



Practical Considerations to Sourcing Fit for Purpose Proficiency Test Material

3rd International Conference on Radioanalytical and
Nuclear Chemistry

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Larry Jassin

6 Key Considerations



1. The Roles of Stakeholders
2. What does ISO 17043 ask of Proficiency Test (PT) participants?
3. What are the DOE/DOD QSM Requirements for Proficiency Tests?
4. Method, Matrix, Analyte: Why this is critical
5. Proficiency Test Material Considerations
 1. Assigned values?
 2. Traceability to national standards?
 3. Grading Criteria?
6. Availability of Fit for Purpose PT materials-Eckert & Ziegler Analytics-Atlanta, GA

The Roles of Stakeholders



- **Is your laboratory's data defensible?**
- **Laboratory Contracting: Project Scope/ Data Quality Objectives**
 - Define objective for each PT material ordered, change focus for each PT round
 - Sensitivity or other criteria for PT's, not just 'should participate in an established PT program
 - Ruggedness testing, matrix interferences
 - Does your current PT provider address current sample types processed by the lab
- **Laboratory Auditors**
 - Be demanding that requirements are met, do not accept that a given PT is not available without further investigation, spoiler alert, it probably is
 - Has the laboratory covered all methods, matrices and analytes
- **PT Providers**
 - Be transparent on achieving traceability, working with Laboratories on acceptance criteria and offering ISO 17043 accredited fit for purpose PT.



From ISO 17043 Annex C (Informative)

“It is the Responsibility of the participants themselves to select the appropriate proficiency test scheme and to evaluate their results correctly”

What are the DOE/DOD QSM Requirements for Proficiency Tests?



Historically, terms proficiency test samples (PT's) and performance evaluation samples (PE's) were used interchangeably, MAPEP was the standard that virtually all DOE/ DoD contracts were tied to...

proficiency testing (Definition from ISO 17043:2010)

evaluation of participant performance against pre-established criteria by means of interlaboratory comparisons

performance evaluation sample (Definition from MARLAP)

Reference material samples used to evaluate the performance of the laboratory

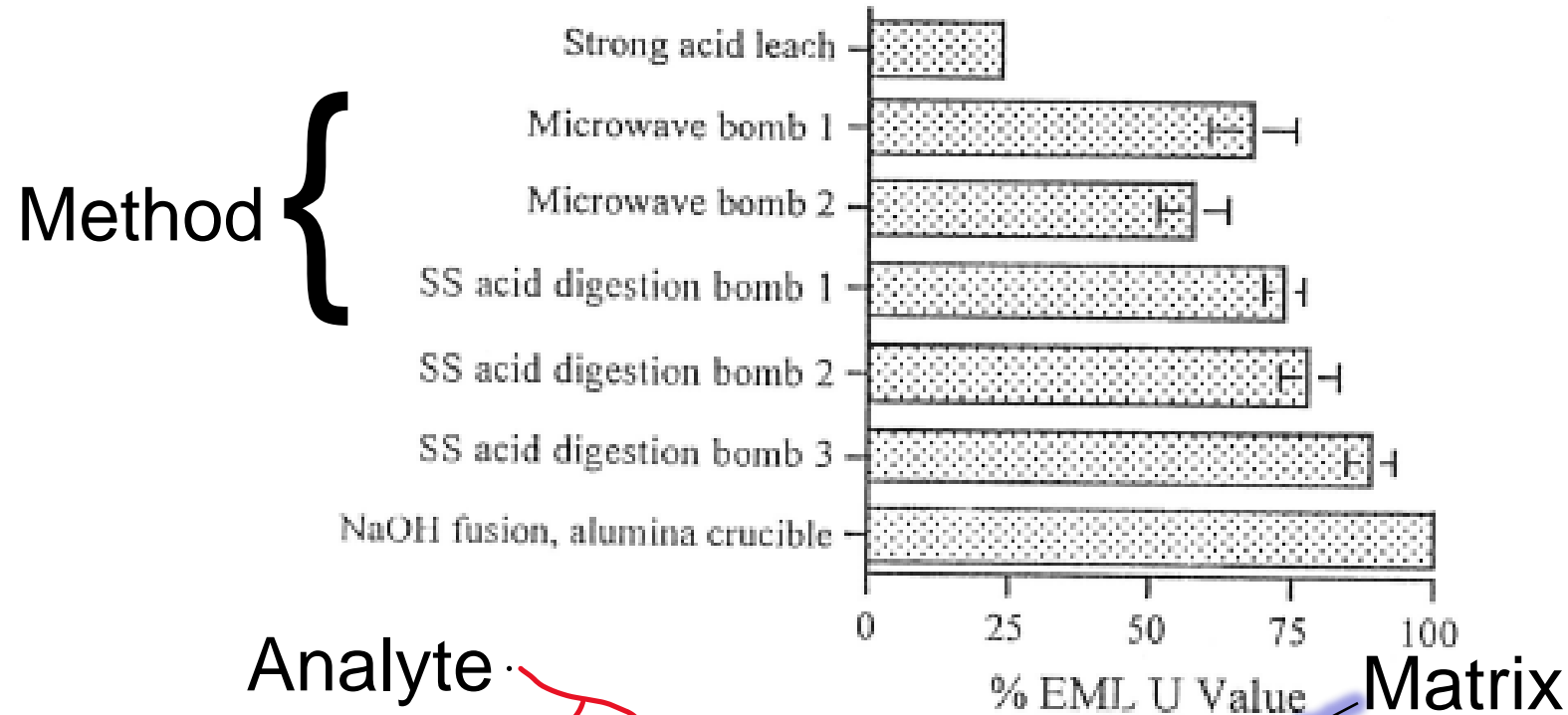
As follows...DoD DOE Consolidated Quality Systems Manual (QSM) for Environmental Laboratories 5.4 2.2.4-'**PT's must be tested and evaluated the same as regular samples.**'

