



**U.S. Department of Energy (DOE)  
Office of Environment, Health,  
Safety and Security**

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Radiation Measurements Cross Calibration IX Workshop  
Amman, Jordan  
October 20-22, 2014

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**DOE Mixed Analyte Performance  
Evaluation Program (MAPEP)**

**Administered by DOE's  
Radiological and Environmental  
Sciences Laboratory (RESL)**

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## RESL – Established Quality Systems

**Accredited to ISO/IEC-17025:2005:**  
*General Requirements for the Competence  
of Testing and Calibration Laboratories*  
(ISO Quality Standard for Laboratory  
Operations and External Dosimetry )

**Accredited to ISO/IEC 17043:2010**  
*General Requirements for Proficiency  
Testing*


**Accredited to ISO Guide 34:2009**  
*General Requirements for Certified  
Reference Material Producer*

**National Institute of Standards and  
Technology (NIST)/RESL Radiological  
Traceability Program**

**Soon to have Certification for ISO 14001:**  
*Environmental Management Systems*




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## RMCC Participation in MAPEP

- **2010** – Radiation Measurements Cross-Calibration (RMCC) Program included 8 radiological and 2 inorganic laboratories
- **2011** – RMCC participation increased by 5 radiological laboratories enrolled for MAPEP Series 23 and 24
- **2012** – A total of 22 laboratories from RMCC participated in MAPEP Series 27 and 28
- **2013** – A total of 17 laboratories from RMCC participated in MAPEP Series 29 and 30

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## Current RMCC Participants in MAPEP




Lab Code	Laboratory Name	Country
IAEA99	International Atomic Energy Agency	Austria
ESDG99	Radiation Laboratory	Egypt (New)
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
ERP99	Ministry of Health Radiation Protection Department Laboratory	Kuwait
PDRL99	Physics Department Radiological Laboratory	Kuwait

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## Current RMCC Participants in MAPEP (cont.)



Lab Code	Laboratory Name	Country
LAEC99	Lebanese Atomic Energy Commission – Environmental Radiation	Lebanon
USED99	National Center for Nuclear Energy, Sciences and Techniques	Morocco
FMEC99	Foods and Water Laboratories Center	Oman
ESCQ99	Environmental Studies Centre	Qatar
UQNP99	Qatar University – Nuclear Physics Laboratory	Qatar
NCNS99	National Center for Nuclear Sciences and Technologies	Tunisia
IUSF99	Istanbul University, Department of Biology, Radioecology Laboratory	Turkey
ADFC99	Abu Dhabi Food Control Authority Laboratories	United Arab Emirates

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## MAPEP 2014 Overview



- Two MAPEP sessions each year (Spring & Fall)
- Series 30 Review
- Inorganic Uncertainties Required
- Unique Proficiency Testing (PT) Results
- MAPEP International Participant Policy
- Series 31 Updates & News

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## MAPEP Series 30 by the Numbers



MAPEP Test Session	Series 30
Dates Shipped	March 2014
<b>Laboratories Participating</b>	<b>128</b>
Domestic	90
International	38
<b>PT Standards Shipped</b>	<b>779</b>
Water	240
Soil	129
Air Filters	252
Vegetation	158
<b>Total Results Reported</b>	<b>5,500</b>

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## MAPEP Series 30 Overview



- Inorganic Uncertainties Are Required Starting in Series 30
- False Positives & Sensitivity Tests
- Europium Interference
- Uranium Isotopes Performance
- Uranium Isotopes Soil Discussion

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## Uncertainty for Inorganic Results – WHY?



The result is not an absolute, and the uncertainty characterizes the range around the result within which the true value is expected to lie.

