



**George E. Detsis**  
Analytical Services Program Manager  
**U.S. Department of Energy**  
**Office of Health, Safety and Security**

**Radiation Measurements Cross Calibration (RMCC) Project**  
**RMCC VIII Workshop**  
**Amman, Jordan**  
**June 17-20, 2013**

7/4/2015 1



## **Presentation Outline**



- ❖ **Mixed Analyte Performance Evaluation System**
- ❖ **Results from Testing Series 27/28**
- ❖ **Results of Second Proficiency Test for Ra-226/U-238**
- ❖ **Ten Common Laboratory Findings**

7/4/2015 2



## U.S. Department of Energy (DOE)

# Mixed Analyte Performance Evaluation Program (MAPEP)



7/4/2015 3



## MAPEP Overview



- ❖ Primarily for analytical service providers performing monitoring/environmental remediation for DOE
- ❖ Initial matrices included water and soil, but MAPEP expanded in 2004 to also include radiological air filter/vegetation matrices
- ❖ Administered by the Radiological and Environmental Sciences Laboratory (RESL) under the direction of the DOE Office of Health, Safety and Security and Office of Nuclear Energy



7/4/2015 4



## MAPEP Overview



### ❖ RESL:

- Reference values are directly traceable to the U.S. National Institute of Standards and Technology (NIST) for preparation and analyses of environmental performance testing samples
- Only DOE reference laboratory for environmental analyses

7/4/2015 5



## MAPEP Overview (cont.)

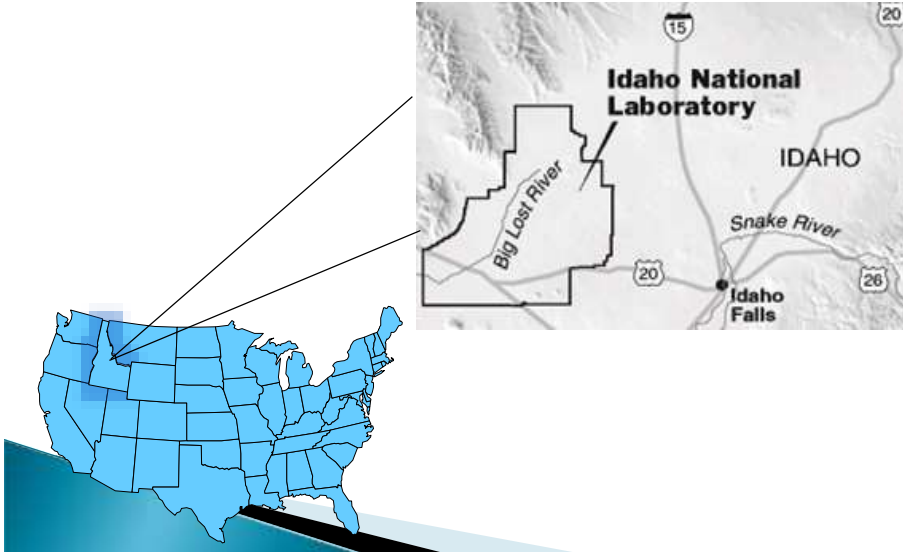


- ### ❖ RESL is accredited by the American Association of Laboratory Accreditation (A2LA) to the following:
- International Organization for Standardization (ISO)/International Electrotechnical Commission (IEC) 17025, *General Requirements for the Competency of Testing and Calibration Laboratories*
  - ISO 17043, *Conformity Assessment – General Requirements for Proficiency Testing*
  - ISO Guide 34, *General Requirements for the Competence of Reference Material Producers*

7/4/2015 6



Idaho National Laboratory



7



Idaho National Laboratory  
Research Center



7/4/2015 8



# Radiological and Environmental Sciences Laboratory



7/4/2015 9



# RESL Staff



7/4/2015 10



## MAPEP Samples



7/4/2015 11



## International Samples Ready For Shipment



7/4/2015 12



## MAPEP Participation Increases



- ❖ **2010** – Radiation measurements cross-calibration (RMCC) included 8 radiological and 2 inorganic laboratories
- ❖ **2011** – RMCC participation increased by 5 radiological laboratories enrolled for Series 23 and 24
- ❖ **2012** – A total of 22 laboratories from RMCC participated in MAPEP Series 27 and 28

