Radiological Traceability Program (RTP) between NIST and the DOE Radiological and Environmental Science Laboratory (RESL)

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Traceability

- The formal definition of traceability is the ability to interrelate uniquely identifiable entities in a way that is verifiable.
- A Radiological Traceability is the ability to relate measurement's result to NIST reference value and well document the recorded identification and evaluation.

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Performance Test / Evaluation

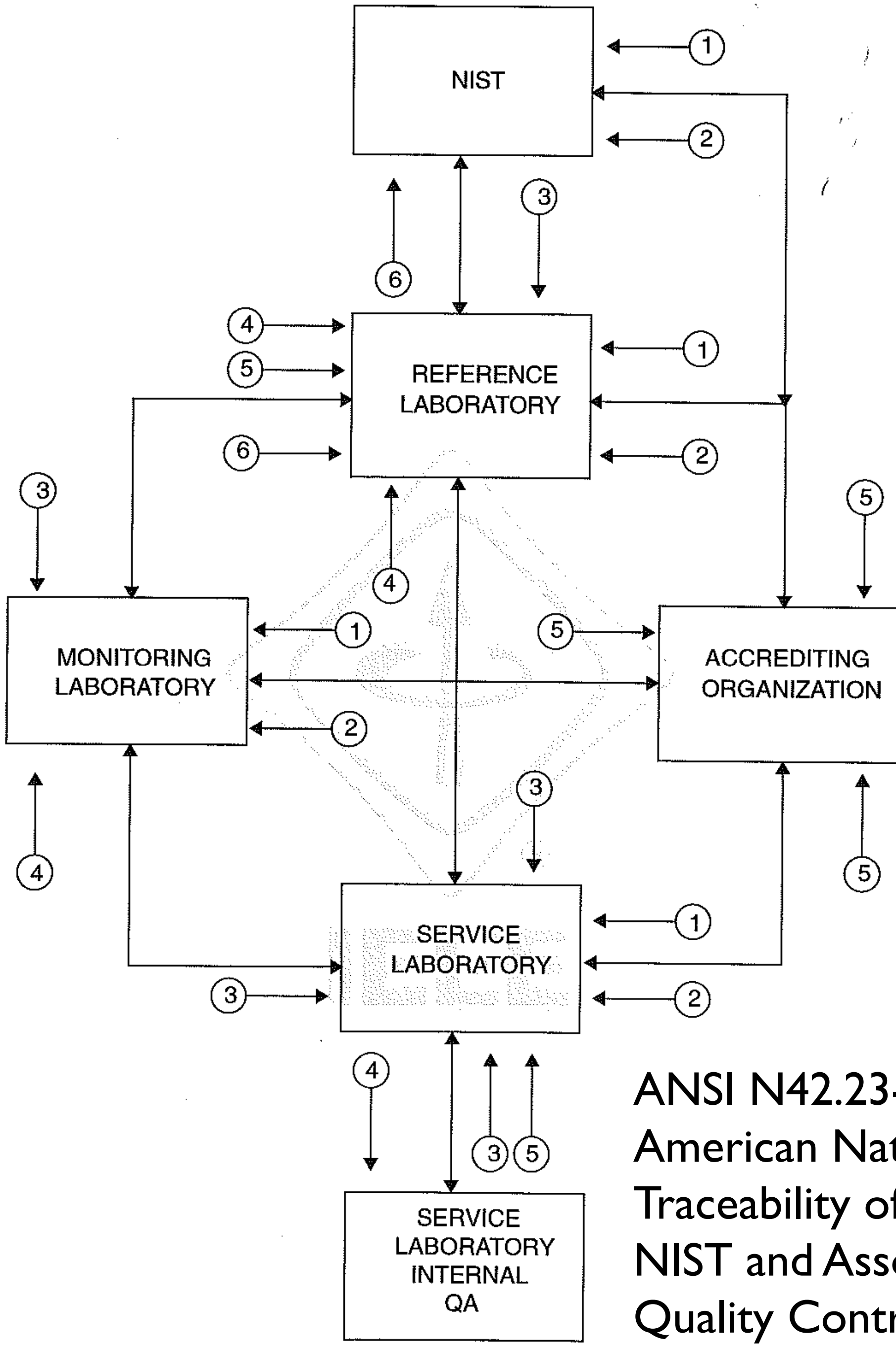
- PT determines the performance of individual laboratories for specific tests or measurements and is used to monitor laboratories' continuing performance.
- A PT is an assessment that requires an examinee to actually perform a task or activity, rather than simply answering questions referring to specific parts.
  - \- what is your dissolution procedure
  - \- what is the tracer
  - \- what is the separation scheme
  - \- what is the source preparation
  - \- what is the spectrum analysis approach
  - … etc.