PE's And PT's Why you Need Both



- **ISO 17025:2017 (E) 7.2.2.3** Laboratory methods shall be *validated* for the intended use, relevant to the customers' needs and consistent with specified requirements.
- **Performance Evaluation Materials** are best used during method validation, ruggedness and sensitivity testing-First Step in bringing a method on-line for a specific project (DQO's)
- **Then Proficiency Test Materials** test the proficiency of a laboratory's processes and personnel to produce analytical results within acceptance criteria for each contracted method, analyte and matrix combination.
Reference QSM 5.4 2.1.1

Method, Matrix, Analyte: Here's why it Matters



Results for total U, relative to the EML value, for soil 9309 based on several different dissolution techniques. Different runs on the microwave and stainless steel (SS) digestion bombs reflect various treatments of the insoluble residues.

Reference: Burnett et. al. Low-Level Measurements of Actinides and Long-Lived Radionuclides, JRNC, Vol 226, pp121-127 (1997)

How Do PT Providers Assigned Values?



- Reference Values(EZA) vs. Consensus Values(ERA)
- Traceable to NIST and other National Metrology Institutes(NMI)
- Eckert & Ziegler Analytics Approach: Establishes value through gravimetric transfer of master solutions which are either directly from NIST/NMI or calibrated using our instrumentation validated through a MAP program
- If natural matrices are used, background characterization is done prior to the introduction of spiking solutions.

Establishing Traceability to NMI's (Bq)



- National Metrological Institutes(NMI's) are coordinated through BIPM, Bureau International des Poids et Mesures
 - The BIPM is an international organization established by the Metre Convention, through which Member States act together on matters related to measurement science and measurement standards.
- ANSI N42.22 Approved August 24, 1995
 - Traceability of Radioactive Sources to NIST and Associated Instrument Quality Control
 - ANSI N42.22 Section 6 calls for the participation in a NIST Measurement Assurance Program (MAP)
 - This standard is under active review, expect consideration to include other NMI's

Intercomparison with PTB



Reference Date: 1-Jun-22

Liquid	PTB 2021-1293		EZN BE-1548 *		Tri-Carb 5110		Ratio	Tri-Carb 2500		Ratio
	PTB 2021-1293		EZN BE-1548 *		EZA p105v292			EZA p105v292		
	Activity Concentration, Bq/g	U %, k = 2	Activity Concentration, Bq/g	U %, k = 2	Activity Concentration, Bq/g	U %, k = 2	Ratio	Activity Concentration, Bq/g	U %, k = 2	Ratio
Am-241	3.99E+04	0.75	1.01E+04	3.00	1.010E+04	2.10	1.004	1.008E+04	2.10	1.001

* EZN BE-1548 produced by diluting 0.5068 g of PTB 2021-1293 up to 2.0090 g

Reference Date: 9-Mar-23

Point	Gravimetric **		HPGe DET H		Ratio
	Activity, Bq	U %, k = 2	Activity, Bq	U %, k = 2	Ratio
Am-241	4.89E+02	3.70	4.91E+02	4.00	1.003