7/4/2015 13



## CURRENT RMCC PARTICIPANTS in MAPEP



| LAB CODE | LAB NAME   | COUNTRY  |
|----------|--|----------|
| IAEA99   | International Atomic Energy Agency                                       | AUSTRIA  |
| MSTH99   | Radioecology   | IRAQ     |
| CPAL99   | Chemical and Physical Analysis Laboratories Directorate                  | JORDAN   |
| JAEC99   | Radiation Measurements Laboratory  | JORDAN   |
| JCAL99   | Royal Scientific Society, Environmental Instrumental Analysis Laboratory | JORDAN   |
| JNRC99   | Jordan Nuclear Regulatory Commission                                     | JORDAN   |
| RMCL99   | Royal Scientific Society - Radiation Protection Laboratory               | JORDAN   |
| ERPD99   | Ministry of Health Radiation Protection Department Lab                   | KUWAIT   |
| KUFS99   | Center for Research in Environmental Radiation                           | KUWAIT   |
| PDRL99   | Physics Department Radiological Laboratory                               | KUWAIT   |
| LAEC99   | Lebanese Atomic Energy Commission – Environmental Radiation              | LEBANON  |
| MALA99   | Asia Lab (M) Sdn. Bhd.   | MALAYSIA |

7/4/2015 14



## CURRENT RMCC PARTICIPANTS in MAPEP



| LAB CODE | LAB NAME  | COUNTRY                  |
|----------|---|--------------------------|
| USED99   | National Center for Nuclear Energy, Sciences and Techniques         | MOROCCO                  |
| FMEC99   | Foods and Water Laboratories Center                                 | OMAN                     |
| EANR99   | The Supreme Council for the Environment and Natural Resources       | QATAR                    |
| ESCQ99   | Environmental Studies Centre (ESC)                                  | QATAR                    |
| UQNP99   | Qatar University- Nuclear Physics Lab                               | QATAR                    |
| SANC99   | RadioAnalysis, South Africa Nuclear Energy Corp.                    | REPUBLIC OF SOUTH AFRICA |
| NCNS99   | National Center for Nuclear Sciences and Technologies               | TUNISIA                  |
| IUSF99   | Istanbul University, Department of Biology, Radioecology Laboratory | TURKEY                   |
| UINP99   | IU  | TURKEY                   |
| ADFC99   | Abu Dhabi Food Control Authority Laboratories                       | UNITED ARAB EMIRATES     |

Note: Saudi Arabia, Bahrain, and Yemen anticipate MAPEP participation in the future.

7/4/2015 15



## NEW MAPEP International Participant Policy



**MAPEP strives to meet all our customers' needs and requests; however, the number of returned and/or refused samples shipped at our international customers' request has grown. For this reason, MAPEP will be implementing a new policy for international participants.**

7/4/2015 16





## NEW MAPEP International Participant Policy (cont.)



- ❖ MAPEP international participants must request their samples with every distribution. By requesting a standard, the participant is agreeing to abide by MAPEP's policies and further agrees to payment of any import duties/fees incurred when the samples are received by your local customs office. MAPEP is not responsible for these fees or duties.
- ❖ ANY MAPEP standard that has to be retrieved from customs or has been returned to RESL, for any reason, after being requested, will result in an automatic and permanent removal of that matrix from any future requests for that matrix by the participant. Failure to adhere to this policy, including the timely response to MAPEP Coordinators' and/or MAPEP freight forwarder's request for information will result in suspension of the customer's participation in MAPEP studies.

