U.S. DEPARTMENT OF ENERGY | Nuclear Energy

The Radiological and Environmental Sciences Laboratory Certified Reference Materials Program

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U.S. Department of Energy
Idaho Operations Office
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U.S. DEPARTMENT OF ENERGY

Nuclear Energy

RESL Social Media Program

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Definitions

- Reference Material (RM) - Material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process
- Certified Reference Material (CRM) - Reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

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Definitions, cont.

- NIST Standard Reference Material® (SRM) - A CRM issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material
- Proficiency Testing (PT) material

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RESL ISO Accreditations

- ISO/IEC 17025:2009 General requirements for the competence of testing and calibration laboratories
- ISO/IEC 17043:2010 General requirements for proficiency testing
  - 17025 + specific requirements for PT
  - Verification of reference values, homogeneity, stability
- ISO/IEC Guide 34:2009 General requirements for the competence of reference material producers
  - 17025 + 17043 + additional rigor
  - ISO 30, 31, 35

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PT vs. CRM Requirements

Requirements/Characteristics	PT	CRM
Defined program (scheme) agreed upon by participants	X	
Certificate		X
Traceability	X	X
Verification of reference value	X	X
Homogeneity	X	X
Short-term (transportation) stability	X	X
Long-term (study) stability	X	X
Storage stability		X
Expiration date		X
Intended use/minimum sample size	X	X
Labeling requirements	X	X
Reference values stated		X
Reference values blind	X	
Internal procedure/method validation		X
External/Independent performance validation	X	

MAPEP

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RESL CRM History

- RESL has prepared custom standards for many years
  - Various DOE sites and federal agencies – radionuclides in vegetation, food, soil, water, biological material.
  - Refractory Pu
  - Kelp, bone, fish
  - Air filters for emergency exercises, IAEA
  - Egg powder, sausages, vegetation for FERN
- RESL standard philosophy
  - Blank natural matrices spiked with known quantities of NIST traceable standards with calculated reference values
  - Standard materials are tailored to specific purposes and include multiple nuclides as varying activity levels to realistically test capability

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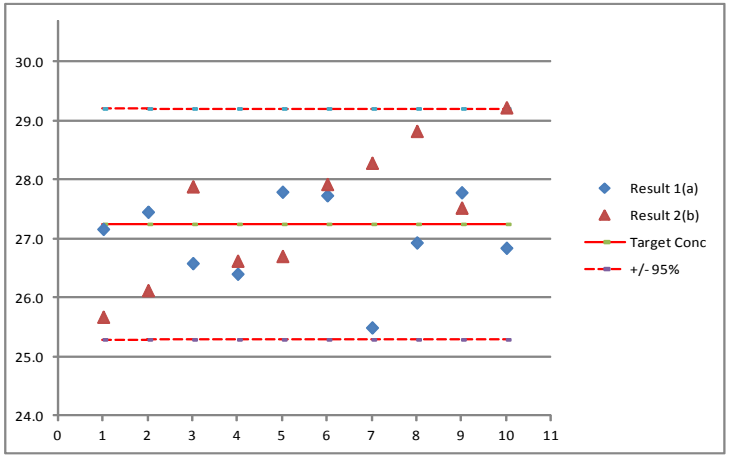
RESL's Approach to Homogeneity (Soil)