**(result +/- uncertainty)**

- ISO/IEC 17025 requires results with uncertainty
- NIST recommends reporting results with uncertainty
- The international laboratory community routinely reports the analytical result with an uncertainty
- ISO/IEC Guide 98-3:2008, *Guide to the Expression of Uncertainty in Measurement*

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## Uncertainty for Inorganic Results



- MAPEP required inorganic uncertainty reporting in Series 30
- Increase in the number of inorganic uncertainties reported

Series 29	Series 30
269	919

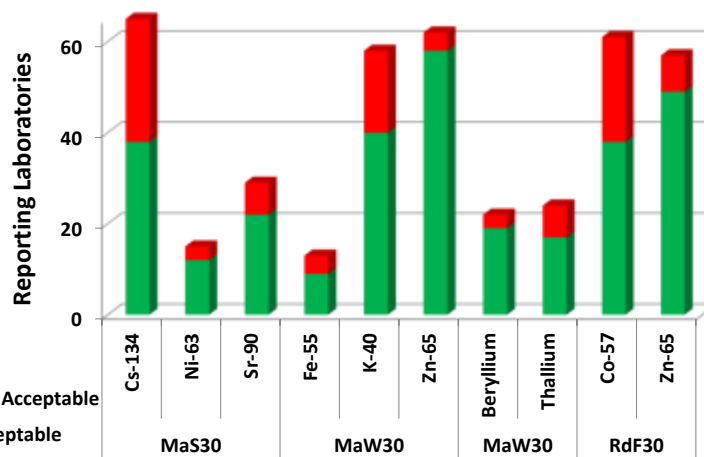
- Inorganic results and associated uncertainties were used in evaluating tests for False Positives and evaluations for Sensitivity/False Negatives

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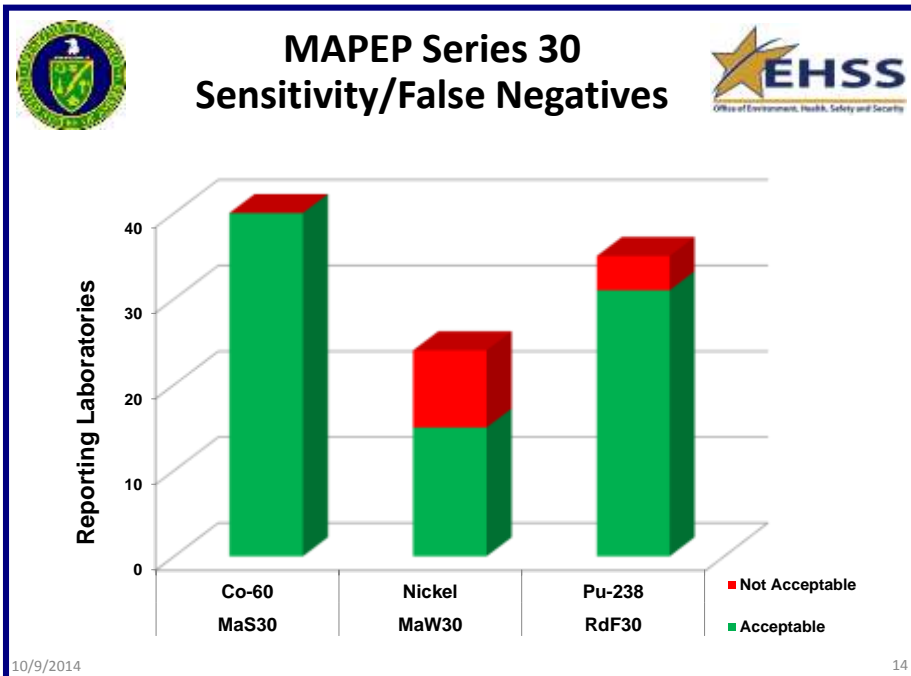
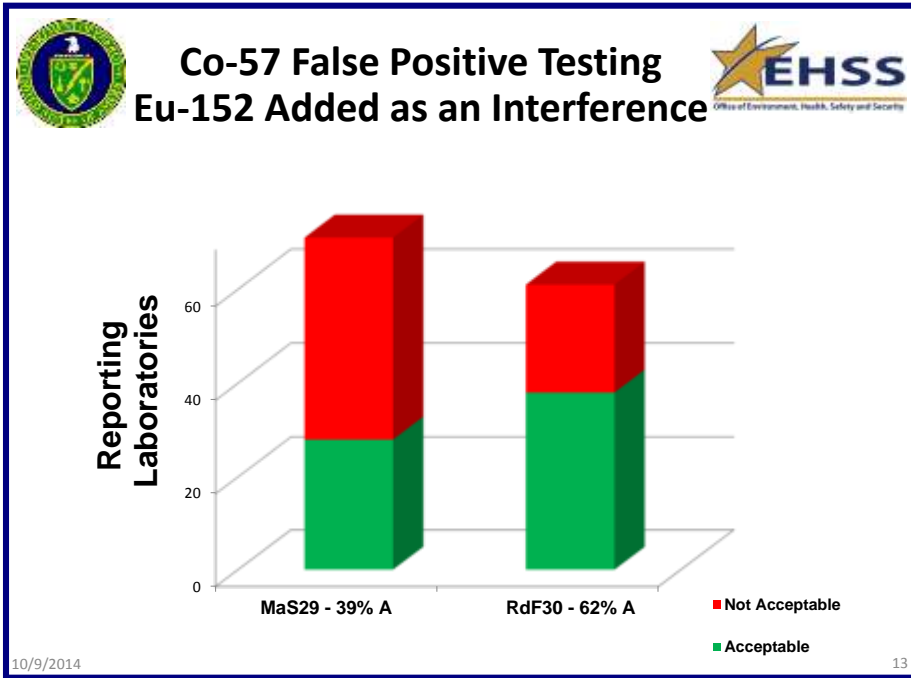