7/4/2015 17



## MAPEP Series 28 Distribution

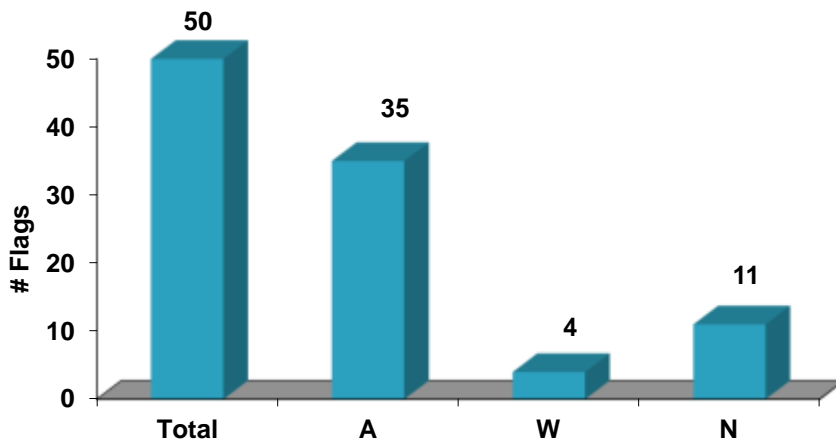


| MAPEP Series 28 Distribution to U.S. and International Laboratories<br>MAPEP Series 28 Samples Shipped in February 2013<br>Reporting Deadline for MAPEP Series 28 was May 1, 2013 |                        |                         |                           |                                      |                                 |                                     |                          |                           |                |
|---|------------------------|-------------------------|---------------------------|--------------------------------------|---------------------------------|-------------------------------------|--------------------------|---------------------------|----------------|
|   | Mixed Analytes in Soil | Mixed Analytes in Water | Gross Alpha/Beta in Water | Radiological Analytes in Air Filters | Gross Alpha/Beta in Air Filters | Radiological Analytes in Vegetation | Organic Analytes in Soil | Organic Analytes in Water | I-129 in Water |
| <b>U.S. Labs</b>  | 87                     | 110                     | 51                        | 68                                   | 58                              | 50                                  | 15                       | 26                        | 27             |
| <b>Foreign Labs</b>   | 39                     | 28                      | 18                        | 24                                   | 15                              | 31                                  | 0                        | 0                         | 3              |
| <b>TOTAL:</b>   | 126                    | 138                     | 69                        | 92                                   | 73                              | 81                                  | 15                       | 26                        | 30             |

