory Program [CLP] Statements of Work [SOWs]). The majority of the data reviewed by the firm in the last 10 years, however, has been from RCRA sites involving US EPA SW-846 analytical methods. Most regulatory agencies have prescribed the use of the US EPA National Functional Guidelines as the guidance document for the performance of data validation for all sites (regardless of the governing regulatory body) because the regulatory agencies do not have published guidelines specific to the published methods.

The changes to the National Functional Guidelines will impact the data usability determinations for the data generated for a large number of sites. Some of the changes in the new National Functional Guidelines alter the interpretation of data. For example, a "not-detected" due to blank contamination result under the former guidelines is interpreted as a not qualified positive result; an estimated result under the former guidelines is interpreted as a rejected result. This presentation will provide information on the changes to the data validation guidelines and a critique of the latest revision.

Vapor Intrusion – A Laboratory Viewpoint

Bryce Stearns
Test America
30 Community Drive
So. Burlington, VT 05403
802-923-1016
bryce.stearns@testamerica.com

ABSTRACT

Vapor Intrusion was an unaddressed exposure pathway for many years. For the past 15-20 years it was suspected as a potential significant exposure risk but it was not until recently that a comprehensive approach to vapor intrusion investigations has been developing to evaluate and address the risks.

In the late 90's Massachusetts & Connecticut published guidance but it was not until the EPA OSWER in 2002 Published "Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Ground Water and Soils" did the evaluation of this exposure pathway really gain National attention. In the past 5 years many States have published draft guidance, published updates to their guidance, and are now have further updates pending.

The EPA with publishing their Vapor Intrusion Guidance in November of 2002 has triggered more States to develop and publish guidance. Many States are holding back and much debate and a rush to collect empirical data associated with investigations of the Vapor Intrusion Pathway is underway.

These Guidance documents, to varying degrees do not address the Laboratory & Data Quality issues related to sampling & analysis for Vapor Intrusion investigations. As empirical data is published from Vapor Intrusion investigations it is becoming more evident that measured exposure concentrations are a critical measurement in the overall exposure risk evaluation process.

NJDEP published their Vapor Intrusion Guidance in October 2005 with conferences and training programs. Earlier this year the first significant update was posted with the announcement that there would be a new Low Level TO-15 method for the determination of Volatile Organic Compounds in air. That update was announce in April 2007 with the publication of the State of New Jersey Department of Environmental Protection Low Level USEPA TO-15 Method (USEPA Method TO15 being the prime method for the analysis of toxic organic compounds in ambient air and soil gas).

Requirements under NJDEPs new Low Level TO-15 analytical method present several changes and new challenges for the analytical testing laboratory. This presentation will highlight the significant changes required by this new method and detail some of the challenges they represent. USEPA Method TO-15 is a widely used analytical technique for the identification and quantification of a broad range of environmental air contaminates. The method employees a suite of quality control checks that ensure the analytical system used to generate and reported

data is reliable and defensible. The new method as proposed by NJDEP builds on that foundation and expands its utility by supporting lower reporting limits. Meeting the expectations of this new method with its additional quality control aspects and achieving the desired reporting limits will present the analytical laboratory with some new, albeit surmountable, challenges.

Trace Metals in the Seagrass *Thalassia testudinum* From Venezuela

Soraya J. Silva, Juan A. Alfonso, and José Azocar; Departamento de Oceanología y Ciencias Costeras, Instituto Venezolano de Investigaciones Científicas, Apartado 21827, Caracas 1020-A, Venezuela; ssilva@ivic.ve
Daniel Palacios; Universidad Simón Bolívar, Apartado 89000, Caracas 1080, Venezuela

ABSTRACT

The globally important seagrass *Thalassia testudinum* is very abundant in Venezuelan coastal zones. We report the concentrations of Cd, Cr, Cu, Ni, V, and Zn in seagrass samples collected from three sites between January and February during 2005 and 2006. The seagrass tissue was separated into blades and roots/rhizomes. Metal concentrations were determined by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). A significant site-specific difference was found only for V. *T. testudinum* is complex in trace metal distribution and appears to vary in concentrations for each metal and morphological unit. Results suggest that if *T. testudinum* is to be used as a biomonitor, care must be taken to analyze the morphological units.

INTRODUCTION

It is well known that seagrasses are critical to the structure and function of many marine ecosystems (Klumpp *et al.* 1989). They provide habitat, sediment stability, nutrients, and a food source. *Posidonia oceanica*, has been studied as a biomarker of trace metal contamination in various parts of the world, especially the Mediterranean coast (Costantini *et al.* 1991, Catiski and Panayotidis 1993). The globally important *T. testudinum* is a climax species (Zieman 1982) very abundant in Venezuelan coastal zones. Along the US Florida coast, their leaves were used to evaluate the As content in several estuaries (Fourqurean and Cai 1993). In the Laguna Madre of Texas, several studies of the trace metal content in *T. testudinum* and *Halodule wrightti* have been reported (Pulich 1980, Whelan *et al.* 2005).

MATERIALS AND METHODS

Study sites: This study was carried-out in three Venezuelan coastal sites (Fig. 1): Isla Buche, *(10°32'36" N-66°05'22" W)*, State of Miranda; Pertigalete *(10°14'38" N-64°34'35" W)*, State of Anzoategui and Isla Larga *(10°20'28" N-64°19'56" W)*, State of Cumana.