** Made using 0.04861 g of p105v292 solution

Intercomparison with NPL-Gamma Solution



* NPL A220528 p40v292 Ref Date: 01-Oct-2022 7:00 AM EST, 5 mL Liquid in NIST FSA

Nuclide	NPL Values			H S8 & K S8		Ratio to NPL Values	H S20 & K S20		Ratio to NPL Values
	Activity Concentration, Bq / g	U (k = 2), Bq/g	Energy	Activity Concentration, Bq / g	U (k = 2), %		Activity Concentration, Bq / g	U (k = 2), %	
Cd-109	2850	35	88.0	2.803E+03	6.41%	0.984	2.827E+03	6.46%	0.992
Co-57	117.1	1.7	122.1	1.184E+02	2.68%	1.011	1.176E+02	3.02%	1.004
Ce-139	115.4	2	165.9	1.166E+02	3.12%	1.010	1.177E+02	3.15%	1.020
Cr-51	2070	30	320.1	2.068E+03	4.16%	0.999	2.045E+03	4.40%	0.988
Sn-113	417.1	6.8	391.7	4.112E+02	2.81%	0.986	4.079E+02	3.29%	0.978
Sr-85	402.6	5.9	514.0	4.034E+02	3.00%	1.002	4.050E+02	3.40%	1.006
Cs-137	509.6	7.5	661.7	5.064E+02	2.99%	0.994	5.068E+02	2.94%	0.994
Mn-54	490.0	11	834.8	4.872E+02	2.59%	0.994	4.882E+02	3.02%	0.996
Y-88	872.1	6.7	898.0	8.583E+02	2.50%	0.984	8.665E+02	3.04%	0.994
Zn-65	1136	16	1115.5	1.132E+03	2.67%	0.996	1.119E+03	2.85%	0.985
Co-60	578.6	4.2	1173.2	5.661E+02	2.65%	0.978	5.650E+02	2.72%	0.977
Co-60	578.6	4.2	1332.5	5.654E+02	2.67%	0.977	5.650E+02	2.78%	0.976
Y-88	872.1	6.7	1836.1	8.546E+02	2.64%	0.980	8.597E+02	2.60%	0.986

Example Acceptance Criteria



- The ratio of participant value and the assigned value is graded against the acceptance criteria, typically

75% < participant value/assigned value <133%

- Acceptable range established by the
 1. Uncertainty of Eckert & Ziegler Analytics measurement for the analyte/matrix, $k=1$
 2. This uncertainty is converted to a resolution and used in a table of acceptance ranges developed by the US NRC: Inspection Procedure 84725 in their effluent monitoring program (RETS)-See next slide

Example Acceptance Criteria



Resolution	Ratio
<4	0.4 – 2.5
4-7	0.5 - 2.0
8-15	0.6 - 1.66
16-50	.75 – 1.33
51-200	.80 – 1.25
>200	.85 – 1.18

- Resolution is the reciprocal of uncertainty i.e. 5 % = $1/0.05 = 20$



- Global Isotope Supply Chain
 - We are the primary calibration source supplier worldwide
 - We are diversified from medical to industrial sources
- For Over 30 Years, we have supplied PT's to the North American Nuclear Power fleet as well as recently adding IAEA, and treaty monitoring organizations
- We Operate two on-going PT programs, Hot (effluents) and Environmental cross-check programs, issued quarterly
- We are expanding our programs, one application and customer at a time, to be fit for purpose, appropriate for any given method, matrix and analyte combination

Eckert & Ziegler Analytics Expertise



- Dr. Levan Tkavadze, Nuclear Metrologist, Head of Calibration Laboratory, Head of Proficiency Test Program
 - Ph.D. Applied Physics, Idaho State University
- Dr. Marisa Alfonso, Production Manager
 - Ph.D. Chemistry Texas A&M
- Dr. Evgeny Taskaev, Research and Development
 - Ph.D. Radiochemical Neutron Activation Analysis, Institute for Nuclear Research and Nuclear Energy, Bulgaria
- Nichole Domineck, Manager, Quality Systems and Regulatory Affairs
 - MS, Quality Assurance, Southern Polytechnic State University



CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

Eckert & Ziegler Analytics
1380 Seaboard Industrial Blvd.
Atlanta, GA 30318

Fulfills the requirements of

ISO/IEC 17043:2010

In the field of

PROFICIENCY TESTING PROVIDER

This certificate is valid only when accompanied by a current scope of accreditation document.
The current scope of accreditation can be verified at www.anab.org.



R. Douglas Leonard Jr., VP, PILR SBU
Expiry Date: 27 November 2023
Certificate Number: AP-2526



SCOPE OF ACCREDITATION TO ISO/IEC 17043:2010

Eckert & Ziegler Analytics

1380 Seaboard Industrial Blvd.
Atlanta, GA 30318

Levan Tkavadze 404-352-8677 x 5195
Levan.Tkavadze@ezag.com

PROFICIENCY TEST PROVIDER

Valid to: November 27, 2023

Certificate Number: AP-2526

Radiometric

Description of Item	Properties Measured	Procedure for Establishing Assigned Value
Solid Samples	Activity	Reference Value
Gas	Activity	Reference Value
Liquid	Activity Concentration (Activity per unit volume)	Reference Value
Flowing Material	Specific Activity (Activity per unit mass)	Reference Value

Note:

- This scope is formatted as part of a single document including Certificate of Accreditation No. AP-2526.