7/4/2015 18



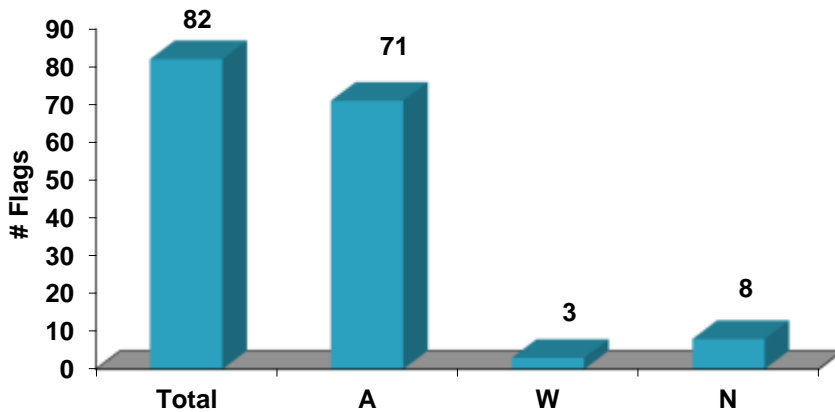
### Inorganic Performance Series 27



7/4/2015 19



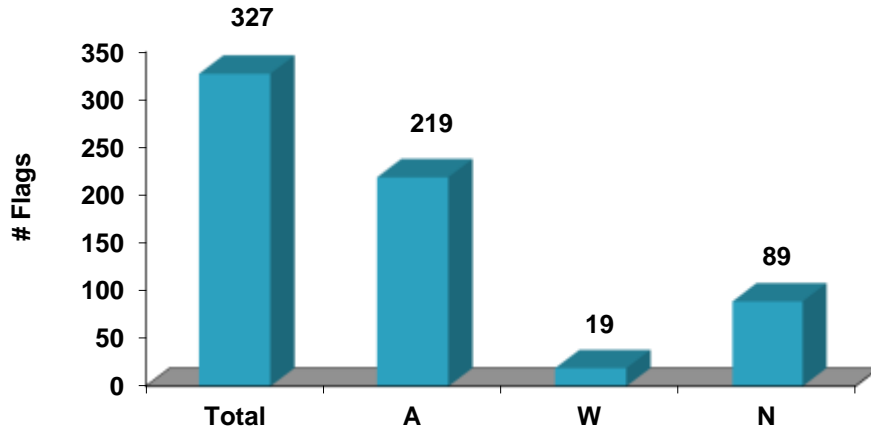
### Inorganic Performance Series 28



7/4/2015 20



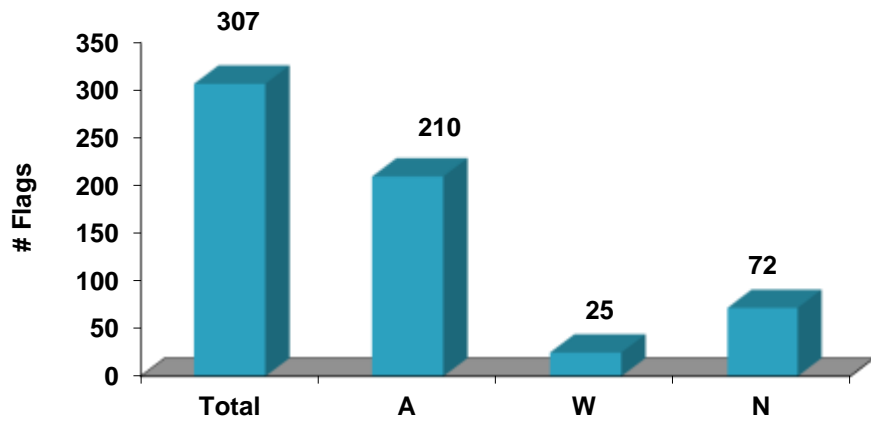
## Radiological Performance Series 27



7/4/2015 21



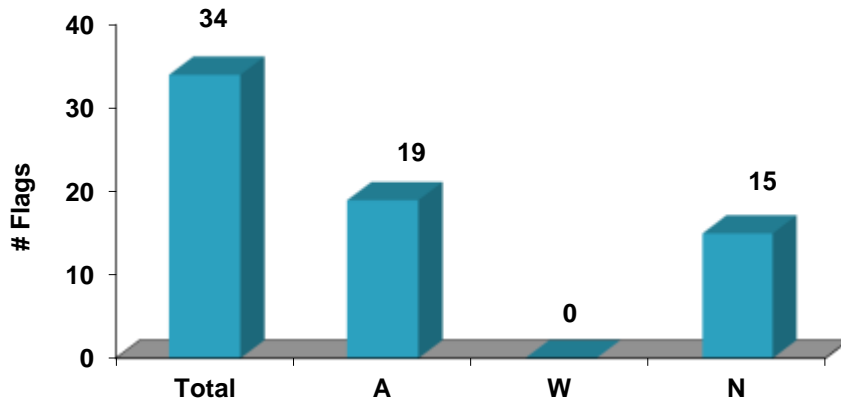
## Radiological Performance Series 28



7/4/2015 22



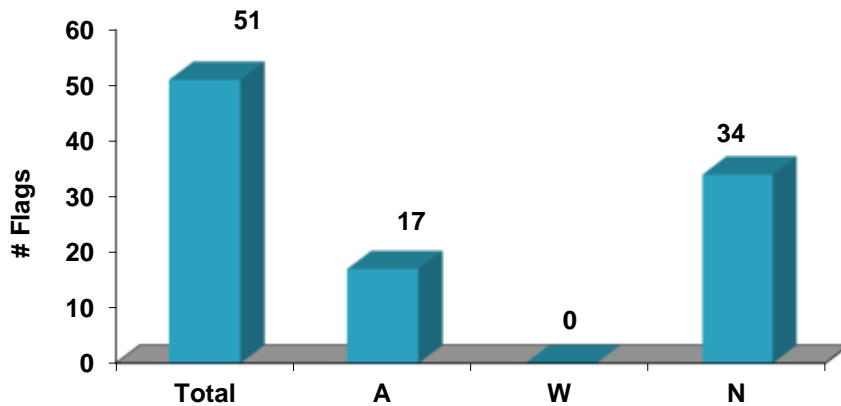
### False Positive Testing Series 27



7/4/2015 23



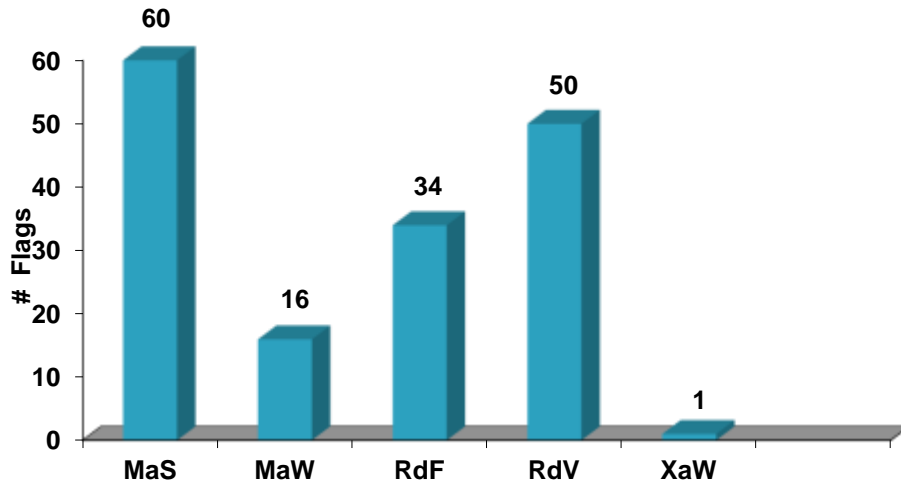
### False Positive Testing Series 28



7/4/2015 24



## “Not Acceptable” Results for Each Sample Matrix in Series 27 and 28



7/4/2015 25



## Reporting Problems



- ❖ Reporting results for MAPEP false positive tests and other special tests near the detection limit are still a major problem
- ❖ A measurement and its uncertainty do not disappear just because the measurement is near the detection limit
- ❖ Failure to report a result and uncertainty for proficiency tests within the laboratories' scope will continue to be “Not Acceptable”

7/4/2015 26



## Ra-226/U-238 Study Results



- ❖ November 2011 – Special soil samples were shipped to 10 RMCC laboratories
- ❖ January 2013 – Second special soil samples shipped to RMCC laboratories containing Ra-226 at 222 becquerel (Bq)/kilogram (kg) and U-238 at 222 Bq/kg
- ❖ Four laboratories participated in the second Ra-226/U-228 Soil proficiency testing study