Sampling and sample pretreatment: The sampling was carried out between January and February during 2005 and 2006. Seagrass tissue was separated into leaf blades and

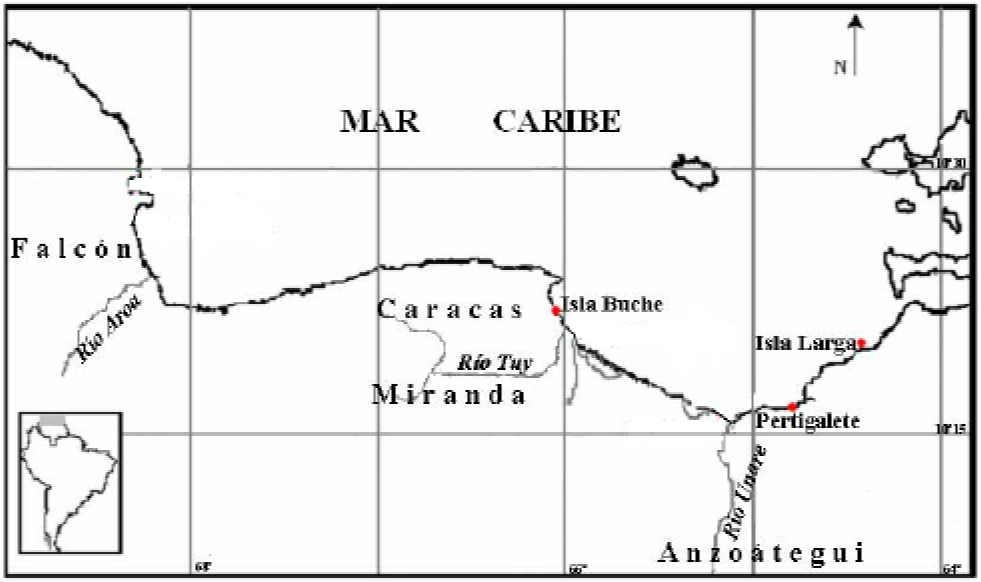

Figure 1. A partial map of Venezuelan coast showing the sampling sites, states and rivers.

roots/rhizomes. Triplicate samples were collected at each sampling site. All samples were rinsed with seawater after collection to remove marine sediments, shells and other debris, placed in plastic bags and transported to the laboratory on ice. Previous studies showed that rinsing seagrasses with distilled water caused premature leaching of metals and other cations (Ledent et al., 1995). Thus, seagrass tissues were blotted with paper towels and set out at room temperature until partially dry in the laboratory. Epiphytes were easily removed from the partially dry leaves by scraping. After partial drying at room temperature, all of the tissue was oven dried at 80 °C for 24 h and cooled in desiccators.

The dried tissue samples were ground into a fine powder with an agate mortar and pestle and well mixed to prepare homogenous samples. One gram portions of the dried tissues were dissolved in 8 ml of concentrated super pure nitric acid (Merck) by heating on a hot plate at about 70 °C for about 8 hours. When the solution cooled, 2 ml of hydrogen peroxide (30 %) (Merck) was added and heated again at 70° C for another 4 hours. Finally, the resulting solution was diluted to 50 ml with distilled deionized water. Duplicate digestions were performed for each sample. Method blanks were always low indicating minimal contamination during sample processing, and they were subtracted from sample readings to give final values of metal concentration.

Chemical analysis: The samples were analyzed by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) using a Perkin Elmer, Optima 3000. Determination of Cd, Cr, Cu, Ni, V and Zn was carried out in the automatic mode, with background correction employing the ICPWinLab Optima 3000 software package. The following wavelengths were used: 226.502 nm for Cd, 205.552 nm for Cr, 324.754 nm for Cu, 231.604 nm for Ni, 292.402 nm for V and 202.548 nm for Zn. A comparison of the trace metals determination using the above described methodology and reported values for plant reference material BCR-60 are presented in Table 1. It can be seen that determined values were very similar to the certified and recommended values, thus the method employed (LaBrecque *et al.* 2004) can be considered accurate. Standard deviations (1σ) of the element determinations (n=6) are small, indicating good precision. The detection limits were determined as three times the standard deviation of the background under the emission lines of the selected trace elements for the blank solution. The detection limits of Cd, Cr, Cu, Ni, V and Zn were 0.79, 0.93, 0.25, 1.1, 0.32 and 0.16 μg/g.

Statistical analysis: A one way ANOVA was performed in order to evaluate the statistical significance of variations between the two sampling times and the sites, for each species and the metals considered. A value of $p<0.05$ was considered to indicate a significant difference. Statistical analyses were carried out using Statistica 6.0 software (StatSoft, USA).

Table 1: The comparison of the determined mean and standard deviation values (n=6) in aquatic plant tissue standard reference material BCR-60 in this study with the certified (CV) values.

Element	BCR-60 Reference material values (μg/g)	This study (μg/g)
Cd	2.20 ± 0.10 (CV)	2.17 ± 0.12
Cu	51.2 ± 1.9 (CV)	51.3 ± 1.1
Zn	313 ± 8 (CV)	311.5 ± 6.3

RESULTS

Results of the one-way ANOVA of comparison between the two sampling times for metal concentrations are presented in Table 2. In all sampling sites, the difference in the mean metal concentrations between the two sampling times was no significant ($p>0.05$), so a combination of 2005 and 2006 data will be considered.

Mean and standard deviation of the trace metal concentrations in *Thalassia testudinum* tissues from the two sampling times are presented in Table 3. Significance levels were fixed at $p < 0.05$ on 95 % confidence level. Mean values of metals with significant concentration differences between the sampling sites are presented marked by an asterisk.

Table 2: Results of the one-way ANOVA of comparison between the two sampling times for metal concentrations.