Proficiency Testing

- The successful completion of a well-designed PT can validate the measurement method, technical training, traceability of standards, and uncertainty budgets of the laboratory.
- PT can provide a good indication about the quality of the reported results.
- Failing a PT can identify nonconformities within the laboratory quality system, and allow the laboratory to improve their processes before the nonconformities come to the attention of the laboratory's customers.
- Laboratories appreciate the lessons learned in the experience.
- PT reports can provide information on where a laboratory may need improvement.

How do you need to perform?

- To assure and maintain your capability to have measurements that pass certain requested criteria to be traceable to NIST.
- If you are a PT Provider, also assure and maintain the capability to prepare testing samples.
- The Quality Assurance guards your performance.



ANSI N42.23-1996
American National Standard – Traceability of Radioactive Sources to NIST and Associated Instrument Quality Control

Who are the Players?

- NIST
- Reference Laboratory
- Accrediting Organization
- Monitoring Laboratory
- Service Laboratory

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Radiological and Environmental Science Laboratory (RESL)

- ✓ RESL is the designated **Reference Laboratory for DOE** and the Nuclear Regulatory Commission (NRC) Radiological Measurement Assurance Program (RMAP).
- ✓ RESL maintains direct traceability to NIST through

  Radiological Traceability Program (RTP).
- ✓ DOE authorized RESL to prepare and distribute PT materials to DOE's laboratories.
- ✓ RESL covers 2 DOE Laboratory Accreditation Programs:
  - Environmental Health (in-vivo & in-vitro bioassay)
  - Environmental Management (monitoring & remediation).

Radiological Traceability Program (RTP)

- The RTP provides an annual exchange of PT materials between NIST and RESL.
- It is designed to provide a mechanism for evaluating the ability of RESL scientists to prepare PT materials containing known activities of various radionuclides and to verify the PT samples they prepared.
- NIST and RESL prepare and exchange PT materials as defined in the RTP's Statement of Work (SOW).

NIST ⟷ RESL

- NIST prepares PT materials for RESL to be analyzed.
- RESL do the analysis (**characterization of the NIST PT material).**
- NIST evaluates RESL's analytical results.
- NIST issues a **Report of Traceability for characterization of PT material for:**

  - air filters
  - soil
  - vegetation
  - water
  - synthetic urine
  - synthetic feces.

RESL ⟷ NIST

- RESL prepares Water PT samples for NIST to be analyzed.
- NIST performs verification measurements on samples and compares the known values with the experimental results generated by NIST.
- A **Report of Traceability for Preparation of PT Material** is issued.
- The traceability criteria for this part of the Program is based on ANSI N42.22.

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Traceability acceptance criteria for ability to Prepare a PT Material

The traceability acceptance criteria for materials prepared by RESL to be consistent with ANSI N42.22-1995. The criteria has a statistical basis that includes the uncertainty of the measurements reported by NIST and RESL.

NIST reviews their measurement results and the reference values stated by RESL and performs the necessary statistical calculations to determine the level of compliance to the traceability criteria stated in ANSI N42.22.

Traceability acceptance criteria for ability to Prepare a PT Material

- The combined uncertainty is calculated according to ANSI N42.22 that incorporates NIST and RESL reported uncertainties.

  $|A_{NIST} - A_{Lab}| < 3 * \text{sqrt}((U_{NIST})^2 + (U_{Lab})^2)$

  A_{NIST} = NIST nuclide concentration value

  A_{Lab} = RESL nuclide concentration value

  U_{NIST} = expanded uncertainty (k=1) for A_{NIST}

  U_{Lab} = expanded uncertainty (k=1) for A_{Lab}

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Traceability Acceptance Criteria for characterization of PT material

The NIST traceability acceptance criteria for the nuclides and matrices prepared by NIST and sent to RESL is based on a relative percent difference from the NIST value, without consideration of the uncertainty of the measurement process.

Stated Traceability limit in the RTP SOW = 9 %.

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Reporting Requirements

- The analytical results are reported in terms of specific activity of the spiking solution (activity per gram), and not the total activity contained in the sample.
- NIST provides RESL with the actual weight of spiking solution used to prepare each sample.
- NIST provides RESL with the actual weight of matrix used to prepare each sample.
- The natural background activity contained in the sample matrix is subtracted from all analytical measurements (blank correction).