7/4/2015 27



## Ra-226/U-238 Data Review



|       | Ra-226               |        | U-238                |        |
|-------|----------------------|--------|----------------------|--------|
|       | Ref. Value 222 Bq/kg |        | Ref. Value 222 Bq/kg |        |
|       | Reported             | Bias   | Reported             | Bias   |
| Lab 1 | 271                  | 22.1%  | 242                  | 9.0%   |
| Lab 2 | 321                  | 44.7%  | 255                  | 14.7%  |
| Lab 3 | 224                  | 0.9%   | 258                  | 16.2%  |
| Lab 4 | 122                  | -45.0% | 128                  | -42.5% |

7/4/2015 28



## Radiological Capability



- ❖ Detection of specific alpha and beta emitters at environmental levels of activity require analytical radiochemistry with expertise in wet chemistry separation methods
- ❖ Wet chemistry expertise is difficult to develop and maintain, and it will take time to acquire
- ❖ RMCC laboratories will be limited in their radiological capability if they focus only on gamma-ray spectrometry and gross alpha/beta detection methods

7/4/2015 29



## MAPEP Participation Benefits/Values



- ❖ Used to improve current proficiencies or develop new methods/internal laboratory quality control
- ❖ Data trending – semiannual testing
- ❖ No cost for participation
- ❖ Technical assistance is available