Sampling site		Cd	Cr	Cu	Ni	V	Zn
Isla Buche	root-rhizome	0.111	0.088	0.241	0.165	0.243	0.058
	leaf	0.201	-	0.301	0.098	0.261	0.079
Pertigalete	root-rhizome	0.251	0.185	0.152	0.231	0.152	0.066
	leaf	0.304	-	0.212	0.259	0.185	0.126
Isla Larga	root-rhizome	0.326	-	0.367	0.127	0.208	0.201
	leaf	0.264	-	0.184	0.210	0.136	0.084

Table 3: Mean concentration of metals in Thalassia testudinum (standard deviation in brackets)

Sampling site		**Cd (µg/g)**	**Cr (µg/g)**	**Cu (µg/g)**	**Ni (µg/g)**	**V (µg/g)**	**Zn (µg/g)**
Isla Buche	root-rhizome	1.52 (0.53)	2.14 (0.30)	5.86 (0.48)	4.62 (2.53)	6.51 (0.25)	25.11 (5.93)
	leaf	1.79 (0.89)	<dl	3.80 (2.61)	3.88 (0.25)	1.60 (0.61)	16.11 (3.48)
Pertigalete	root-rhizome	1.49 (0.24)	1.81 (0.48)	4.73 (1.28)	4.27 (0.18)	15.14* (1.65)	32.47 (4.68)
	leaf	1.6 (0.37)	<dl	6.01 (1.62)	4.83 (2.34)	2.20 (0.81)	14.07 (2.14)
Isla Larga	root-rhizome	1.70 (0.40)	<dl	6.26 (2.22)	3.14 (1.17)	4.49 (0.15)	32.58 (7.02)
	leaf	1.62 (0.26)	<dl	5.41 (0.52)	2.80 (1.44)	1.09 (0.92)	12.31 (3.10)

* = mean values of elements with significant concentration differences between the sampling sites
<dl = below detection limit

DISCUSSION

At Pertigalete, the mean concentration of V in the seagrass root/rhizome tissue was significantly higher and the seagrass leaf tissue contained the highest values for this metal. This coastal area contains very extensive petroleum refining and transportation activities, and so, it was not unexpected the higher value of V. Thus, the present results are consistent with the use of V as possible marker for oil transportation and exploitation activities. The values of the other metals studied did not show significant differences between leaf and root/rhizome tissue. Sampling at three sites at the Venezuelan coast revealed significant site-specific concentration differences for V in *T. testudinum* tissue. No known values have been reported for trace metals for this seagrass in Venezuela.

CONCLUSION

T. testudinum is complex in trace metal distribution and appears to vary in concentration for each metal and morphological unit. Theses results suggest that if *T. testudinum* is to be used as a biomonitor, care must be taken to analyze the morphological units. The variability in trace metal content between the morphological units is highly dependent upon the metal in question.

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Speciation in the Field: A Quick and Easy Way to Preserve Species Information

Jacob Meyer
Speciation Solutions, Inc.
951 Industry Drive
Tukwila, WA 98188
206-219-3779
jacob@appliedspeciation.com

ABSTRACT

Field speciation techniques for the determination of arsenic and chromium species in aqueous samples were compared to proven laboratory analytical methods for application to risk assessment and treatability. The valence state and molecular form of metalloid molecules dictates the toxicological affects and efficacy of treatment options. These disposable field speciation kits have been evaluated to provide an economical method for individuals to assess aqueous systems. The efficacy, limitations, and advantages of the field speciation kits will be discussed.

FORMS II Lite and SCRIBE: A Comparison of Environmental Field Sampling Software

Michelle Berardino and Keri Schaffer
Computer Sciences Corporation
15000 Conference Center Drive
Chantilly, VA 20151
703-818-5264
mberardino@fedcsc.com

ABSTRACT

Accurate documentation of environmental samples and measurements collected in the field is the first step in establishing and maintaining data integrity. The use of field documentation software is vital in creating a chain of custody for sample tracking and the generation of reliable data at the laboratory. Through a comparison of field sampling software, the essential features and functions for proper sample documentation can be identified. EPA's Office of Solid Waste and Emergency Response (OSWER) currently supports two automated data collection tools; FORMS II Lite and Scribe. The efficiency of these tools, in multiple user communities, was evaluated for effectiveness in meeting the technical requirements described in The NELAC Institute's "General Requirements for Field Samples and Measurement Organizations". A side by side comparison of the overall navigation of each program was assessed by compiling a list of all features and functions. A table was generated to record the unique and shared aspects of each program. The use of both programs has automated many of the procedures that must be followed to assure useable data. This, in turn, provides users with both significant cost and time savings and enhanced data quality.

INTRODUCTION

Data integrity is vital when collecting and analyzing environmental field samples and accurate documentation is the first step in the generation of reliable results. FORMS II Lite and Scribe are two data collection tools used to aid in the documentation of samples collected from potential and known hazardous waste sites. Both programs aid in document control and data integrity which are addressed in the ISO 17025 and NELAC standards. FORMS II Lite and Scribe are free to government organizations and are currently used by a variety of user communities. While both programs are able to process and manage environmental data, their individual features and functions differ, resulting in an overall difference in design. In this comparison, features were defined as each main section of the software. Functions were defined as all actions available within each feature. A comparison of the overall navigation of each program was assessed by compiling a list of all features and functions. This list was used to evaluate the effectiveness of each program in documenting field sampling activities.