R. Douglas Leonard Jr., VP, PILR SBU

Effluent Monitoring PT Program



Sample Analysis	Form	Activity
Fe-55	20-mL liquid	18.5 – 185 Bq/g
Ni-63	20-mL liquid	18.5 – 185 Bq/g
Gross alpha (Am-241)	20-mL liquid	0.37-3.7 Bq/g
Gross beta (Cs-137)	20-mL liquid	18.5-185 Bq/g
Gamma Isotopic (Ce-141, Co-58, Cr-51, Mn-54, Cs-134, Cs-137, Zn-65, Co-60, Fe-59)	20-mL liquid	37-370 Bq/g/nuclide
Sr-89	20-mL liquid	185- 1850 Bq/g
Sr-90		18.5-185 Bq/g
Tritium	20-mL liquid	18.5-185 Bq/g

Effluent Monitoring PT Program



Sample Analysis	Form	Activity
Gross alpha (Am-241)	Evaporated Salt (Filter Paper)	18.5-185 Bq
Gross beta (Cs-137)	Evaporated Salt (Filter Paper)	18.5-185 Bq
Gamma Isotopic (Ce-141, Co-58, Cr-51, Mn-54, Cs-134, Cs-137, Zn-65, Co-60, Fe-59)	Evp. Salt (Filter Paper), Solid (Resin), Soil, Sand Simulated Gas, Charcoal Cartridge, Point, Veg.	370-3700 Bq/nuclide
Sr-89 Sr-90	Evp. Salt (Filter Paper)	18.5- 185 Bq 1.85-18.5 Bq
I-131	Solid, Char. Cartridge	3700-37,000 Bq
Gamma Whole Body(3 nuclides)	Solid (Resin)	7400-55,500 Bq/nuclide
Noble Gases (Kr-85,Xe-133)	14 cc ampoule	<3.7E5 & 3.7E4 Bq
Noble Gases (Kr-85-Xe-133)	33 cc sphere	<3.7E6 & 3.7E5 Bq

Environmental Monitoring PT Program



Analysis	Radionuclides	Form	Activity, Bq/(L,g,-) /nuclide
Alpha/Beta	Am-241, Cs-137	Water, Filter	0.37– 11.1
Tritium	H-3	Water	74 – 555
Gamma-ray emitters	Ce-141, Co-58, Cr-51, Mn-54, Cs-134 Cs-137, Zn-65, Co-60, Fe-59	Water, Soil, Filter, Vegetation, Milk, Char. Cartridge	1.85 – 11.1 <1.85E-3/g (soil, veg)
Radiochemical	I-131, Sr-90	Milk, Charcoal Cartridge (I-131)	0.37 – 3.7
Natural Radioactivity	U-Nat, Th-Nat or Ra-228 and Ra-226	Water, Filter	0.925 – 3.7
Beta Emitters	Sr-89, Sr-90, Tc-99	Water, Soil, Filter	0.37 – 3.7
TRU's	Pu-238, 239 & Am-241	Water, Soil, Filter	0.037-3.7

Custom Blending of Matrices Available

Consider using whole sample, to reduce cost of preparation, including verifying homogeneity, especially for Alpha and Beta measurements requiring dissolution. A set of five one-gram samples, for example, may be less costly than providing 50 grams with homogeneity verified to 1 gram.

- Olivine (low background)
- Plant and Animal
- Lake and River Sediment
- Aerosols and Air Sediment
- Soil
- CaSO_4 (low background)
- SiO_2 (low background)



Olivine Matrix Available



Radioactivity concentration for Olivine and Macon Soil for comparison:

Isotope	Olivine		Macon Soil	
	Activity Concentration (mBq/g, k=2)		Activity Concentration (mBq/g, k=2)	
U-234	0.31	± 0.25 ^{a,c}	15.54	± 1.48 ^a
U-235	0.06	± 0.02 ^c	0.78	± 0.20 ^a
U-238	0.30	± 0.25 ^{a,b,c}	15.91	± 1.48 ^a
Th-232	0.15	± 0.11 ^{a,b,c}	40.7	± 3.70 ^a
Th-230	0.50	± 0.10 ^a	18.57	± 1.85 ^a
Ra-226	0.55	± 0.24 ^{a,c}	42.18	± 1.50 ^c
K-40	10.18	± 0.62 ^c	29.23	± 1.10 ^c
Cs-137			2.78	± 0.19 ^c

