Radioanalytical Data Validation

Issues and Solutions

Presented by:

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Purpose of Data Validation

The Presentation is Based on this Goal for Radioanalytical Data Validation:

- To produce radiochemical data of known quality that is *fully defensible in a court of law*.

Purpose of Presentation

- To inform the listener about particular QA/QC considerations in radioanalytical data validation.
- To assert the need for particular validator qualifications for adequate validation of radioanalytical data.
- To recommend DOD adoption of ANSI/ANS 41.5 (2012) as a quality affecting standard for use in the DOD radioanalytical data verification and validation process.

Radiochemistry Recognition as a Specialized Discipline

- Radiochemistry as unique, specialized and independently disciplined like metals, organics, and wet chemistry.
- Radiochemistry presents technical challenges different from the above disciplines that require specialized knowledge.

Errors in Perception of Radioanalytical Data Validation

- **Error No. 1:** A non-radiochemist with a strong stable chemistry background can be trained as a radioanalytical data validator without having performed radiochemical analysis in the lab.
- **Error No. 2:** A short training course provides sufficient qualification to validate radioanalytical data.
- **Error No. 3:** A radioanalytical data validator does not need to be as highly qualified as radiochemistry laboratory personnel, and should defer heavily to lab personnel judgment in the validation process.
- **Error No. 4:** A project validation specification "checklist" with rules for flag assignments will itself result in defensible radioanalytical data.
- **Error No. 5:** It is not necessary for the radiochemistry validator to check the lab's calculations, or other in-depth technical aspects of the data generating process, in a comprehensive fashion.
- **Error No. 6:** Automated processes of validation of radiochemical data negate the need for experienced radiochemists as data validators.

Radioanalytical Data Validation

Some of these ideas have been presented as acceptable practice by data validation companies in this EMDQ forum in the recent past.

Validata would like to present our perspective on these errors based on 25 years of data validation experience.

Radioanalytical Data Validation – Realistic Perceptions of Expertise Required

Must Build the Case for Defensibility

- Substantiate lab results from raw data
- Verify and validate practices used to obtain results
- Consider usability for defined DQOs

Recognize Every Element of Potential Indefensibility

- When science of radiochemistry as set forth by industry standards is not achieved.
- Professional judgement based on knowledge of individual radioanalytical disciplines.

Examples of Fundamental Issues Recognizable by Validator with Lab Expertise - Rather than Prescriptive Approaches

Inappropriate Calibration:

Matrix matching in calibration source is not adequate. Examples:

- Headspace in a Ra-226 analysis by gamma in solids
- Gross alpha calibration source is on wrong filter type for the project
- Unmatched quench LSC

Geometry of a sample does not match that of the calibration source. Examples:

- The precipitate on a disc in liquid scintillation vial instead of dissolved in cocktail in the scintillation vial.
- The one liter sample bottle does not match the 1 liter Marinelli beaker geometry of the calibration source.

Source material does not meet industry standards. Examples:

- The standard certificate does not contain the ANSI required information for uncertainties and contaminants.

Validator Laboratory Expertise

Incorrect Identifications:

- Identify a spectrum peak as not that of the target analyte reported from the peak data, based on energy distribution – Examples:
  - Beta-max and beta-average do not match target analyte characteristics in liquid scintillation analysis.

Interferences:

- Example: Alpha peaks upfield in an alpha spectrum indicate contamination of known nuclides which also have peaks in the ROI of the target analyte.

Resolution:

- Spectrum does not have adequate resolution – Examples:
  - Gamma spectrum peaks present and overlapping
  - Background data are not adjusted when spectrum region of interest is manually changed due to spectral resolution issues.

Validator Laboratory Expertise

Possess a Breadth of Experience in Multiple Methodologies – Example:

- Essential to be knowledgeable in multiple preparations and multiple instrument types.

Demonstrate a Thorough Understanding Of Radiochemistry Principles – Example:

- Analyte relationships to time and their means of decay, including progenitors and progeny and the effects on analytical processes.