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Resolution of Results Outside of the Acceptance Criteria

If the difference between the RESL and NIST results is outside of the acceptance criteria, the RESL and NIST POCs will attempt to resolve the discrepancy. Additional PT samples may be submitted to RESL or NIST for re-analysis if resolution cannot be reached from discussions.

Radionuclides for the Radiological Traceability Program (RTP)

Nuclides	Category
Th-230, U-238, U-234, Np-237, Pu-238, Pu-239/240, Am-241	Alpha Emitting Radionuclides
H-3, Sr-90	Beta Emitting Radionuclides
Cs-137, Cs-134, Co-60, Mn-54, Co-57, Zn-65	Gamma Emitting Radionuclides
I-125, I-131	Isotopically Pure Radionuclides

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Matrix, Radionuclide, Activity Ranges, and Sample Size for RTP (NIST Samples Sent to RESL)

Matrix	Spike	Sample mass	Activity
Water	Alpha (no Np or Th)	5 g	0.1 – 1.0 Bq/ sample
	H-3	500 g	1 – 10 Bq/g
	Sr-90	5 g	1 – 10 Bq/ sample
	I-125, I-131	5 g	100-2000 Bq/sample
	Gamma	5 g	10-100 Bq/ sample

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Matrix, Radionuclide, Activity Ranges, and Sample Size (NIST Samples Sent to RESL)

Matrix	Spike	Sample mass	Activity
Glass Fiber Filters	Alpha (no Np or Th)	1 filter	0.1 – 1.0 Bq/sample
	Sr-90	1 filter	1 – 10 Bq/sample
	Gamma	1 filter	10 – 100 Bq/sample
Soil	Alpha (no Np or Th)	1 g	0.1 – 1.0 Bq/ sample
	Sr-90	1 g	1 – 10 Bq/ sample
	Gamma	1000 g	0.1 – 1.0 Bq/g
Vegetation	Alpha (no Np or Th)	1 g	0.1 – 1.0 Bq/ sample
	Sr-90	1 g	1 – 10 Bq/ sample
	Gamma	1 g	10-100 Bq/ sample

Matrix, Radionuclide, Activity Ranges and Sample Size for RTP (NIST Samples Sent to RESL)

Matrix	Spike	Sample mass	Activity
Synthetic Fecal	Alpha	~100 g SF sample	0.1 – 1.0 Bq/ sample
	Sr-90	~100 g SF sample	1 – 10 Bq/ sample
	Gamma	~100 g SF sample	10-100 Bq/ sample

Synthetic Urine	Alpha	34 g SU salt	0.1 – 1.0 Bq/ sample
	H-3	500 g SU solution	1 –10 Bq/g
	Sr-90	34 g SU salt	1 – 10 Bq/ sample
	Gamma	34 g SU salt	10-100 Bq/ sample

Synthetic Urine Samples Sent to RESL by NIST

Nuclide	Activity Range (Bq/Sample)	Sample Size	Spiked Samples	Blank Samples	Traceability Acceptance Criteria
Th-230, U-238, U-234, Np-237, Am-241,Pu-238, Pu-239/240	0.1 – 1.0	34g SU Salt	6	2	≤ 9%
H-3	1 – 10 Bq/g	17g SU Salt in 500g Sol. (Glass Bottle)	1	1	≤ 9%
Sr-90	1 – 10	34g SU Salt	5	2	≤ 9%
Cs-137, Co-60, Mn-54, Co-57, Zn-65, Cs-134	10-100	34g SU Salt	5	2	≤ 9%

Note: Acceptance Criteria from the RTP SOW.
Replicates are provided.

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Water Matrix Sent to NIST by RESL.

Nuclide	Activity Range (Bq/g)	Sample Size	Spiked Samples	Blank Samples
U-238, U-234 Pu-238, Pu-239/240, Am-241	0.001 – 0.01	1000g	1 bottle	1 bottle
H-3	1.0 – 10	500g	1 bottle	1 bottle
Sr-90	0.01 – 0.1	1000g	1 bottle	1 bottle
Cs-137, Co-60, Mn-54, Co-57, Zn-65, Cs-134	0.1-1.0	1000g	1 bottle	1 bottle

Note: Acceptance Criteria of ANSI N42.22
Aliquot subsamples for replicated measurements

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Driving considerations for preparation of a spike mixture or a Master Solution

- Minimum spike mass 0.1 g.
- Composition must comply with the RTP SOW requirements.
- Decay corrections are not compromising the activities level over a few years.
- To analyze the Master Solution as a verification of the massic activities derived from the gravimetric measurements.