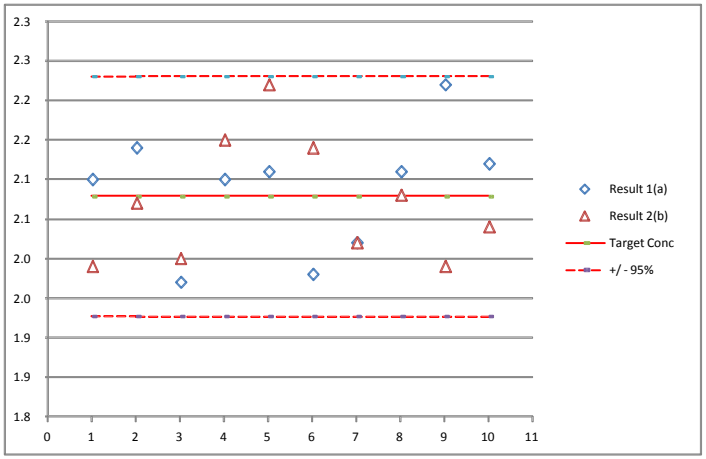
Cs-137 Homogeneity Test Data

	Target Conc.	26.500	0.80	(based on 3% target S.D.)	
Sample	Result 1(a)	Result 2(b)	(a-b)	a+b	D^2=(a-b)^2
1	27.2	25.7	1.49	52.83	2.22
2	27.5	26.1	1.33	53.57	1.77
3	26.6	27.9	-1.30	54.46	1.69
4	26.4	26.6	-0.22	53.02	0.05
5	27.8	26.7	1.09	54.49	1.19
6	27.7	27.9	-0.19	55.65	0.04
7	25.5	28.3	-2.79	53.77	7.78
8	26.9	28.8	-1.89	55.75	3.57
9	27.8	27.5	0.26	55.30	0.07
10	26.8	29.2	-2.38	56.06	5.66
				Sum D^2	24.04
Average	27.2			Cochran's Test	0.324 No Evidence for Outliers
Std. Dev	1.0			Analytical Variance (San^2)	1.202
%RSD	3.6%			S^2 (sam)	-0.258
BIAS	2.8%			Target Sample Variance (Sall^2)	0.014
				Critical Value	1.241 Sufficiently Homogeneous at the 95% confidence interval



Pu-239 Homogeneity Test Data

	Target Conc.	2.100	0.06	(based on 3% target S.D.)	
Sample	Result 1(a)	Result 2(b)	(a-b)	a+b	D^2=(a-b)^2
1	2.1	2.0	0.11	4.09	0.01
2	2.1	2.1	0.07	4.21	0.00
3	2.0	2.0	-0.03	3.97	0.00
4	2.1	2.2	-0.05	4.25	0.00
5	2.1	2.2	-0.11	4.33	0.01
6	2.0	2.1	-0.16	4.12	0.03
7	2.0	2.0	0.00	4.04	0.00
8	2.1	2.1	0.03	4.19	0.00
9	2.2	2.0	0.23	4.21	0.05
10	2.1	2.0	0.08	4.16	0.01
				Sum D^2	0.12
Average	2.1			Cochran's Test	0.447 No Evidence for Outliers
Std. Dev	0.1			Analytical Variance (San^2)	0.006
%RSD	3.6%			S^2 (sam)	0.000
BIAS	-1.0%			Target Sample Variance (Sall^2)	0.000
				Critical Value	0.006 Sufficiently Homogeneous at the 95% confidence interval



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RESL's Approach to storage stability (Shelf-Life)

Long Term Process Stability Test - Pu-238

	Time (Years)	RATIO EXP'L/KNOWN	Uncertainty RATIO	
MAW14	7.06	0.94	0.04	Stable
MAW16	6.06	0.99	0.05	Stable
MAW18				
MAW20	3.55	1.01	0.05	Stable
MAW22	2.39	0.98	0.04	Stable
MAW24	1.47	0.99	0.05	Stable

(Uncertainties reported with a coverage factor k=1)

Number of Data Points		5
Slope of Line	b_1	0.09
Intercept of Line	b_0	0.60
Std Dev of Points	s^2	0.51
Unc of Slope	$s(b_1)$	0.10
Student t-factor *	$t_{0.95,n-2}$ *	3.18

(* The Student's t-factor for n-2 degrees of freedom and p=0.95 (95% level of confidence)

Abs. Value of t_1'	$\|b_1\|$	0.09	
	$t_{0.95,n-2}*s(t_1)$	0.33	Slope is insignificant

If $|t_1|<t_{0.95,n-2}*s(b_1)$ then the slope is insignificant indicating stability.

MAPEP Water process stability test indicates that Pu-238 in RESL prepared reference water is stable for at least 7 years.

Reference: ISO G35:2006 (E) Section 8.3
ANSI N42.22

Long Term Process Stability Test - Co-60

	Time (Years)	RATIO EXP'L/KNOWN	Uncertainty RATIO	Stability Test ANSI N42.22
MAW14	7.06	1.01	0.03	Stable
MAW16	6.06	1.00	0.07	Stable
MAW18	4.55	0.98	0.04	Stable
MAW20	3.55	1.01	0.03	Stable
MAW22				
MAW24	1.47	1.01	0.02	Stable

(Uncertainties reported with a coverage factor k=1)

Number of Data Points		5
Slope of Line	b_1	0.15
Intercept of Line	b_0	0.31
Std Dev of Points	s^2	0.10
Unc of Slope	$s(b_1)$	0.06
Student t-factor *	$t_{0.95,n-2}$ *	3.18

(* The Student's t-factor for n-2 degrees of freedom and p=0.95 (95% level of confidence)

Abs. Value of b_1	$\|b_1\|$	0.15	
	$t_{0.95,n-2}*s(b_1)$	0.19	Slope is insignificant

If $|b_1|<t_{0.95,n-2}*s(b_1)$ then the slope is insignificant indicating stability.