Validator Laboratory Expertise

Possess Depth of Experience in Radiological Calculations – Examples:

- Must develop key calculations on project-specific basis.
- Are there calculation errors at times in radiochemistry data packages? Absolutely **yes**! Examples of calculation errors Validata has found in past data packages:
  - **Errors in Calibration Standards**
  - **Errors in Analyte Quantitation**
  - **Errors in Uncertainty Calculation**
  - **False Positives**
- Combined Standard Uncertainty – the essential data qualifier.
- Minimum Detectable Activity (MDA) and Critical Value (CV) or Decision Level – the meanings and assessment in validation.

Alpha Isotopic Calculations

Concentration of Analyte at Sampling Date = A /(TrY*Abd*C*D*U)

where:

S	=	Analyte Gross Counts
B	=	Background Counts
T	=	Sample Count Time (min)
X	=	Background Count Time (min)
A	=	Net Counts per minute (CPM)
U	=	Sample aliquot (g, L)
TrY	=	Tracer Yield
C	=	2.22 DPM to pCi
BR	=	Branching Ratio
D	=	Decay
Eff	=	Detector Efficiency

Sample/Analyte Info	
Analyte	U-238
Sample	298159001
Detector	173

Calculation, Alpha Isotope Activity in Sample

S	=	139	
B	=	0	
T	=	43200	sec
X	=	60000	sec
A	=	0.193	CPM
TrY	=	0.7560	
%Abd	=	100.000	
D	=	1.0000	
U	=	0.502	g or L
Eff	=	0.267067	
Alpha Isotope Conc., pCi/g	=	**0.86**	**pCi/g or L**
Reported	=	0.878	

Blue fields are input fields (except this one)

RPD = -2.28%

Decay and Yield Data

Tracer dpm correction			Tr Yield		Sample analyte decay correction		
DPM	ref date	half-life(d)	Tr Cnts	Bkgnd Cnts	smpling date	count date	half-life (d)
4.8842	12/18/2009	2.52E+04	694	0.72	3/20/2012	5/4/2012	1.64E+12
	Corrected Tracer dpm	4.7689	Tr Yield =	0.756			

Counting Uncertainty Calculation for Alpha Isotope

Count Uncertainty (CU) @ one sigma level = SQRT((S/T²)+(B/X²))/(TrY*Abd*C*D*U*Eff)

CU	=	0.0728	pCi/g or L
Reported CU	=	0.0745	pCi/g or L

RPD = -2.32%

Combined Standard Uncertainty Calculation for Alpha Isotope

TPU = (SQRT(SQRT(S/T²+B/X²))²+(S/T-B/X)²*((Eff Err/Eff)²+(Aliq Dev)²+(TrY Err)²))/(TrY*Abd*C*D*U*Eff)

% Eff Err =	0.5154
Aliq. Dev. =	0.00652
Yield Err =	0.065

CSU	=	9.33E-02	pCi/g or L
Reported CSU	=	9.55E-02	pCi/g or L

RPD = -2.27%

Critical Value Calculation for Alpha Isotope

Critical Value (CV) = (1.645*SQRT(B/X*2/T)/(TrY*Abd*C*D*U*Eff)

CV	=	0.00E+00	pCi/g or L
Reported CV	=	0.00E+00	

RPD = 0.00%

Minimum Detectable Concentration Calculation for Alpha Isotope

MDC = (3.29*SQRT(B/X^2+B/X*T)+2.71/T)/(TrY*Abd*C*D*U*Eff)

MDC	=	1.67E-02	pCi/g or L
Reported MDC	=	1.71E-02	pCi/g or L

RPD = -2.18%

Verification of Cal Source Decay Correction

Cal Source ID	Cal Source Nuclide	Cal Source Ref. Date	Nuclide Half Life	Cal Source Count Date	CPM on Count Date	g Cal Source X recovery	Detector Efficiency	Calc. DPM/mL on Ref Date	DPM/g on Ref Date	RPD
1216-C	CM-244	6/19/2008	6614.7	12/18/2011	3.32E+02	9.9820E-01	3.41E-01	1.112E+03	1.110E+03	0.17%