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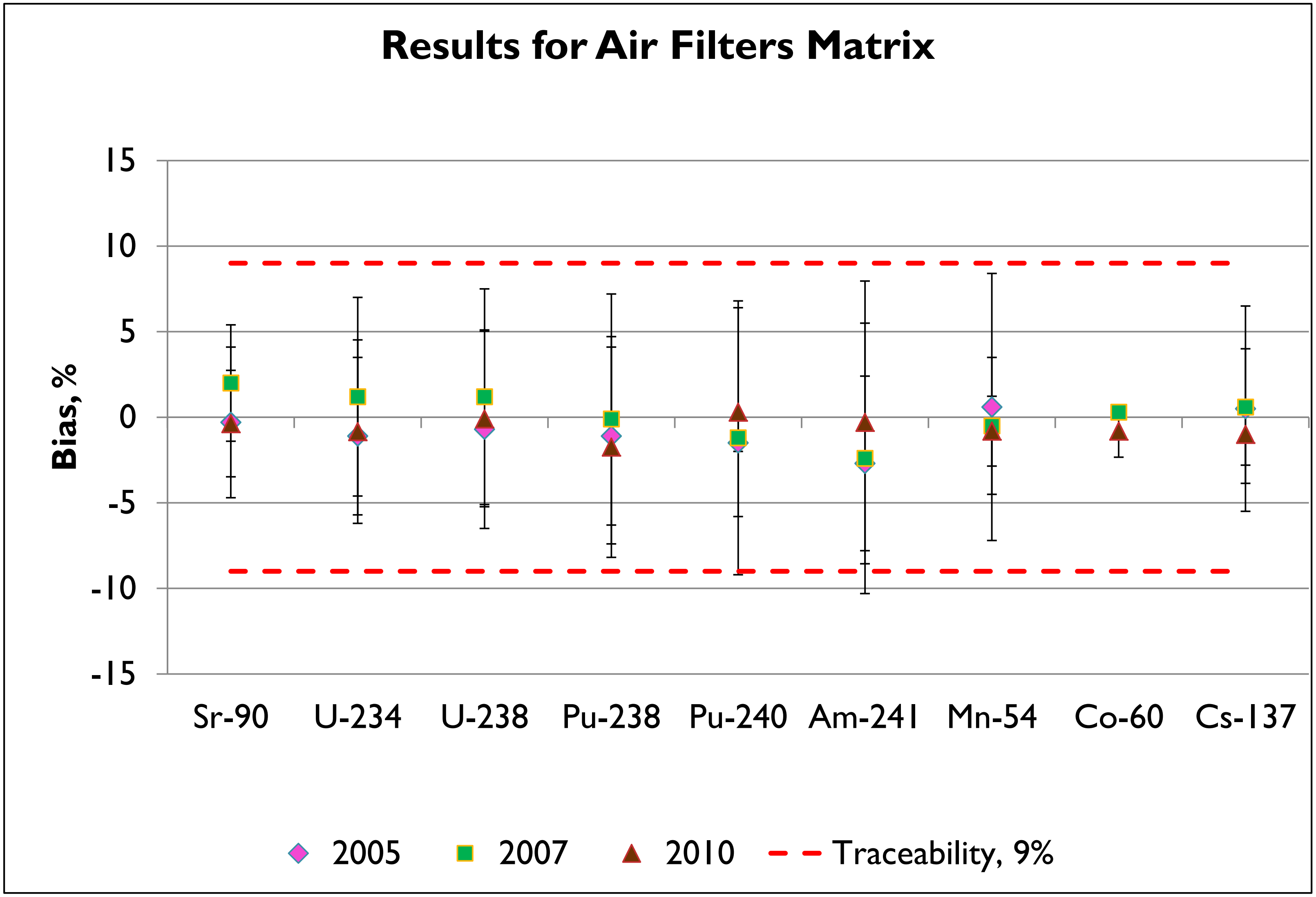