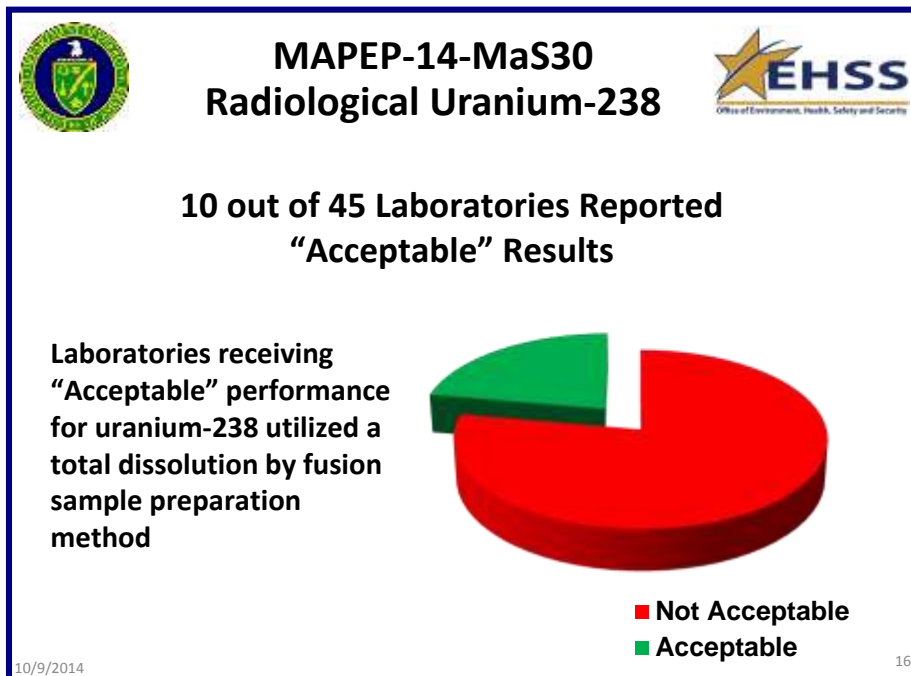
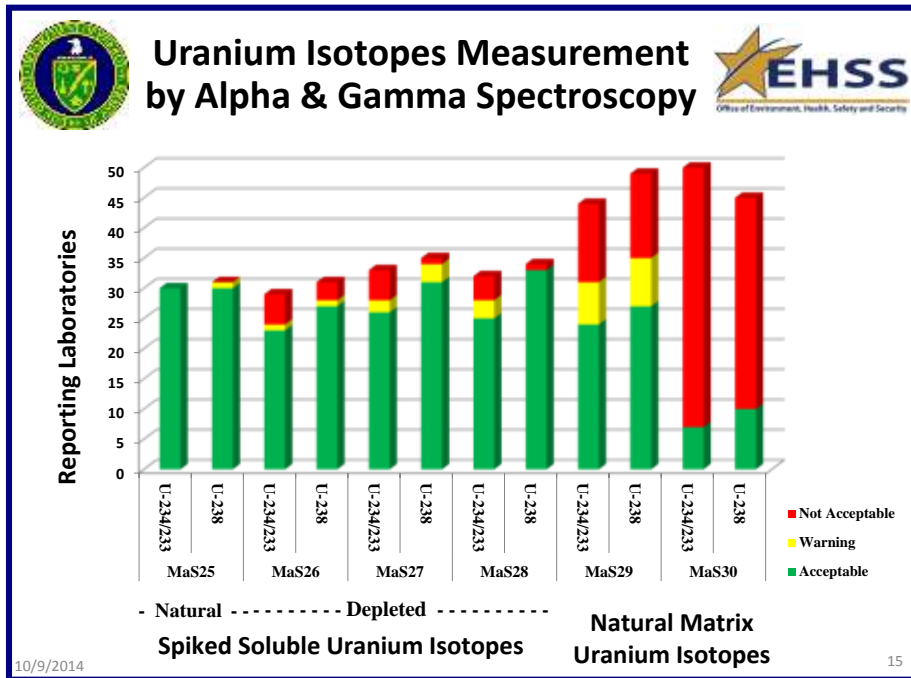
## MAPEP Series 30 False Positives