HISTORY

FORMS II Lite

The Field Operations and Records Management System (FORMS) II Lite software was developed to support the mission of EPA and its contractors who visit hazardous waste sites to collect samples of soil, air, water, or other matrices. FORMS II Lite is a consensus software that was created to be used by multiple sampling organizations. It is a versatile stand-alone Windows-based application that automates many of the procedures that must be followed to assure quality data from the analyses conducted on environmental samples (*FORMS II Lite Version 5.1 Training Guide*, 2003). FORMS II Lite is most commonly used for all steps in the CERCLA pipeline except emergency response. As an example of FORMS II Lite's versatility, it was used to document samples for an asbestos indoor air study during EPA's characterization and remediation of the World Trade Center site in New York City after September 11th. FORMS II Lite was created to allow users to print sample documentation in the field, to minimize the time spent on the completion of manual sample documentation, and assist in the electronic recording of data both prior to and during field sampling activities.

FORMS acted as the precursor to FORMS II Lite and was developed by EPA's National Enforcement Investigations Center (NEIC) and used during the late 1980's through the early 1990's. The first version of FORMS II Lite, version 4.0, was released in 1999 and was a Microsoft Access-based program. In 2001, version 5.0 was released and included an upgrade to using a combination of Microsoft Access and Visual Basic. The most current version of FORMS II Lite is version 5.1 which was released in 2002. Version 5.1 contains several new features including the addition of a computer based training (CBT) which allows users to proceed at their own pace and run FORMS II Lite at the same time to practice steps while walking through the CBT.

As of January, 2008 a total of 22 states, one territory, and three tribes have used the FORMS II Lite software. Since September of 2002, EPA has provided FORMS II Lite training for 160 organizations at 53 sessions, for a total of 772 individuals. This number includes contractors, state personnel, EPA personnel, and other personnel within each EPA Region. As of January 1, 2003, FORMS II Lite was established as the mandatory system to use on all Contract Laboratory Program (CLP) samples. Between October 2006 and September 2007, the use of FORMS II Lite saved approximately $650,000.00 and 17,000 hours for both field personnel and EPA. Since October 2000 to present, the use of FORMS II Lite has saved $3.7 million and 95,000 hours for both field personnel and EPA.

Scribe

Scribe is a software tool developed by the USEPA's Environmental Response Team (ERT) to assist in the process of managing environmental data and provides a flexible user interface to manage, query and view data. Scribe is also a required deliverable for all EPA Emergency Response contracts (*Scribe Basic Info Sheet*). It is used primarily for emergency response in time critical removals by ERT. For example, it was used during EPA's restoration actions at the World Trade Center site in New York City after September 11th.

Scribe was developed in the 1990's to support EPA's Response Engineering and Analytical Contract (REAC). Although the initial development was intended solely for use by REAC laboratories, it became apparent that the software had the ability for use at other laboratories as well. Earlier versions of Scribe included 3.5 and 3.6 which introduced Scriblets and Scribe.NET. Scriblets provide a handheld data capture extension for Scribe and Scribe.NET is a subscription service for enabling a connected system of Scribe clients and distributed Enterprise Systems. Version 3.7 is the most current edition of Scribe and was released in 2005. It includes updates to the user interface and database, and included additional support for Staged Electronic Data Deliverable (SEDD) and FORMS II Lite XML formats (*Scribe v3.7 Release Notes*). Similar to FORMS II Lite, Scribe offers cost and time savings; however, Scribe's ability to be used as a data management tool allows for additional savings.

SUMMARY OF THE SOFTWARE

FORMS II Lite

FORMS II Lite uses a step-by-step wizard approach which follows a logical sequence of steps that parallel the sampling process. Site information is entered through a flexible design that enables data entry prior to and/or during sampling in the field. This data may be replicated and edited multiple times.

FORMS II Lite also offers a variety of user customization functions such as reference tables, user defined preferences within the wizard, and formatting of sample documentation. The reference tables allow data to be predefined according to EPA Regional requirements. Users are able to create and modify sample labels, sample information in the QuickView feature, and Traffic Report/Chain of Custody (TR/COC) report titles and footers.

The exchange of electronic data between multiple users is easily accomplished using the import and export features of FORMS II Lite. Users may import and export site files (.F2L), label templates, and reference tables. The final step in FORMS II Lite allows the export of the Traffic Report/Chain of Custody (TR/COC) report data as an Extensible Markup Language (XML) file that meets EPA CLP requirements.

Scribe

Scribe is a Microsoft Access based program that allows users to capture sampling, observational, and monitoring field data. The navigation menu format facilitates simple movement between sections and is comprised of options for a wide variety of sampling events.

Scribe has the ability to import electronic data including analytical lab result data (EDD) and sampling location data such as GPS. Scribe supports handheld extensions, called Scriblets, to capture and import sampling and monitoring data collected on handheld PDAs. Scribe exports include labels for collected samples, COC generation and analytical lab result data reports. Scribe also supports exporting electronic data for user services such as GIS tools and spreadsheets so sampling data may be further analyzed and incorporated into report writing and deliverables (*Scribe Profile*).

COMPARISON

Through the side by side evaluation of FORMS II Lite and Scribe, a greater understanding of the similarities and differences between the two programs was obtained. Each program was assessed for navigation, data entry format, and the creation of documentation and reports. The following is a summary of the significant shared and unique properties of FORMS II Lite and Scribe. A complete comparison of the features and functions of both programs may be found in Table 1.