Validator Laboratory Expertise

Utilize Knowledge of Vendor Software Tools and Limitations – Examples:

- Calibrations – Calibrated instrument vs calibrated methods.
- Background corrections – Instrument background vs "appropriate blank" (MARLAP)
- Interference and compensation techniques – Recognizing interference, its source, its effects, and how to qualify data.

Validator Laboratory Expertise

Utilize Professional Confidence to Challenge an Analytical Result when Necessary - for Example:

- Question the analysis, when unsure, until it is shown to be correct with adequate response from the laboratory.
- Stand your ground when response(s) is/are not adequate.
- The necessary level of professional confidence needed in radiological data validation is derived largely from radiological laboratory experience – provides a means to judge from direct experience, not only theory.

Corrections to the Perception Errors

- **Correction 1:** A radioanalytical data validator needs a strong background in the radiochemistry lab - ANSI/ANS 41.5 (2012)
- **Correction 2:** A radioanalytical validator must be able to understand the lab activities and confirm or appropriately qualify results independently and without reliance on lab personnel expertise. This is true data validation.
- **Correction 3:** A short training course to be a "certified radioanalytical data validator" without radiochemistry laboratory background is not sufficient qualification - ANSI/ANS 41.5 (2012)
- **Correction 4:** Checklists and flagging rules will assist in data validation but cannot replace knowledge of analytical processes and result calculations, for example.
- **Correction 5:** Data validation involves a comprehensive reconstruction of technical laboratory practices, including calculations.
- **Correction 6:** Automated processes cannot replace the mind of a seasoned radiochemist.

Conclusions

Radiochemistry is a Self-Actualized Entity Like Other Disciplines of Analytical Chemistry

- Radioanalytical data validation requires sufficient training, experience, and knowledge.
- Data defensibility and usability are dependent upon expertise that cannot be attained without sufficient radiochemistry laboratory background.
- Request for the DoD to take the lead to adopt the ANSI standard, **verification and validation of radiological data for use in waste management and environmental remediation,** ANSI/ANS 41.5 (2012) to help accomplish this needed advancement.

Radiochemistry Laboratory Expertise

Kevin Harmon

- Founded Validata Chemical Services, Inc.
- Served as Technical Director, Project Manager, and Client Services Director
- 25 Years of Data Validation Project Management
- Managed the successful completion of over 300 data validation projects of all types in the USA and abroad
- Consulted to EPA Regions, DOE, many DOD prime contractors

Expertise Needed for Radiochemistry: from a Project Management Standpoint

- As we have shown, a radiochemistry data validator must have sufficient radiochemistry laboratory background.
- Laboratory experience is key for both radiochemistry and stable chemistry validators.
- Must have experience on the particular instrument performing the relevant environmental methods.

Validator Laboratory Expertise

- **Right now, there are no DOD Standards for Radiochemistry Data Validators.**
- **Or if they do exist, they are not implemented on the validation level.**
- As a result, the Radiochemistry laboratory experience of Data Validators varies *very significantly* between different companies - from those with ***no*** Rad. Lab background to ***many years*** of experience.
- **For Example:**

Data Validation Company No. 1

Radiochemistry Validation Group Lead

(Title given by company in recent EMDQ presentation.)

Qualifications (As Posted on LinkedIn)

August 2007 – April 2009 (1 year 9 months)

Maintained and ran ICP, ICP-MS, and CVAA.

April 2009 – Present (6 years)

Data validation including metals, general chemistry, physical chemistry, bacteriological, and radiochemistry.