a Alpha Spectrometry | b ICP-MS | c Gamma Spectrometry



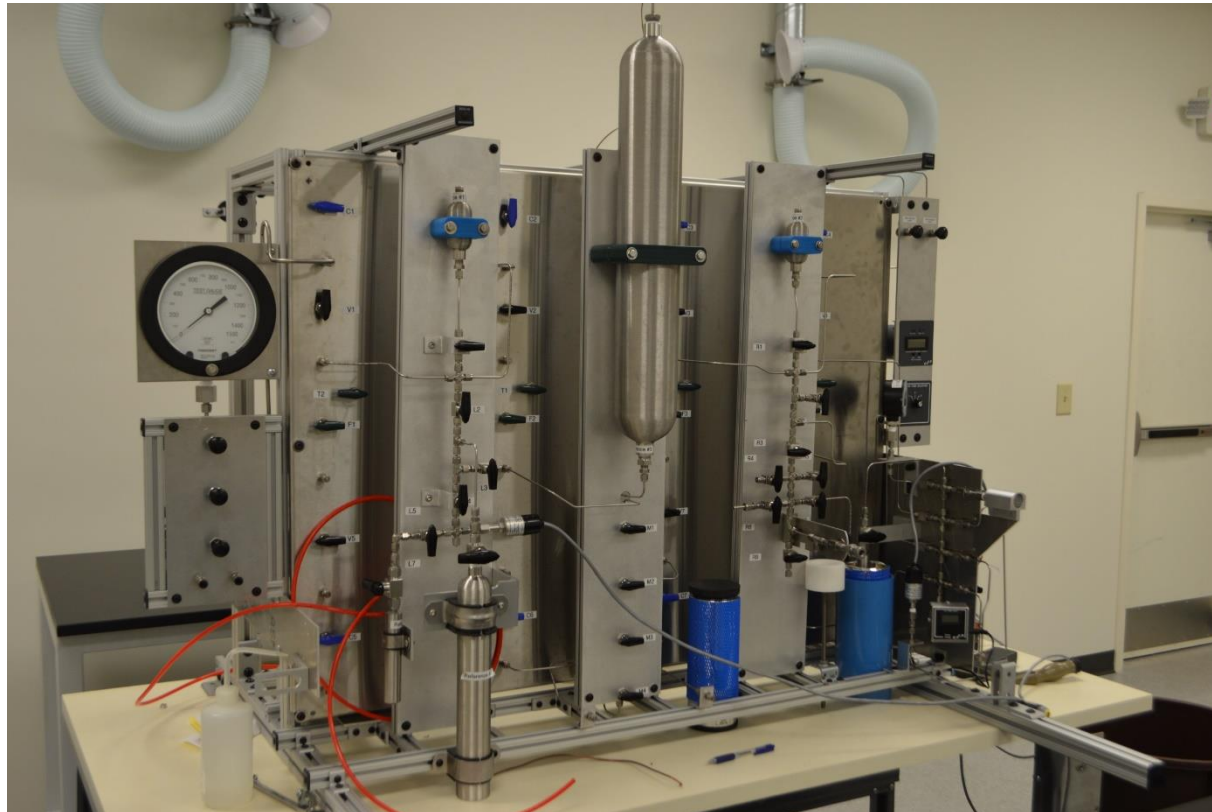
- Complex Matrices
 - Especially for low energy gammas, consider impact of
 - Homogeneity, Chemical Mixture (Z)
 - Eckert & Ziegler Analytics offers both actual and simulated concrete
 - Simulated Concrete is a blend of polyester resin, calcium carbonate and heavy aggregate to achieve desired density

Chemical Composition of heavy aggregate, (%)	
Fe	65
SiO₂	4
CaO	2.3
MgO	0.9
Al₂O₃	0.61

Density of heavy aggregate is 4.74 g/cm³.

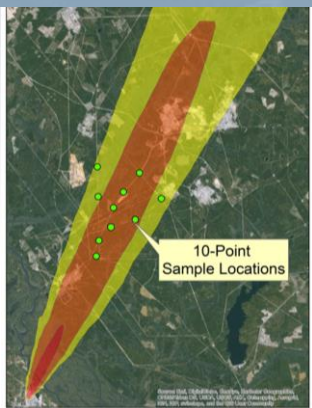
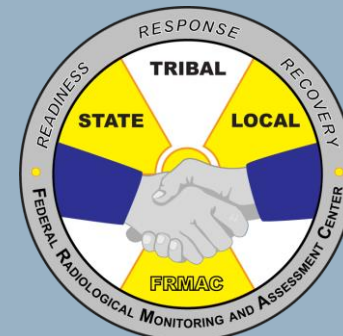
Expand Gas Capabilities

- In addition to existing Xe-133, Xe-131m, Xe-127 and Kr-85 standards and PT samples, we are adding Xe-133m and Xe-135 standards and PT samples
- Increase variety of the types of containers and carrier gases