7/4/2015 30



## MAPEP Websites



<http://www.inl.gov/resl/mapep>

- ❖ Public access
- ❖ Statistical summary

<http://mapep.inl.gov>

- ❖ Requires account /password
- ❖ Used for reporting/reviewing data
- ❖ Various search utilities, historical performance reviews, graphs, individual laboratory reports, sample descriptions, program information

7/4/2015 31



## U.S. Department of Energy Consolidated Audit Program

(DOECAP)

7/4/2015 32





## DOECAP Mission

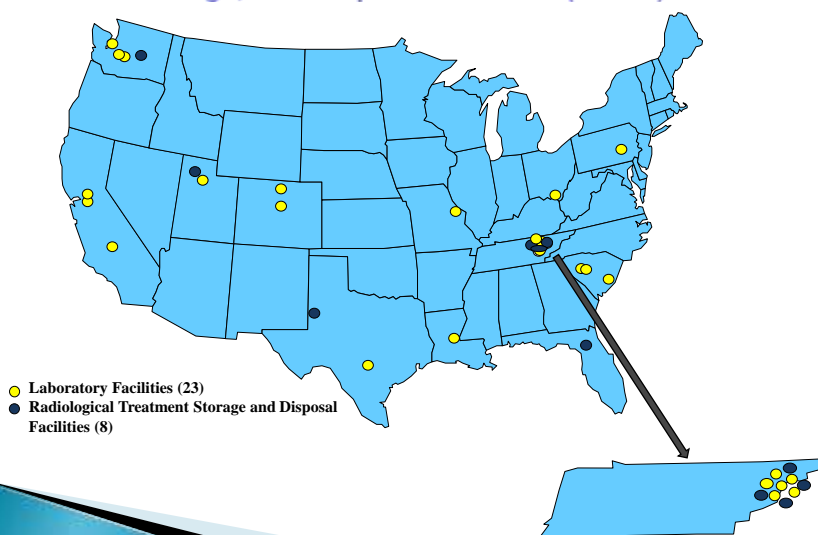


- ❖ Purpose
  - Eliminate/minimize audit redundancies
  - Standardize audit methodology, policies, and procedures
  - Communicate lessons learned
  - Reduce overall DOE risks and liabilities
- ❖ Involve all DOE line organizations/field operations
- ❖ Volunteer auditors – DOE federal employees and contractors
- ❖ Auditor training

7/4/2015 33



## Fiscal Year (FY) 2012 DOECAP-Evaluated Laboratories and Treatment, Storage, and Disposal Facilities (TSDFs)



7/4/2015 34



## Audit Areas



### Laboratory

- ❖ Quality Assurance & General Laboratory Practices
- ❖ Organic Analysis
- ❖ Inorganic Analysis
- ❖ Radiochemistry Analysis
- ❖ Laboratory Information Management Systems (LIMS)/Electronic Data Deliverable (EDD)
- ❖ Hazardous and Radioactive Materials Management
- ❖ Aquatic Toxicity
- ❖ Nondestructive Assay (NDA)

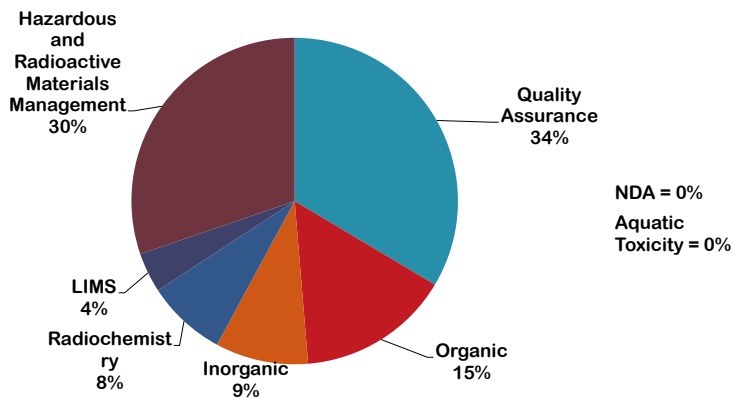
### TSDF

- ❖ Quality Assurance
- ❖ Sampling and Analytical Data Quality
- ❖ Waste Operations
- ❖ Environmental Compliance and Permitting
- ❖ Radiological Control
- ❖ Industrial and Chemical Safety
- ❖ Transportation Management