MAPEP Water process stability test indicates that Co-60 in RESL prepared reference water is stable for at least 7 years.

Reference: ISO G35:2006 (E) Section 8.3
ANSI N42.22

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RESL CRMs

- Over the years RESL has received a number of requests for higher levels of activity in a variety of environmental matrices
- Needs have been identified in the homeland security/consequence management area
- RESL is already preparing a variety of real-world matrix standards
- RESL now has the ISO 34 accreditation to produce CRMs

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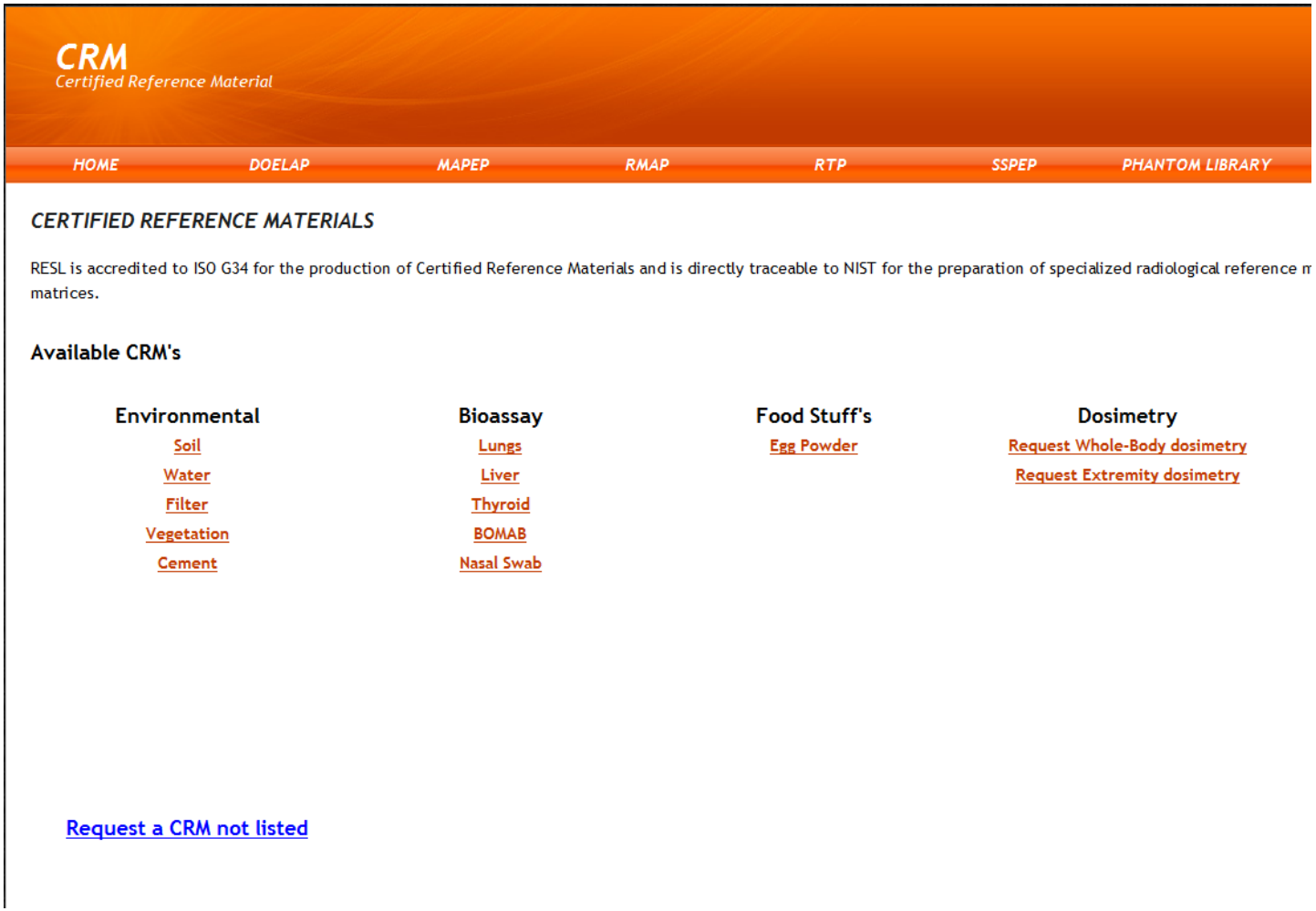
RESL Website



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Example Certificate

Certified Reference Material

Certificate of Traceability

U.S. Department of Energy
Radiological and Environmental Sciences Laboratory
1955 Fremont Avenue, MS 2112
Idaho Falls, Idaho 83415
(208) 526-8031

CRM ID: CRM Cement 0813

This Certified Reference Material (CRM) contains a known quantity of analyte(s) in a stable and homogeneous matrix. This material is intended for the calibration of instruments and for the verification of the accuracy and precision of analytical measurements. The reference value listed for each analyte is mathematically derived from a certified value traceable to a national or international standard. The traceability of the CRM is maintained through an unbroken chain of comparisons, all having stated uncertainties, calculated according to ISO and NIST Guidelines.