Cobalt Magnet '22 – Lab Analysis Lessons from Search-to-CM Full Scale Exercise



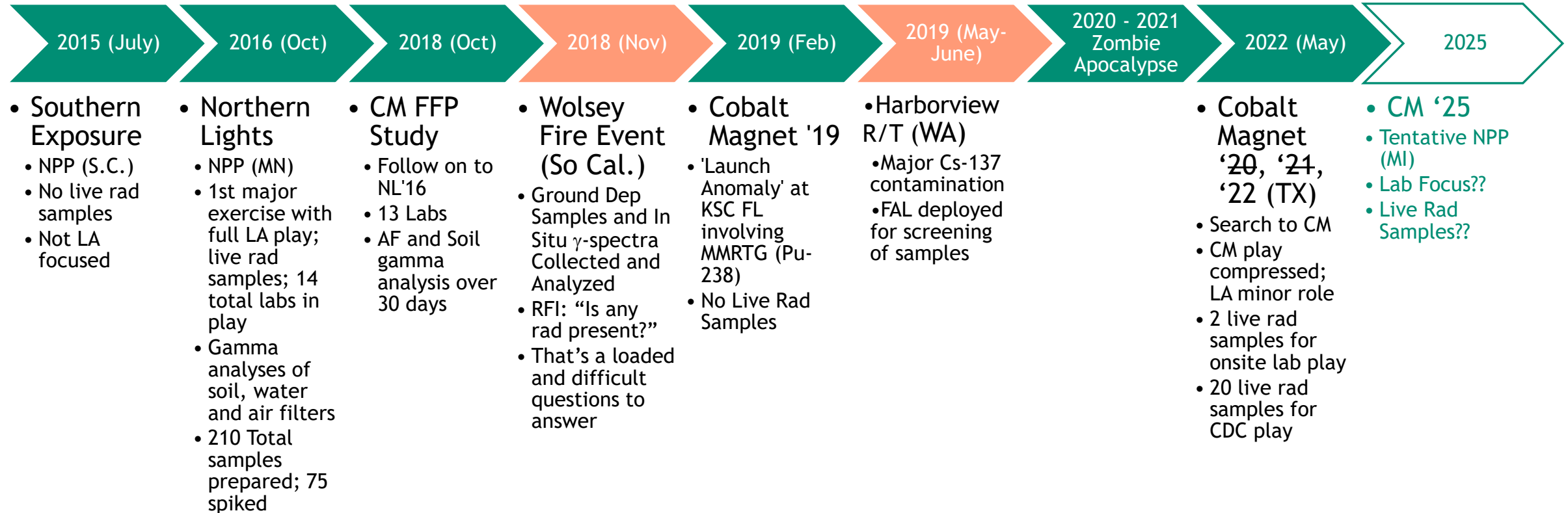
Philip C. Torretto, LLNL FRMAC Lab Analysis

LLNL-PRES-841710



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A brief history of Lab Analysis CM Exercises, Drills, Events and Studies



CM'22: "Search-to-CM" Scenario for multiple RDDs (^{60}Co , ^{137}Cs , ^{241}Am)