Table 1: Functions and Features of FORMS II Lite and Scribe

FUNCTION	FEATURE	FORMS II Lite	Scribe
New Project	Open project	X	X
System Maintenance	Restore from Backup		X
	Repair Database	X	
	Compact Database	X	X
	Backup project		X
Web Data Exchange	Scribe.NET		X
	Electronic Sample Documentation System (ESDS)	X	
Handheld software extension			X
Maintaining Reference Tables/Lists		X (15)	X (62)
Import	Import F2L 5.x file	X	X
	Import Templates	X	X
	Import F2Lite 4.x mdb file	X	
	Import Reference Tables/Lists	X	
	Custom Import		X
	Scriblets		X
	ADR		X
	NARAC XML		X
	RAT		X
	SEDD Stage 1		X
Export	Export project file	X	X
	Export Template	X	
	Export Reference Tables/Lists	X	
	HTML		X
	Export to text	X	X
	Export Spreadsheet	X	X
	Export .xml	X	X
	Export to Dbase	X	
Initiate Event	Enter site information	X	X
	Enter property information		X
	Enter instrument information		X
	Auto population of select fields	X	
Select Sampling Team	Enter sampler name	X	
	Enter organization		X
Select Analyses	Add/edit analyses	X	X
	Enter turnaround time	X	X
	Enter number of bottles	X	
	Enter preservative	X	
	Enter program	X	X
Enter Sampling Locations	Enter location measurements	X	X
	Auto population of system time	X	
	Enter matrix	X	X

FUNCTION	FEATURE	FORMS II Lite	Scribe
Assign Samples	Assign sample number	X	X
	Auto assign customer specific sample number	X	
	Auto increment tag number	X	
	Assign multiple analyses to the same bottle	X	X
	Designate samples for laboratory QC	X	
	Record sample weights	X	
Select Laboratory	Enter laboratory information	X	X
	Assign samples	X	X
Select Carrier	Enter carrier information	X	X
	Assign samples	X	X
Create Chain of Custody	Enter chain of custody number	X	X
	Assign samples	X	X
	Customize header/footer information		X
	Indicate project complete	X	
Reports	Templates	X	X
	Sample numbers	X	
	Chain of custody	X	X
	Sample weight	X	
	Receipt for samples	X	
	Sampler comments	X	
User Interface/Customization	Sort columns	X	X
	Filter	X	X
	Create/save custom layouts		X
	Copy	X	X
	Select all		X
	Clear all		X
	Find and replace		X
	View summary of all data entered	X	
	Customize Field Labels	X	
	Customize Preferences	X	X
	Edit data in summary view	X	
	Wizard navigation	X	
Quality Control	No duplication of sample numbers within a project	X	
	Laboratory QC reminder	X	
	Cannot assign same airbill number to different laboratories	X	
GIS and Laboratory Results	GIS data		X
	Laboratory results		X
User Support	User manual	X	X
	Help desk phone line	X	X
	Help desk email address	X	X
	Computer based training	X	
	Hands-on training program	X	
	Software free to anyone doing work on behalf of the Federal Government	X	X
	Total Features	**63**	**62**

FORMS II Lite and Scribe are both EPA developed field sampling tools that automate the sample documentation process. This includes the creation of TR/COCs which contain the pertinent information necessary for accurate transport and analysis of samples at the receiving laboratory. The ability for user customization through several add/edit features is another shared characteristic. Data exchange between users is a necessary capability for many government organizations and field sampling companies. The import and export features of both programs allow users to record known information, such as site information and analyses, before arriving at the sampling site. These features also permit users to share information after sampling has occurred through the use of exporting reference tables or lists, TR/COCs, and the complete site file.

The primary difference between the two programs is that FORMS II Lite was designed to support one main program, CLP, while Scribe was designed with multiple users in mind. FORMS II Lite uses a wizard throughout the entire sampling process while Scribe only uses a wizard for the first step of creating a new site. In many instances, it is necessary to assign multiple analyses to the same sample or same bottle. This is easily done in FORMS II Lite because the control and shift keys can be used to select multiple analyses at the same time. Scribe does not offer this feature when assigning analyses. In order perform this action, the user must create a new analysis. FORMS II Lite offers quality control (QC) features such as pop-up reminders to supply sufficient volume for laboratory QC samples. In addition, the program does not allow the same airbill number to be entered for samples shipping to different laboratories.

Many of the features Scribe contains that FORMS II Lite does not are due to the fact that Scribe was created from a laboratory perspective. Scribe has the ability to import, store, and manage data associated with measurements generated in the field and at the laboratory through the GIS and laboratory results sections. Scriblets, a handheld extension of Scribe, is a unique feature that may be used in lieu of a computer. This is helpful when samplers are unable to, or it is impractical to bring a computer into the field. Finally, users are able to create custom layouts in all screens whereas with FORMS II Lite, custom layouts are only available in the QuickView, Preliminary Results, Weight Tables, and Generate Labels screens.

CONCLUSION

Without computerized data collection tools, sample documentation must be completed manually, causing an increase in the possibility of human error. Although FORMS II Lite and Scribe were created for different purposes, both now serve as valuable and effective tools in the environmental sampling field. These programs assist organizations in complying with internationally established standards for quality systems, such as ISO 17025 and NELAC. Both programs allow users to not only record information, but also store it for later use and transfer to other data management systems. The use of both FORMS II Lite and Scribe has automated many of the procedures that must be followed to assure useable data. This, in turn, provides users with both significant cost and time savings and enhanced data quality.