Note: Counting statistics drives the reported uncertainty

Performance for AF analysis

Radionuclide	Bias average, %	Bias variation, %		Average reported uncertainty, %
		from	to	%
Sr-90	0.44	-0.37	2.0	3.6
U-234	-0.25	-1.1	1.2	5.3
U-238	0.13	-0.70	1.2	5.7
Pu-238	-0.98	-1.7	-0.10	6.3
Pu-240	-0.80	-1.5	0.30	4.9
Am-241	-1.80	-2.7	-0.30	7.1
Mn-54	-0.24	-0.81	0.60	4.6
Co-60	-0.26	-0.81	0.30	3.5
Cs-137	0.03	-1.0	0.60	4.1

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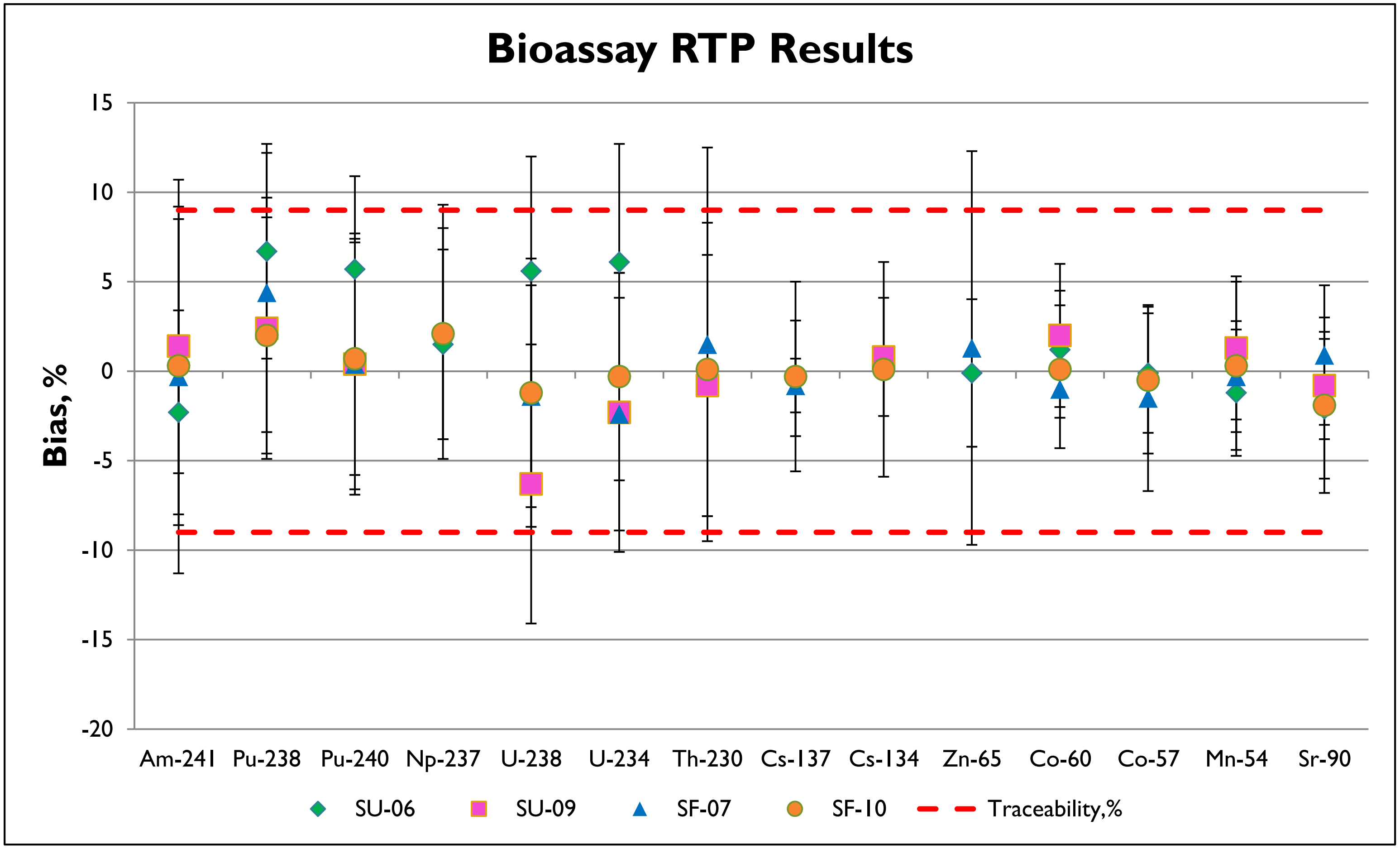
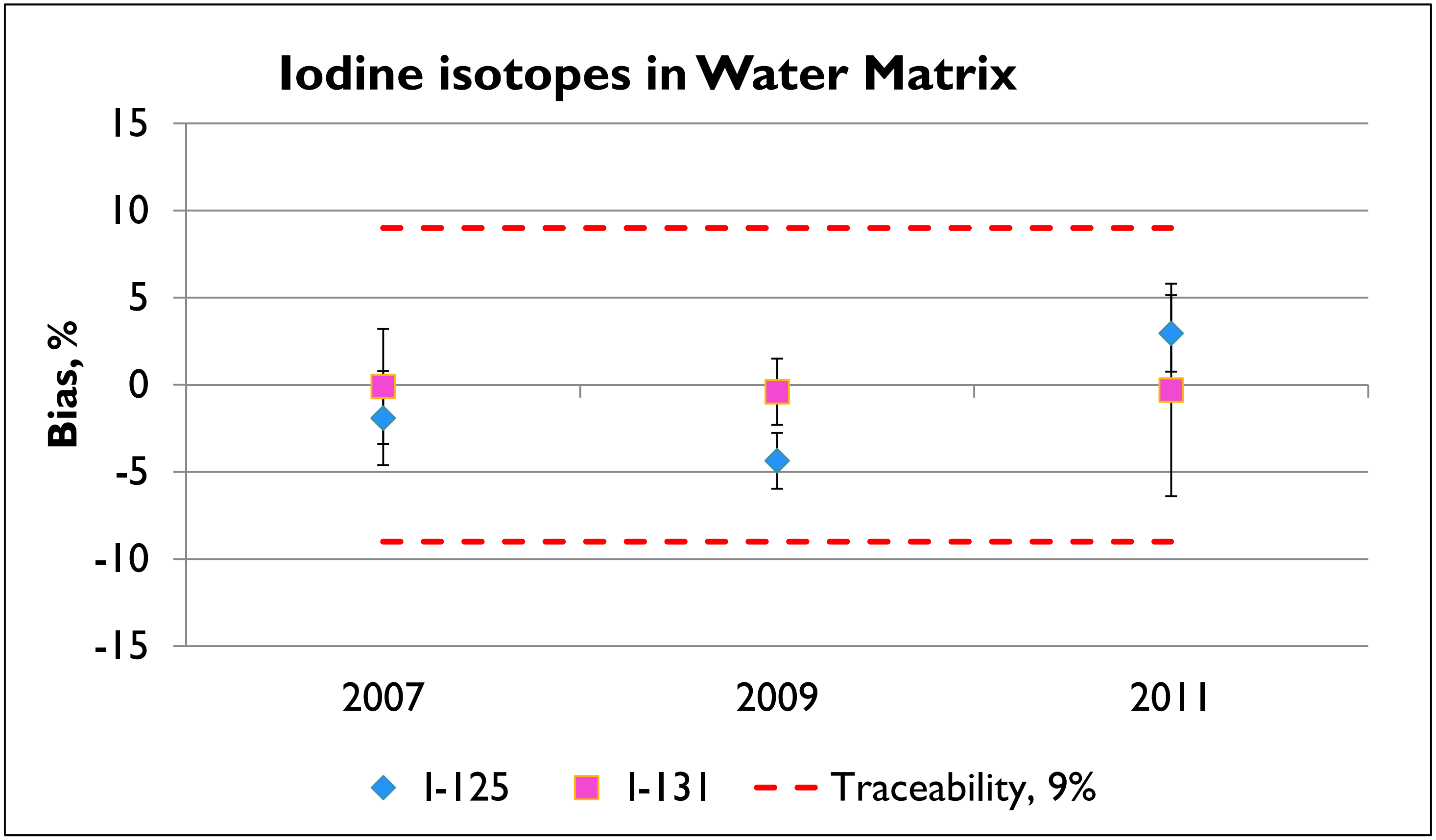