This CRM was prepared and the reference value(s) were verified by the Radiological and Environmental Sciences Laboratory (RESL). RESL maintains direct traceability to the United States National Institute of Standards and Technology (NIST) through successful participation in the NIST/RESL Radiological Traceability Program. RESL is accredited to ISO 17043 (2377.02) as a Performance Testing Provider, ISO 17025 (2377.01) as a Chemical Testing Laboratory, and ISO G34 (2377.03) as a Reference Material Producer by The American Association for Laboratory Accreditation.

David S. Sill
Senior Technical Manager - Chemistry

RESL CRM
Certified Reference Material

ISO/IEC GUIDE 34:2009
ACCREDITED
REFERENCE MATERIAL PRODUCER
CERTIFICATE NO. 2377.03

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USDOE-RESL CRM ATTACHMENT

CRM ID: CRM Cement 0813 **Matrix: Cement**

Reference Date: 8/15/2013

Radionuclide	Reference Value*
Am-241	4.91 +/- 0.13 E1 pCi/g
Cm-244	6.75 +/- 0.11 E1 pCi/g
Pu-238	4.89 +/- 0.12 E1 pCi/g
Pu-239	4.91 +/- 0.13 E1 pCi/g
U-234	6.67 +/- 0.15 E0 pCi/g
U-235	6.76 +/- 0.15 E-1 pCi/g
U-236	2.37 +/- 0.05 E-1 pCi/g
U-238	5.05 +/- 0.11 E1 pCi/g

*Uncertainties are reported with a coverage factor of k=1.

Special Instructions for the Proper Use of the CRM

This CRM has been determined to be homogeneous to the 100 mg sample size at the 95% confidence interval and can be subdivided for analysis.

Minimum sample size for use is 100 mg.

Storage and Handling of CRM

This CRM should be stored and used at normal laboratory operating temperatures. This CRM should be stable under normal laboratory conditions until the date listed in the Expiration Date.

If the validity of the CRM becomes questionable or technical assistance is needed please contact RESL.

Verification of Certified Reference Activity

The analyte(s) in this CRM have been verified by alpha spectrometry, liquid scintillation counting and high resolution gamma spectrometry against an independent source(s) which are traceable to the National Institute of Standards and Technology.

Hazards

This CRM material does not contain any chemical or radiological hazards in its current configuration.

Preparation Date: 8/15/2013
Certificate Issue Date: 2/5/2014
Expiration Date: 8/15/2020

END OF CERTIFICATE

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Cement CRM Verification

	1	4	8	10	16	Average	Known	Bias
U-238	51.6 ± 1.7	49.0 ± 1.8	47.5 ± 1.7	50.5 ± 1.8	50.3 ± 1.7	49.8 ± 2.1	50.5 ± 1.1	-0.014
U-234	7.1 ± 0.4	6.7 ± 0.4	6.8 ± 0.4	6.8 ± 0.4	7.0 ± 0.4	6.9 ± 0.3	6.67 ± 0.15	0.028
Pu-238	48.7 ± 1.7	48.4 ± 1.8	48.7 ± 1.9	48.1 ± 1.9	50.2 ± 1.8	48.8 ± 1.7	48.9 ± 1.2	-0.001
Pu-239	48.5 ± 1.7	48.5 ± 1.7	47.7 ± 1.8	47.8 ± 1.9	49.7 ± 1.8	48.5 ± 1.4	49.1 ± 1.3	-0.013
Am-241	49.2 ± 2.8	48.5 ± 2.9	47.1 ± 3.0	49.5 ± 2.9	46.7 ± 2.8	48.2 ± 2.4	49.1 ± 1.3	-0.018

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Conclusion

- RESL has the capability and accreditations to produce PT material and CRMs in a variety of matrices
- After a MAPEP session is finalized, RESL issues certificates for soil and water matrices. Certificates may be downloaded from the RESL website.
- Contact RESL for any specific CRM questions or needs