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Total Metallic Mercury Analysis in Compact Fluorescent Lamps - Sample Preparation and Analysis

Raj Singhvi
USEPA Region 2
2890 Woodbridge Ave.
Edison, NJ
singhvi.raj@epa.gov

ABSTRACT

The USEPA/ERT is conducting a study to determine mercury vapor emission rates resulting from broken compact fluorescent lamps (CFLs) in a residential setting. The overall objectives of the study are to obtain a mercury emission model and provide the homeowner with cleanup procedures and disposal options for broken CFLs. One of the more important tasks to achieve these objectives is the determination of total mercury content in the CFLs for selection of bulbs for the emission study. Most of the currently available CFLs in the market are manufactured in China for US companies. Mercury content typically varies from 2 to 5 milligrams (mg) depending on product design, lamp requirements, whether the lamp contains metallic mercury or an amalgam, and how mercury is introduced during the manufacturing process. Seven different types of CFLs were purchased from local stores and the cap and electronic parts were removed without breaking the lamp. The CFLs were then placed into a 2-liter polypropylene bottle containing glass marbles with a mixture of nitric acid and bromine monochloride solution. The mixture was then shaken for 24 hours to break the lamp thus minimizing the loss of mercury vapor into the air. The digested samples were analyzed for mercury using standard EPA/ERT cold vapor atomic absorption (CVAA) methods. Results and analytical method development, including sample preparation, will be discussed.

Trace Metals in Bivalves from Venezuelan Coastal Sites

Juan A. Alfonso, José Azocar, and Soraya J. Silva; Departamento de Oceanología y Ciencias Costeras, Instituto Venezolano de Investigaciones Científicas, Apartado 20632, Caracas 1020-A, Venezuela; jalfonso@ivic.ve
Daniel Palacios; Universidad Simón Bolívar, Apartado 89000, Caracas 1080, Venezuela

ABSTRACT

The bivalve mollusks *Tivela mactroides* and *Crassostrea rhizophorae* are abundant along the Venezuelan coast and are widely consumed as seafood. We report the concentrations of Cd, Cr, Cu, Ni, V, and Zn in soft parts of *Tivela mactroides* and the concentrations of Cd, Cr, Cu and Ni in the soft parts of *Crassostrea rhizophorae* collected from seven Venezuelan coastal sites between January and February during 2005 and 2006. Metal concentrations were determined by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). Statistical analyses (one-way ANOVA) indicated that the organisms had different levels of metal bioaccumulation, and might be used as biomonitors for trace metals. Significant site-specific differences were found for Cd, Cr, and V in *Tivela mactroides*; and for Cd and Cr in *Crassostrea rhizophorae*.

INTRODUCTION

Bivalves have been extensively used as bio-indicators to assess the environmental conditions of coastal aquatic environment (Liang *et al.* 2004, Silva *et al.* 2006), since they can accumulate trace metals and other substances. *Tivela mactroides* and *Crassostrea rhizophorae* are two species of bivalves abundant along the Venezuelan coast and widely consumed by local population. The clam *Tivela mactroides* has been considered to be a potential biomonitor of trace metals in Venezuelan marine ecosystems (LaBrecque *et al.* 2004a, Alfonso *et al.* 2005, Jaffe *et al.*, 1995).

MATERIALS AND METHODS

Study sites: This study was carried-out in sampling six Venezuelan coastal sites (Fig. 1): Los Pozones *(10°55’32” N-68°16’42” W)*, in Golfete de Cuare, State of Falcon; Boca de Aroa *(10°45’49” N-68°19’24” W)*, State of Falcon; Isla Buche *(10°32’36” N-66°05’22” W)*, in Carenero, State of Miranda; Playa Chocolate *(10°27’48” N-66°04’59” W)*, in Higuerote, State of Miranda; El Hatillo *(10°08’34” N-65°10’21” W)*, State of Anzoategui; and Isla Larga *(10°20’28” N-64°19’56” W)*, in Mochima, State of Cumana. Each sampling site corresponds to an area of 30-60m^2. Samples of *Tivela mactroidea* were collected at Boca de Aroa, Playa Chocolate and El Hatillo, while samples of *Crassostrea rhizophorae* at Los Pozones, Isla Buche and Isla Larga.

Figure 1. A partial map of Venezuelan coast showing the sampling sites, states and rivers.

Sampling and sample pretreatment: The sampling was carried out between January and February during 2005 and 2006. Samples of *Tivela mactroidea* were colleted by hand from the marine sediment, while samples of *Crassostrea rhizophorae* were collected using a stainless steel knife. Each *Tivela mactroidea* sample consisted of a group of at least 20 clams 3-4 cm in size, whereas *Crassostrea rhizophorae* sample of a group of at least 15 oysters 6-7 cm in size.

In the laboratory, the complete soft tissue of mollusks was removed from the shell and washed with deionized water to remove marine sediment and others debris. The bivalves tissue was transferred to clean one-liter beakers and washed several times with deionized water until the solution was clear. After discarding the excess liquid, the beakers were placed in a drying oven at about 65 °C until most of the liquid was evaporated, then the temperature was increased to 80 °C for 24 hours, then cooled in desiccators.

The dried tissue samples were ground into a fine powder with an agate mortar and pestle and well mixed to prepare homogenous samples. One gram portions of the dried tissues were dissolved in 8 ml of concentrated super pure nitric acid (Merck) by heating on a hot plate at about 70 °C for about 8 hours. When the solution cooled, 2 ml of hydrogen peroxide (30 %) (Merck) was added and heated again at 70° C for another 4 hours. Finally, the resulting solution was diluted to 50 ml with deionized water. Duplicate digestions were performed for each sample. Method blanks were always low indicating minimal contamination during sample processing, and they were subtracted from sample readings to give final values of metal concentration.