Bioassay RTP Results
Bias, %
15
10
5
0
-5
-10
-15
-20
Am-241
Pu-238
Pu-240
Np-237
U-238
U-234
Th-230
Cs-137
Cs-134
Zn-65
Co-60
Co-57
Mn-54
Sr-90
SU-06
SU-09
SF-07
SF-10
Traceability,%

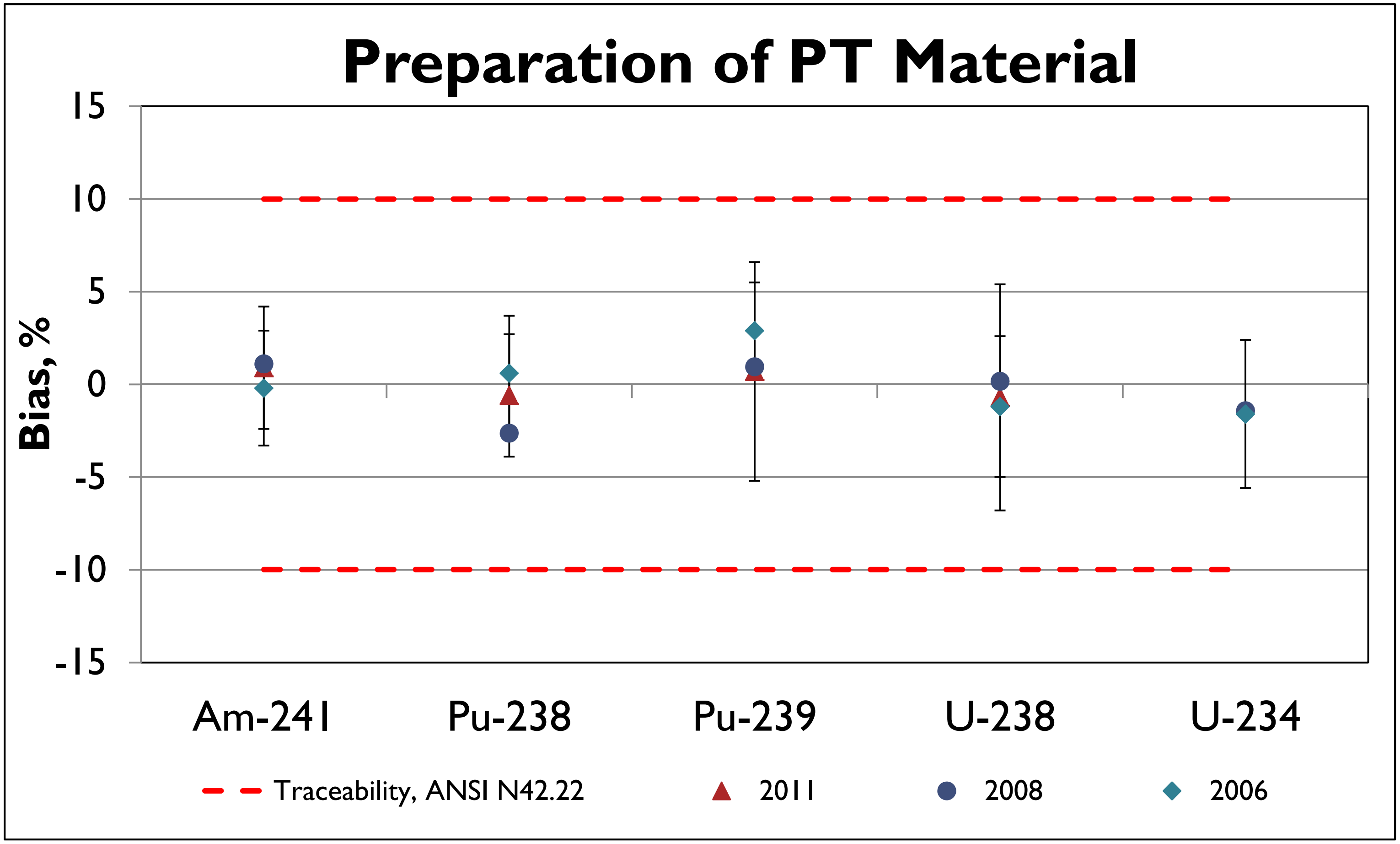
Performance for Bioassay analysis

Radionuclide	Bias average, %	Bias variation, % from	to	Average reported uncertainty, %
^{241}Am	-0.2	-2.3	1.4	8.2
^{238}Pu	3.9	2.0	6.7	6.9
^{239}Pu	1.8	0.4	5.7	6.5
^{237}Np	1.9	1.5	2.2	6.1
^{238}U	-0.8	-6.3	5.6	7.0
^{234}U	0.3	-2.4	6.1	6.7
^{230}Th	0.3	-0.8	1.5	8.8
^{137}Cs	-0.5	-0.8	-0.3	3.3
^{134}Cs	0.5	0.1	0.8	4.7
^{65}Zn	0.6	-0.1	1.3	7.6
^{60}Co	0.6	-1.0	2	3.1
^{57}Co	-0.7	-1.5	-0.1	4.2
^{54}Mn	0.0	-1.2	1.3	3.8
^{90}Sr	-1.0	-2.1	0.9	3.9



I-125 59.4 days
I-131 8.0 days

Note: There is no matrix effect or separation involved.