7/4/2015 35



## Percent Distribution of FY 2012 Laboratory Findings



7/4/2015 36



## FY 2012 Findings



- ❖ DOECAP conducted 29 audits in FY 2012
- ❖ 87% of all FY 2011 audit findings were closed during the FY 2012 audits:
  - 88% of the Laboratory findings
  - 86% of the TSDf findings
- ❖ New findings identified for laboratories and TSDFs since 2006:

FY 2007 = 374

FY 2010 = 227

FY 2008 = 216

FY 2011 = 215

FY 2009 = 277

FY 2012 = 187

7/4/2015 37



## Top Ten Common Analytical Laboratory Findings



### 1. Control of Records

- ❖ Establish/maintain procedures to control quality and technical system records
- ❖ Traceability of observations and derived data performed/recorded (e.g., temperature data of shipping containers, refrigerator storage)
- ❖ Standards and reagent origin, receipt, preparation, and use with analytical run logs, bench sheets, and notebooks maintained
- ❖ Equipment used in analytical testing

### 2. Equipment

- ❖ Initial equipment calibration
- ❖ Annual calibration or verify the equipment for the entire range for which the equipment is used

7/4/2015 38



## Top Ten Common Analytical Laboratory Findings (cont.)



### 3. Quality System/Management System

- ❖ Establishment of laboratory procedures (reviewed annually to reflect laboratory practices)
- ❖ Internal audits/management audits
- ❖ Employee training – general, and specific (initial demonstration of capability [DOC] and annual)

### 4. Handling Samples

- ❖ Procedures for handling samples must clearly describe the process used to uniquely label/record all samples
- ❖ Chain of custody

7/4/2015 39



## Top Ten Common Analytical Laboratory Findings (cont.)



### 5. Personnel

- ❖ Laboratories personnel records
- ❖ Initial data integrity training and annual refresher training

### 6. Management Reviews

- ❖ Annual review of quality systems must include the effectiveness of the system
- ❖ Maintain procedure for management review process
- ❖ Reviews are documented

7/4/2015 40



## Top Ten Common Analytical Laboratory Findings (cont.)



### 7. Standard Operating Procedures (SOPs)

- ❖ implementation of SOPs linked to analytical methods
- ❖ SOPs cover all laboratory activities, including waste management, pollution prevention, worker safety

### 8. Document Control

- ❖ Control of internally generated and external source documents (e.g., regulations, standards, software specification, instruction manuals, material safety data sheets, etc.)
- ❖ Laboratory must establish/maintain procedures for approval/issuance, and changes to all documents that form part of the laboratory's quality system (e.g., logbook revisions)

7/4/2015 41



## Top Ten Common Analytical Laboratory Findings (cont.)



### 9. Internal Audits

- ❖ Establish laboratory internal audit procedure
- ❖ Conduct internal audits of "all" activities to verify laboratory operations that comply with requirements

### 10. Review of request, tenders and contracts

- ❖ Contract review procedure to ensure client requirements and expectations are met.
- ❖ Laboratory contract review procedure must include plan for contracting samples in case of unforeseen circumstances
- ❖ Client complaint documentation and corrective action

7/4/2015 42



## Thank You



- ❖ Opportunity to explain DOE's proficiency testing program and common audit findings
- ❖ Jordan Hosts: MESIS/JAEC
- ❖ RMCC Project Participants
- ❖ Mark Ballantyne U.S. Department of State, Partnership for Nuclear Security
- ❖ Dr. Amir Mohagheghi and Dr. David Betsill, Global Security Engagement, Sandia National Laboratories

**George E. Detsis**

**[George.Detsis@hq.doe.gov](mailto:George.Detsis@hq.doe.gov)**

**(301) 903-1488**



7/4/2015 43