Chemical analysis: The samples were analyzed by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) using a Perkin Elmer, Optima 3000. Determination of Cd, Cr, Cu, Ni, V and Zn was carried out in the automatic mode, with background correction employing the ICPWinLab Optima 3000 software package. The following wavelengths were used: 226.502 nm for Cd, 205.552 nm for Cr, 324.754 nm for Cu, 231.604 nm for Ni, 292.402 nm for V and 202.548 nm for Zn. A comparison of the trace metals determination using the above described methodology and reported values for mussel tissue reference materials NIST-2977, NIST-2977 are presented in Table 1. Determined values were very similar to the certified and recommended values, therefore the method employed (LaBrecque *et al.* 2004b) can be considered accurate. Standard deviations (1σ) of the element determinations (n=6) are small, indicating good precision. The detection limits were determined as three times the standard deviation of the background under the emission lines of the selected trace elements for the blank solution. The detection limits of Cd, Cr, Cu, Ni, V and Zn were 0.79, 0.93, 0.25, 1.1, 0.32 and 0.16 μg/g.

Statistical analysis: A one way ANOVA was performed in order to evaluate the statistical significance of variations between the two sampling times and the sites, for each species and the metals considered. A value of $p<0.05$ was considered to indicate a significant difference. Statistical analyses were carried out using Statistica 6.0 software (StatSoft, USA).

Table 1: The comparison of the determined mean and standard deviation values (n=6) in mussel tissue standard reference materials NIST-2976 and NIST-2977 in this study with the certified (CV), recommended (RV) and information (IV) values.

Element	NIST-2976 Reference material values (μg/g)	This study (μg/g)	NIST-2977 Reference material values (μg/g)	This study (μg/g)
Cd	0.82 ± 0.16 (CV)	0.81 ± 0.05	0.179 ± 0.003 (CV)	<dl
Cr	0.50 ± 0.16 (RV)	<dl	3.91 ± 0.47 (RV)	3.92 ± 0.07
Cu	4.02 ± 0.33 (CV)	4.08 ± 0.32	9.42 ± 0.52 (CV)	9.40 ± 0.13
Ni	0.93 ± 0.12 (RV)	<dl	6.06 ± 0.24 (CV)	6.78 ± 0.49
V	-	-	1.1 (IV)	1.06 ± 0.05
Zn	137 ± 13 (CV)	134.8 ± 5.0	135 ± 5 (RV)	129.5 ± 5.8

<dl = below detection limit

RESULTS

Results of the one-way ANOVA of comparison between the two sampling times for metal concentrations are presented in Table 2. In all sampling sites, the difference in the mean metal concentrations between the two sampling times was no significant ($p>0.05$), so a combination of 2005 and 2006 data will be considered.

Mean and standard deviation of the trace metal concentrations in *Tivela mactroides*, and *Crassostrea rhizophorae* are shown in Tables 3 y 4, respectively. Significance levels were fixed at $p < 0.05$ on 95 % confidence level. For each organism studied, mean values of metals with significant concentration differences between the sampling sites are presented marked by an asterisk.

DISCUSSION

The mean concentrations of Cd, Cr and Ni in *Tivela mactroides* samples were significantly higher in Playa Chocolate. This was not unexpected, considering the fact that this site are located west of the mouth of the Tuy River, which has a plume known to move in a north-westerly direction and receives the waste water effluents from the metropolitan area of Caracas, a city of approximately 6 million inhabitants, via the Guaire River. Most of this waste water undergoes only minor treatment. In addition, the Tuy River drainage basin, of approximately 6600 km^2, covers an active industrial and agricultural area which also affects water quality (Jaffe et al., 1995).

Vanadium in *Tivela mactroides* was significantly higher in El Hatillo. This coastal area contains very extensive petroleum refining and transportation activities and is located near of the mouth of the Unare River, which flows along the one of the most important oil producing areas for Venezuela. Vanadium is typically associated to oil (Macías-Zamora et al., 1999; Agusa et al., 2004). The mean concentrations of Cd and Cr in *Crassostrea rhizophorae* samples were significantly higher in Los Pozones. This region is one of the most important tourist (boats and jet skis) zones from Venezuela. Cd and Cr are typically associated to coatings (anti-corrosives), fossil fuel combustion, paints and pigments, and others industrial processes (Siegel 2002).

In contrast to the others elements with significant differences between sites, the difference in the mean concentrations of Cu and Zn in *Tivela mactroides* and Cu and Ni in *Crassostrea rhizophorae*, between the sampling sites where these bivalves were studied is negligible. In contrast to Zn, Ni bioaccumulation was examined in both species of bivalves. Ni concentrations found in *C. rhizophorae* at sampling sites are low compared to those obtained for *T. mactroides.* Some mussel species were found to be able to regulate the level of these biologically essential elements (Phillips, 1985; Malinovskaya and Khristoforova, 1997; Gunther et al., 1999), if the concentration in the aquatic environment does not exceed certain levels. As a consequence of this effect, a direct relationship between biologically essential element concentrations in the environment on one hand, and in the biomonitors on the other hand, would be impossible to observe.

Table 2: Results of the one-way ANOVA of comparison between the two sampling times for metal concentrations.