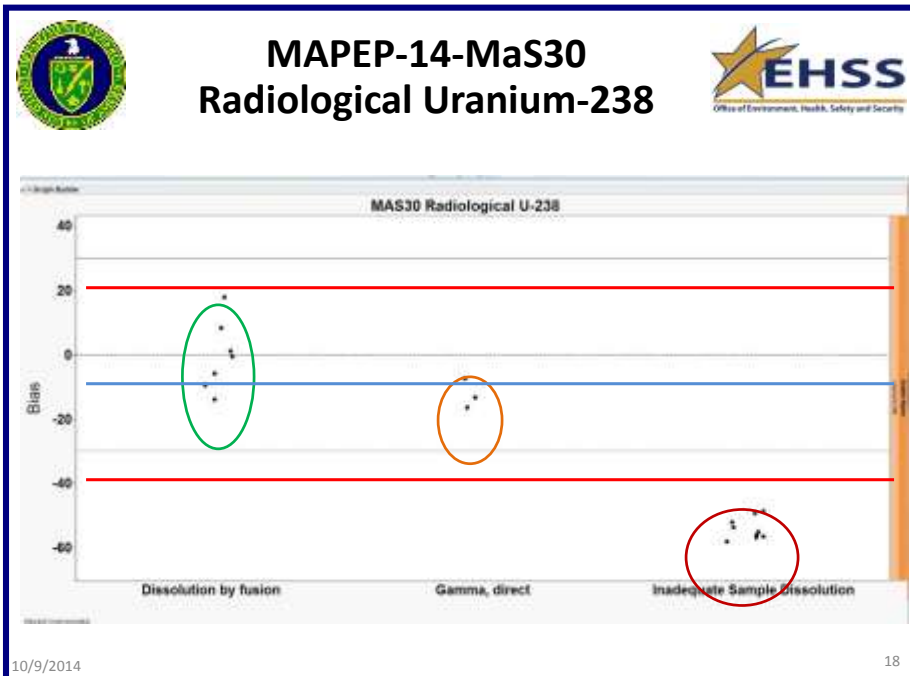
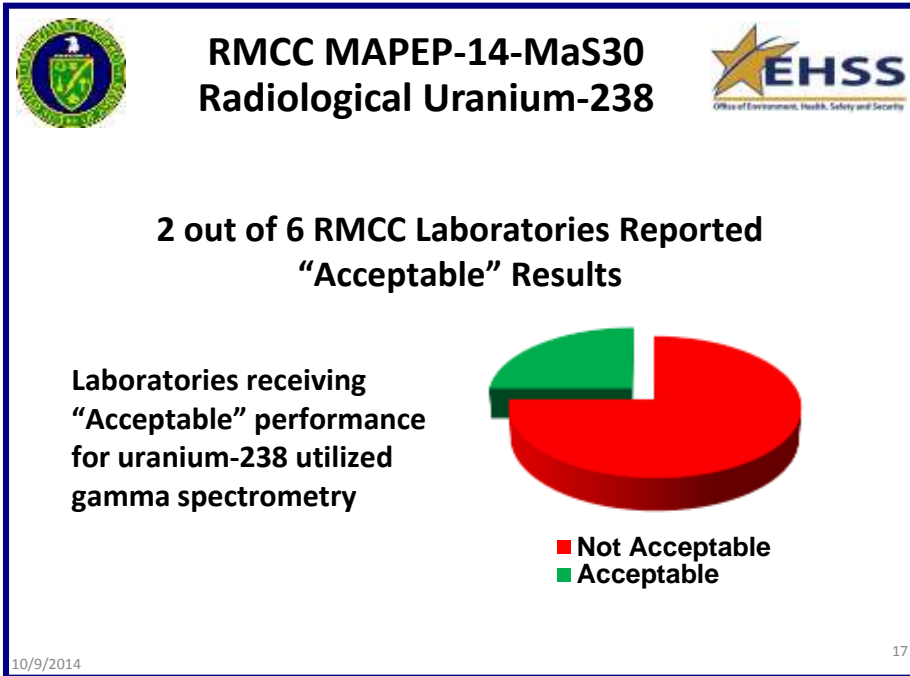
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## Difficulties with Uranium Isotopes