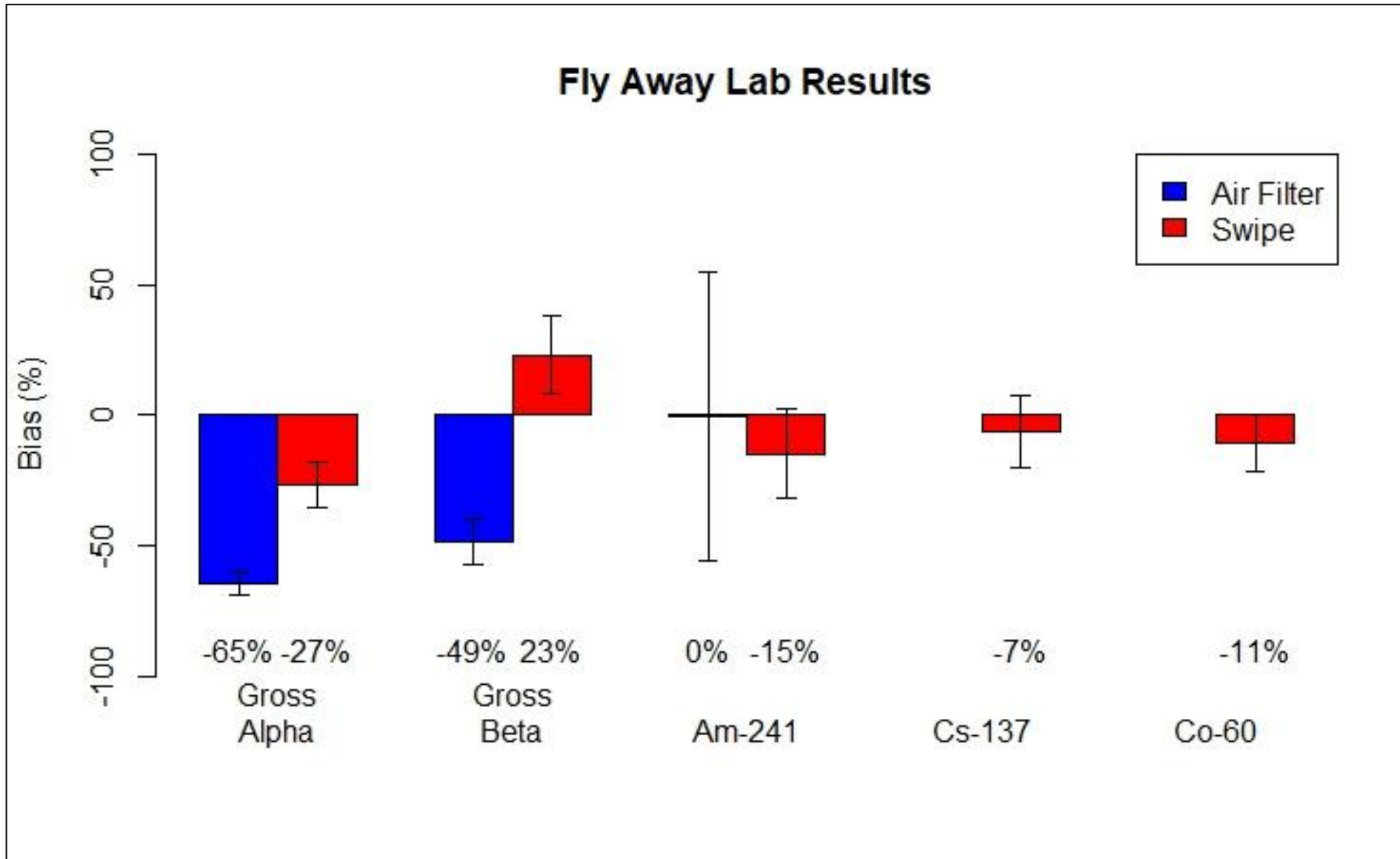


- CM22 Exercise Scenario
 - The CM22 exercise scenario will replicate the search phase of the response, detonation, and the early stages of recovery.
 - It will be a full-scale exercise with federal, state, and local partners managing a unified response to the all-hazards incident, **including integrated radiological field monitoring and sampling activities.**
 - This exercise will enable a full range of capabilities to be fielded together and examine the operational connection between major assets, discover any resource shortages associated with conducting multiple mission areas simultaneously or in close succession, and identify any challenges related to leadership.
- Lab Analysis Play
 - On Site: Mobile and Local labs were deployed and in play.
 - Prepared (E&Z Analytics) a spike swipe and air filter sample for inject in to exercise play
 - Off Site: CDC clinical bioassay lab was in play for public bioassay screening of spot urine samples.
 - 20 spiked urine samples prepared (E&Z Analytics) for inject into CDCs Inorganic and Radiation Analytical Toxicology Branch Clinical Bioassay Laboratory (I will not be presenting these results, but I heard the exercise was successful and useful for their laboratory; See Jonathan Button for more information)
 - Note: FRMAC assets including Lab Analysis were not “in play” until day 3 of the exercise.

Spikes Samples Prepared by Eckert & Ziegler Analytics



	CM22 Swipe	CM22 Air Filter
E&Z Source Number	CS5182	CS5183
E&Z Source Description	50.8 mm Diameter Glass Fiber Filter in Mylar/Tape	101.6 mm Diameter Glass Fiber Filter in Mylar/Tape
Am-241	2,650 Bq [7.16E-02 μ Ci]	53.4 Bq [1.44E-03 μ Ci]
Cs-137	118 Bq [3.19E-03 μ Ci]	2.39 Bq [6.46E-05 μ Ci]
Co-60	160 Bq [4.32E-03 μ Ci]	3.23 Bq [8.73E-05 μ Ci]