Sampling Site (organism)	Cd	Cr	Cu	Ni	V	Zn
Boca de Aroa (*T. mactroides)*	0.062	0.207	0.103	0.189	0.096	0.384
Playa Chocolate (*T. mactroides)*	0.056	0.162	0.078	0.069	0.281	0.212
El Hatillo (*T. mactroides)*	0.260	0.399	0.114	0.223	0.192	0.321
Los Pozones (*C. rhizophorae*)	0.102	0.083	0.219	0.312	-	0.241
Isla Buche (*C. rhizophorae*)	0.241	0.221	0.384	0.351	-	0.281
Isla larga (*C. rhizophorae*)	0.401	0.198	0.219	0.254	-	0.199

Table 3: Mean concentration of metals in *Tivela mactroides* (standard deviation in brackets).

Sampling Site	Cd (µg/g)	Cr (µg/g)	Cu (µg/g)	Ni (µg/g)	V (µg/g)	Zn (µg/g)
Boca de Aroa	3.67 (0.13)	2.51 (0.16)	26.32 (1.32)	11.63 (0.28)	1.31 (0.09)	78.13 (9.7)
Playa Chocolate	4.39* (0.28)	3.67* (0.17)	26.20 (1.35)	13.26* (0.33)	1.21 (0.07)	87.89 (7.3)
El Hatillo	3.68 (0.16)	2.69 (0.11)	24.70 (2.32)	11.75 (0.37)	4.03* (0.14)	80.62 (6.8)

* = mean values of elements with significant concentration differences between the sampling sites

Table 4: Mean concentration of metals in *Crassostrea rhizophorae* (standard deviation in brackets).

Sampling Site	Cd (µg/g)	Cr (µg/g)	Cu (µg/g)	Ni (µg/g)
Los Pozones	2.5* (0.6)	2.4* (0.5)	38 (4.73)	1.70 (0.38)
Isla Buche	1.0 (0.1)	1.7 (0.3)	40 (4.67)	1.51 (0.09)
Isla larga	1.6 (0.2)	1.8 (0.6)	35 (4.51)	1.49 (0.20)

* = mean values of elements with significant concentration differences between the sampling sites

CONCLUSIONS

Sampling of *Tivela* mactroides, and *Crassostrea rhizophorae* at sites along the Venezuelan coast revealed significant site-specific concentration differences for the elements Cd, Cr, Ni and V in *Tivela mactroides* tissue, and Cd and Cr in *Crassostrea rhizophorae* tissue.

The mean concentrations in soft *Tivela* mactroides tissue found in Playa Chocolate are within the range of values determined in previous work for the same species of bivalve at the same sampling area (LaBrecque *et al.* 2004a, Alfonso *et al.* 2005). No known values have been reported for trace metals for *Crassostrea rhizophorae* in Venezuela.

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The PCB Shadow and Other Interferences with HRMS PCB Congener Analysis

Steve Wilding
USEPA Region III
701 Maples Road
Fort Meade, MD 20755
410-305-2606
wilding.steve@epa.gov

ABSTRACT

Interferences in the analysis of sediment samples by Method 1668A for PCB congeners were investigated. EPA Method 1668A is an isotope dilution method used to separate and analyze all 209 PCB congeners using high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). This method uses select ion monitoring (SIM) of the exact mass to four decimal places, with a resolution of 10,000. Resolution is determined as Mass/Delta Mass – example: for heptachlorobiphenyl's M+2 mass of 393.8025, an interfering compound would need 0.0394 amu difference in mass to be detected by the HRMS. While the resolution capability of HRMS usually precludes any interference from other compounds, unknown peaks do appear in sediment samples that are not within the retention time of an expected PCB congener. This brings into question whether the detected PCB congeners are actually present or possibly inflated from interfering compounds. Potential interfering compounds with masses within the 10K resolution window of PCB congeners were evaluated to determine whether they matched previously detected interferences.

Another interference investigated was the PCB "shadow". Interferences can be produced from PCBs themselves. For example – when hextachlorobiphenyl fragments during ion bombardment, the molecule could lose one or two chlorine molecules, producing a tetrachlorbiphenyl or pentachlorobiphenyl. When a hexachlorobipenyl coelutes with a tetrachlorbiphenyl or pentachlorobiphenyl, it would inflate the peak area. This "shadowing" effect of higher chlorinated congeners with coeluting lower chlorinated congeners was measured to find out how significant this shadow contributes to an actual congener peak.

Addressing the EPA Recommended Water Quality Criteria for Methylmercury: A Scalable Prep and Automated Analysis System for Mercury Speciation and Ultra-Trace Detection in Biotic Matrices

Christopher Shade
Quicksilver Scientific
1376 Miners Dr.
Lafayette, CO 80026
303-263-6903
chris@quicksilverscientific.com

ABSTRACT

Hg is well-known for is bioaccumulative nature and is a toxin of great importance due to its presence in the food supply. Understanding of the bioaccumulation potential in any waterbody or watershed requires analysis of both the bioaccumulative monomethylmercury (CH3Hg+) and its precursor, inorganic mercury (HgII). EPA's Recommended Water Quality Criteria for Methylmercury involves measurement of CH3Hg+ in biota in the waterbody. Adoption of such criteria requires analytical support that is both robust and economically feasible. We report on a simple scalable leaching procedure that couples to an automated analysis of CH3Hg+ and HgII that employs quantitative on-line preconcentration, thiourea complex separation, and cold vapor atomic fluorescence (CVAFS) detection. The system (Mercury-Thiourea Complex Liquid Chromatography/CAVFS) has an absolute detection limit of 0.4 pg for CH3Hg+ and 0.7 pg for HgII, yielding relative detection limits of 4 and 7 pg g-1, respectively, for 100-mg samples. Sample throughputs are less than 10 min per sample and the method has been extensively validated with fresh and dried fish tissues and macroinvertebrates through the course of two years of use in a commercial laboratory.

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