- Natural matrix zirconium-uranium compound in soil difficult to digest
- Poor performance for natural matrix uranium isotopes indicates inadequate procedures for analyzing complex samples by laboratories
- Adequate procedures critical for accurate analysis of actinide isotopes in DOE's environmental samples
- Discussed impacts of poor laboratory performance with the Assistant Secretary for Environmental Management (EM-1), EHSS Quality Council, Nuclear Energy, and other DOE sites' representatives

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## Radiological Capability



- Detection of 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
- Total sample dissolution techniques correctly identified the activity of uranium present
- Inadequate dissolution techniques reported results that were approximately 40% of the reference value
- See the *Uranium White Paper* for more information  
<http://www.id.energy.gov/res/mapep/MAPEPUraniumSoilSeries30.pdf>

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## New in MAPEP Series 31



- MAPEP developed the new Strontium (Sr)-89/90 Air Filter PT Standard for Series 31
- MAPEP Series 31 by the Numbers
- Review of MAPEP's International Participant Policy

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## Sr-89/90 Air Filter MAPEP Series 31




- MAPEP queried it's radiological participants if they would be interested in analyzing a Sr-89/90 air filter
- There are 32 MAPEP laboratories that routinely report Sr-90
  - Only 12 out of the 32 (40%) of the routine laboratories requested the Sr-89/90 air filter
  - Surprisingly, 11 laboratories that have never reported Sr-90 requested the Sr-89/90 air filter
    - Of the 11 "new" requestors; 7 are foreign and 4 are domestic.

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## MAPEP Series 31 Distribution




**MAPEP Series 31 Distribution to U.S. and International Laboratories**  
**MAPEP Series 31 Samples Shipped in August 2014**  
**Reporting Deadline for MAPEP Series 31 is Nov 5, 2014**

	Mixed Analyte 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	Sr-89/90 Filter
U.S. Labs	82	105	50	68	57	51	15	24	23	13
Foreign Labs	39	26	23	30	20	34	0	0	8	10
<b>TOTAL:</b>	<b>121</b>	<b>131</b>	<b>73</b>	<b>98</b>	<b>77</b>	<b>85</b>	<b>15</b>	<b>24</b>	<b>31</b>	<b>23</b>

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## MAPEP International Participant Policy



- Agree to abide by MAPEP's *Export Control Agreement*
- Agree to payment of any import duties/fees incurred
- Participants' use of local customs brokers helps ensure successful delivery of MAPEP samples
- If MAPEP PT samples are returned to RESL or the results are not reported for two MAPEP Series, then that sample matrix is removed from any future requests from the laboratory
- Failure to adhere to this policy, including timely response to MAPEP and/or a freight forwarder's request for information, will result in suspension of the laboratory's participation in MAPEP studies

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# MAPEP System Usage Agreement

**MAPEP** Mixed Analyte Performance Evaluation Program

User: RESLAB  
Logout

**System Usage Agreement**

**RESL Customer Export Control Agreement**

Your acknowledgment and acceptance of the below has expired, it is a prerequisite to using the MAPEP application and must be renewed each stack cycle.