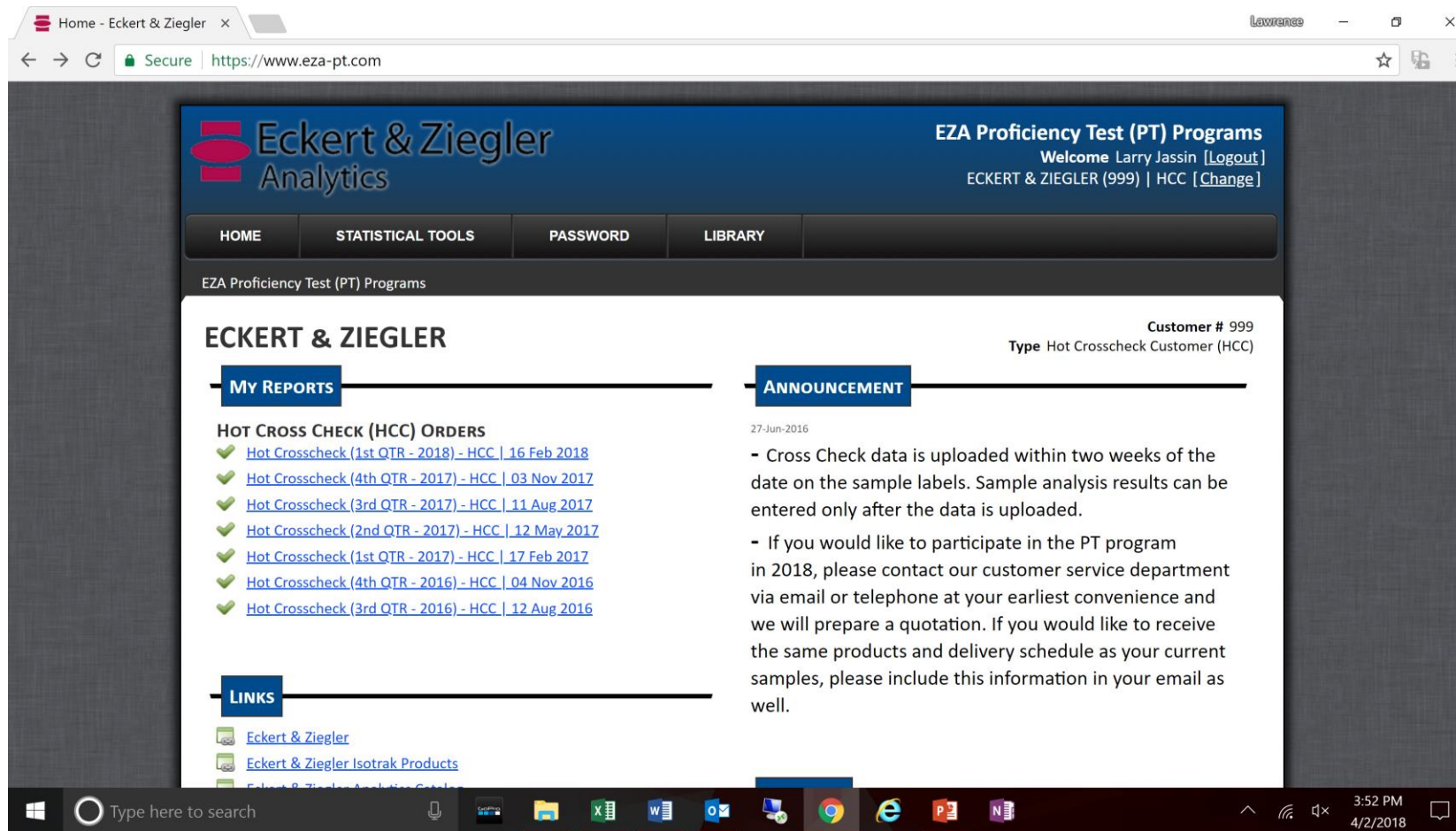


Fit For Purpose PT's



- PT's For Decommissioning?
 - H-3 in Concrete, other isotopes?
- PT's to validate Survey Meter Use (Anodized Sources customized to contain hot spots)
- Refractory Pu-239 and Pu-238 isotopes vs. leachable (method appropriate)
- Customized mass spectrometry isotope mixtures, including HEU spiked with other actinides (forensics)
- Emergency Response Food Matrices
- Fresh Mixed Fission samples
- Hydraulic Fracturing NORM (Fracking)
- Fukushima Waste Effluents

Participant Enters Method Used, will be listed for each sample prepared in final signed report



Home - Eckert & Ziegler x

Secure | <https://www.eza-pt.com>

Eckert & Ziegler Analytics

EZA Proficiency Test (PT) Programs
Welcome Larry Jassin [Logout]
ECKERT & ZIEGLER (999) | HCC [Change]

HOME STATISTICAL TOOLS PASSWORD LIBRARY

EZA Proficiency Test (PT) Programs

ECKERT & ZIEGLER Customer # 999
Type Hot Crosscheck Customer (HCC)

MY REPORTS

HOT CROSS CHECK (HCC) ORDERS

- ✓ [Hot Crosscheck \(1st QTR - 2018\) - HCC | 16 Feb 2018](#)
- ✓ [Hot Crosscheck \(4th QTR - 2017\) - HCC | 03 Nov 2017](#)
- ✓ [Hot Crosscheck \(3rd QTR - 2017\) - HCC | 11 Aug 2017](#)
- ✓ [Hot Crosscheck \(2nd QTR - 2017\) - HCC | 12 May 2017](#)
- ✓ [Hot Crosscheck \(1st QTR - 2017\) - HCC | 17 Feb 2017](#)
- ✓ [Hot Crosscheck \(4th QTR - 2016\) - HCC | 04 Nov 2016](#)
- ✓ [Hot Crosscheck \(3rd QTR - 2016\) - HCC | 12 Aug 2016](#)

LINKS

- [Eckert & Ziegler](#)
- [Eckert & Ziegler Isotrak Products](#)

ANNOUNCEMENT

27-Jun-2016

- Cross Check data is uploaded within two weeks of the date on the sample labels. Sample analysis results can be entered only after the data is uploaded.
- If you would like to participate in the PT program in 2018, please contact our customer service department via email or telephone at your earliest convenience and we will prepare a quotation. If you would like to receive the same products and delivery schedule as your current samples, please include this information in your email as well.

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4/2/2018

Conclusions and Comments



- To keep improving, it takes all stakeholders working together from the contracting organization to the auditor
- In establishing Data Quality Objectives related to selection of an analytical method, the limits of the method's performance should be established through use of Performance Evaluation samples (sample type (matrix), ruggedness testing, sensitivity testing, uncertainty)
- For continuous monitoring of laboratory proficiency, by method, matrix, and analyte (QSM), Proficiency Testing samples should be fit for purpose, traceable and fall within the scope of accreditation of the provider.

Thank you, Any Questions?