Traceability limits (ANSI N42.22) = minimum value over the time.

The PT samples preparation process is under control.

Preparation of Performance Evaluation Material

U.S. DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, MD

REPORT OF TRACEABILITY
Radiological and Environmental Sciences Laboratory of the US DOE
Idaho Falls, Idaho

Test Ide[illegible]	
Matrix D[illegible]	
Test Activity Range	1 $mBq \bullet g^{-1}$ to 10 $mBq \bullet g^{-1}$
Reference Time	12:00 EST, May 5, 2011

Measurement Results

Nuclide	NIST Value[2, 3]		RESL Value[4]		Difference[5]
	Massic Activity $mBq \bullet g^{-1}$	Relative Expanded Uncertainty (%, k=2)	Massic Activity $mBq \bullet g^{-1}$	Relative Expanded Uncertainty (%, k=2)	(± %)
^{238}Pu	1.824	2.5	1.808	3.3	0.9
^{239}Pu	1.344	1.8	1.352	3.3	-0.6
^{234}U	2.028	1.7	2.015	5.9	0.7
^{238}U	2.055	2.8	2.071	6.1	-0.7

Methods

	NIST[6]	Reporting Laboratory[7]
Activity Measurements	Alpha- Spectrometry	Alpha - Spectrometry

Evaluation (per ANSI N42.22)[8]

Nuclide	ANSI N42.22 Traceable	Traceability Limit (±Percent)
^{238}Pu	Yes	6
^{239}Pu	Yes	6
^{234}U	Yes	9
^{238}U	Yes	9

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Characterization of Materials Containing Radionuclides

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National Institute of Standards and Technology
Gaithersburg, MD

REPORT OF TRACEABILITY
Radiological and Environmental Sciences Laboratory of the US DOE

Test Activity

Reference Time: 12:00 EST, December 23, 2010

Measurement Results

Nuclide	NIST Value [2,3]		Reported Value [4]		Difference [5]
	Massic Activity Bq•g^{-1}	Relative Expanded Uncertainty (%, k=2)	Massic Activity Bq•g^{-1}	Relative Expanded Uncertainty (%, k=2)	(±% Bias)
^{241}Am	0.897	1.0	0.90	8.9	0.3
^{238}Pu	0.598	0.7	0.61	6.6	2.0
^{239}Pu	0.924	0.8	0.93	6.5	0.7
^{237}Np	1.338	1.3	1.366	5.9	2.1
^{238}U	1.073	0.7	1.06	7.5	-1.2
^{234}U	1.034	1.0	1.03	5.8	-0.3
^{230}Th	1.219	0.7	1.22	8.2	0.1
^{137}Cs	305.0	0.9	304	5.3	-0.3
^{134}Cs	133.9	1.9	134	6.0	0.1
^{60}Co	228.8	0.7	229	4.4	0.1
^{57}Co	34.1	1.9	33.9	4.1	-0.5
^{54}Mn	42.8	1.5	42.9	4.7	0.3
^{90}Sr	82.5	0.8	81	4.9	-1.9

Methods

	NIST [6]	Reporting Laboratory [7]
Activity Measurements	Alpha- and Beta-, and Gamma-Spectrometry Mass Spectrometry	Alpha-, Beta-, and Gamma- Spectrometry

Evaluation (per RTP Criteria) [8]

Nuclide	Traceable	Traceability Limit (±Percent)
^{241}Am	Yes	9
^{238}Pu	Yes	9
^{239}Pu	Yes	9
^{237}Np	Yes	9
^{238}U	Yes	9
^{234}U	Yes	9
^{137}Cs	Yes	9
^{134}Cs	Yes	9
^{60}Co	Yes	9
^{57}Co	Yes	9
^{54}Mn	Yes	9
^{90}Sr	Yes	9

Samples Distributed 25 October, 2010
Reporting Data Received 10 March, 2011

For the Director

Michael Unterweger,
Group Leader
Radioactivity Group
Physics Laboratory

National Institute of Standards and Technology

Conclusion

- The RTP is successful.
- The Program provides a link between NIST and DOE Service Laboratories through RESL.

Future PT Programs:
With FDA
CDC
EPA

Future addition to the RTP:

- May extend for in-vivo measurements as well.

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Acknowledgment

All Colleagues from the DOE Radiological and Environmental Science Laboratory.

THANK YOU

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