It is the Biological and Environmental Sciences Laboratory's (RESL) policy to conduct business in accordance with all applicable U.S. export control laws and regulations. A part of this requirement is maintaining user laboratory information, such that MAPEP can communicate via email with its customers. It is also RESL's policy that its Customers comply with U.S. export control laws and regulations. Therefore, Customer agrees to the following:

1. Because products, technical data, and technical assistance (i.e., services) provided to Customer by RESL may be subject to U.S. export control laws and regulations, (i) transactions with certain persons and companies and (ii) the exportation or reexportation of certain types and levels of products, technical data, and services are prohibited or restricted.
2. Customer acknowledges that it is responsible for its own compliance with U.S. export control laws and regulations. Customer further agrees that it assumes the responsibility to obtain all necessary U.S. export licenses or other U.S. governmental authorizations, as well as all liability for the failure to do so.
3. Customer acknowledges that export control requirements may change and that the export or reexport of RESL products, technical data, and services without an export license or other appropriate governmental authorization may result in criminal and/or civil liability.
4. Customer agrees to payment of any import duties/taxes incurred when RESL samples are received by customer's local customs office.
5. The obligations and requirements described herein shall survive the expiration or termination of any agreement or contract between RESL and Customer.
6. Failure to adhere to RESL policy, including the acceptance of this notice, timely response to MAPEP Coordinators' requests for information and keeping laboratory's contact information current will result in suspension of Customer's participation in MAPEP activities.

By clicking the acceptance button below, Customer agrees to be bound by the terms of this RESL Customer Export Control Agreement.

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# Export Control Agreement

**MAPEP** Mixed Analyte Performance Evaluation Program

Current Data | Reports | Tools

**Account Tools**

- 
- 
- 
- 
- 
- 
-

**Tools**

✔ Your acceptance of the system's usage agreement has been logged.

Make a selection from the menu on the left

**System Usage Agreement =  
Export Control Agreement**

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## System Usage Agreement





Current Data   Reports   Tools

**Account Tools**

- [Edit Lab Info](#)
- [Edit Study Participation](#)
- [View Ship/Tracking Info](#)
- [Change Password](#)
- [FAQ](#)
- [Sys Usage Agrmt](#)
- [Customer Survey](#)


### RESL Customer Export Control Agreement

It is the Radiological and Environmental Sciences Laboratory's (RESL) policy of this requirement is maintaining user laboratory information such that with U.S. export control laws and regulations. Therefore, Customer agrees:


1. Because products, technical data, and technical assistance (i) regulations, (i) transactions with certain persons and compar data, and services are prohibited or restricted.
2. Customer acknowledges that it is responsible for its own com the responsibility to obtain all necessary U.S. export licenses
3. Customer acknowledges that export control requirements ma an export license or other appropriate governmental authoriz
4. Customer agrees to payment of any import duties/fees incur
5. The obligations and requirements described herein shall sur
6. Failure to adhere to RESL policy, including the acceptance of laboratory contact information correct will result in suspensio

Not accepted on Aug 13, 2014 at 10:21 AM.

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## Conclusion & Reminders



- All analytical results should be reported with an associated uncertainty
- Use analytical methods & personnel that assures accurate results for:
  - False positive & sensitivity tests
  - Full sample dissolution
- MAPEP wants our participants to participate and improve performance

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## MAPEP Application



Easy to fill out MAPEP application at:

<http://www.id.energy.gov/resl/mapep/mapep.html>

Send completed forms to: [MAPEP@ID.DOE.GOV](mailto:MAPEP@ID.DOE.GOV)

For questions contact:

MAPEP Coordinator  
 Guy Marlette (208-526-2532)  
[marletgm@id.doe.gov](mailto:marletgm@id.doe.gov)

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## MAPEP Contact Information



- **Guy Marlette** (208-526-2532), [marletgm@id.doe.gov](mailto:marletgm@id.doe.gov), MAPEP Coordinator
- **Shane Steidley** (208-526-8249), [steidlsd@id.doe.gov](mailto:steidlsd@id.doe.gov), MAPEP website data entry and reports
- **David Sill** (208-526-8031), [sillds@id.doe.gov](mailto:sillds@id.doe.gov), Senior Technical Manager for chemistry and Technical Lead for radiological analyses
- **Leon Jensen** (208-526-4591), [jensenll@id.doe.gov](mailto:jensenll@id.doe.gov), Technical Lead for stable inorganic analyses
- **Steve Bohrer** (208-526-0784), [bohrerse@id.doe.gov](mailto:bohrerse@id.doe.gov), Technical Lead for organic analyses

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## 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

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## Department of Energy Consolidated Auditing Program (DOECAP)



- Implemented the DoD/DOE *Consolidated Quality Systems Manual (QSM) for Environmental Laboratories*, and updated the Laboratory Audit Checklists
- 20 Laboratory Audits, which includes 1 Laboratory Surveillance and 4 Laboratory Closure Audits
- 8 Treatment, Storage, and Disposal Facility (TSDF) Audits
- Processed Corrective Action Plans
- Maintained a cadre of 133 trained volunteer auditors

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## DOECAP



- DOECAP TSDF Checklist Pilot – The facility completed the audit checklists in advance and self-identified deficiencies (treated DOECAP “SI” issues as observations rather than audit findings)
- Updated the TSDF checklists to cover new and revised regulations
- Initiated a DOE Technical Standard on Laboratory PT
- Engaged DOE managers – Briefings
- Analytical Services Program – Fiscal Year (FY) 2013 Report

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## Making a Difference



- Laboratory/TSDF Performance Improvements
- Guard against **Complacency**
- Audit report quality improvements – findings are tied to requirements, specific requirements are cited, and the supporting evidence identified by the auditors is explained
- DoD/DOE QSM provides laboratories with one-stop shopping for quality system requirements
- Reduction/elimination of aged legacy waste

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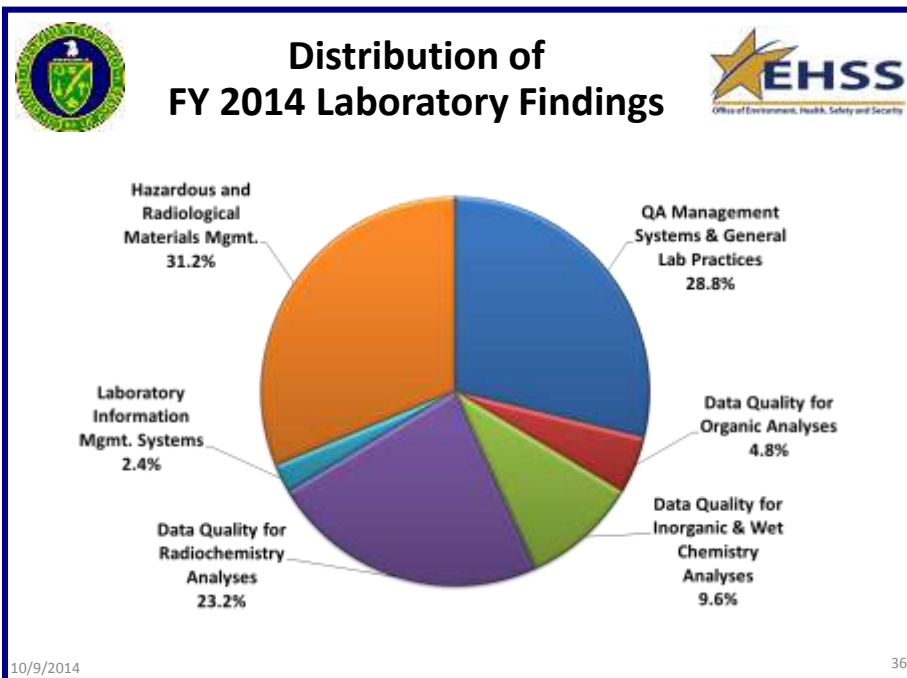
## Laboratory Audits








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## Commonalities Between the FY 2014 Laboratory Findings



- Procedures:
  - Laboratory practices/standard operating procedures (SOPs) do not accurately reflect the current analytical method
  - Lack of necessary SOPs
  - Employees not trained to latest/approved procedures (lack of training documentation)
  - Employees not following approved laboratory procedures
  - Procedures not updated annually
- Not running quality control samples at required frequency
- Records not maintained for equipment (preventive maintenance) and supplies (reagents/solvents)
- Opening incoming sample shipments outside of a ventilation hood, which means that personnel are potentially being exposed to hazardous materials

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## Commonalities Between the FY 2014 Lab Findings (cont.)



- Performing calculations associated with sample analysis using spreadsheets with formula cells that are not write-protected
- Using acceptance criteria for analytical balances and pipettes that do not meet the QSM requirements
- Lack of analytical waste segregation, labeling, storage and disposal practices
- Lack of internal audits, and annual management review to foster continuous improvements
- Not updating the Quality Assurance Management Plan
- Logbook documentation issues

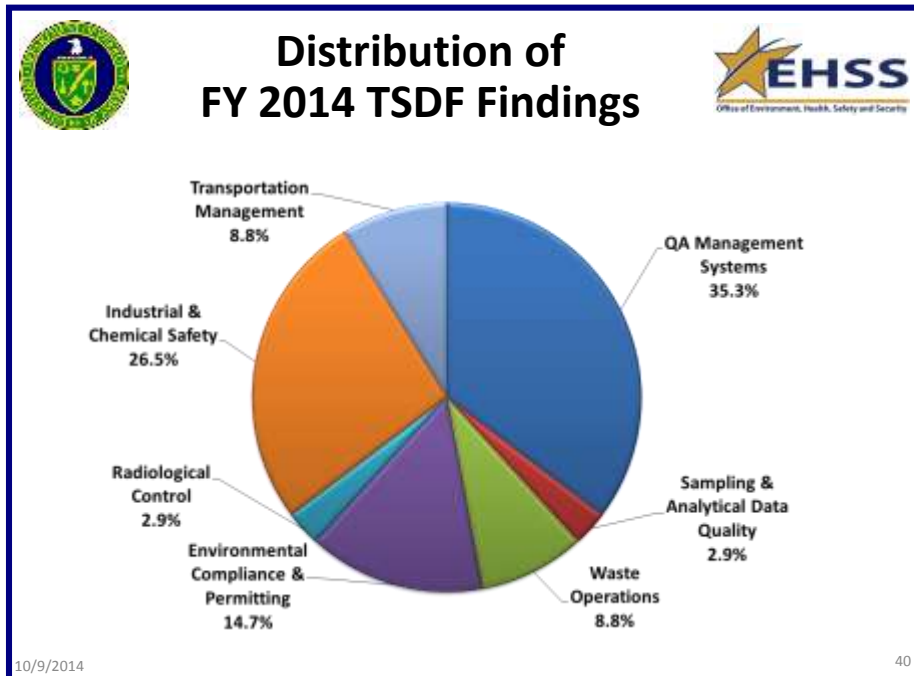
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 **DOECAP TSDF Audits**   
Office of Environment, Health, Safety and Security



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## Commonalities Between the FY 2014 TSDF Audit Findings



- Outbound shipping paperwork does not meet the requirements; for example:
  - Technical Name requirements for hazardous material shipments
  - Shipping Description and Hazard Class for hazardous material shipments
  - Required content and signatures on outbound shipping papers
- Not ensuring that workers complete the required training for work under the hazardous waste management permit and/or failing to properly document completion of the required training

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## Commonalities Between the FY 2014 TSDF Audit Findings (cont.)



- Not meeting the requirements for:
  - Chemical storage (incompatibles)
  - Waste segregation
  - Universal waste labeling and marking
- Failing to properly label measuring and test equipment with the calibration status.

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## Contact Information



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## Questions?



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