Laboratory Experience Summary:

- No radiochemistry laboratory experience
- Less than 2 years of inorganic laboratory experience

Data Validation Company No. 2

Radiochemistry Validation Group Lead

- Highly experienced Radiochemist with over 20 years of Radiochemistry laboratory experience in instrumental analysis, analytical laboratory support and data validation for remediation efforts.

Experience Summary:

- Radiochemistry Laboratory Experience – 22 years
- Gamma Spectrometry Experience – 20 years
- Alpha Spectroscopy Experience – 15 years
- Gas Proportional Counting – 20 years
- Kinetic Phosphorescence Analysis by Laser (KPA) – 5 years
- Stabilized Assay Meter (SAM) – 2 years
- Radiation Detection Meters – 22 years
- Miscellaneous Laboratory Equipment – 24 years
- Rad. Lab Data QA/QC Review –15 years
- Rad. Lab Manager – 5 years
- Radiological Data Validation Experience – 5 years

Radiochemistry Laboratory Experience

	DV Company #1 Rad. Lead (Years)	DV Company #2 Rad. Lead (Years)
Radiochemistry Laboratory Experience (All)	0	22
Gamma Spectrometry Exp.	0	20
Alpha Spectroscopy Exp.	0	15
Gas Proportional Counting	0	20
Kinetic Phosphorescence	0	5
Stabilized Assay Meter (SAM)	0	2
Radiation Detection Meters	0	22
Radiological Lab Data Internal QC Review	0	15
Radiological Lab Manager	0	5
Non-Rad. Lab. Equipment	<2 Years	24
Radiological Data Validation	6	5

Validator Laboratory Expertise

When data validation companies are left to create their own standards:

- Some companies tend to use highly experienced people.
- Others do the opposite, using people who have never even ventured into a radiochemistry laboratory.

There is a **Huge** disparity between different companies.

Is your radiochemistry data being validated correctly???

- **The Key Question is:** What minimum radiochemistry laboratory experience should a data validator have to be considered qualified to validate radiochemistry data?

Validator Laboratory Expertise

Why don't we rely on the experts for that answer? There are 2 primary sources of radiochemistry expertise, both of which are cited heavily in the DOD QSM 5.0:

- MARLAP and ANSI

- The DOD QSM 5.0 References MARLAP - 73 Times
- DOD QSM 5.0 References ANSI – 12 Times
- MARLAP References ANSI – 34 Times

If the QSM references these industry documents for principles to be used in its radiological laboratory data generation and validation, it should also implement the minimum experience recommendations the documents intend to accompany the use of those principles.

Radiochemistry Laboratory Experience

	DV Company #1 Rad. Lead (Years)	DV Company #2 Rad. Lead (Years)	ANSI Minimum Qualifications for Rad. Data Validator (Years)
Radiochemistry Laboratory Experience (All)	0	22	3
Gamma Spectrometry Exp.	0	20	3
Alpha Spectroscopy Exp.	0	15	3
Gas Proportional Counting	0	20	3
Kinetic Phosphorescence	0	5	3
Stabilized Assay Meter (SAM)	0	2	3
Radiation Detection Meters	0	22	3
Radiological Lab Data Internal QC Review	0	15	0
Radiological Lab Manager	0	5	0
Non-Rad. Lab. Equipment	<2 Years	24	0
Radiological Data Validation	6	5	2

Validator Laboratory Expertise

ANSI also Requires:

- Familiarity with the DQO process and statistical concepts, inferences, interpretation, and tests in the area being validated. (Specific Instruments, Sample Prep., Data Validator Training).

- A B.S. or B.A. in Chemistry or Related Physical Science or Engineering. We agree with that minimum education requirement.

We agree these requirements are a good minimum baseline for radiochemistry data validation.

Validator Laboratory Expertise

Are your DOD radiochemistry data being validated correctly???

Without the minimum lab experience listed above, the experts say, No!

- Validata would like to recommend that the DOD adopt and implement the ANSI experience requirements for Radiochemistry data validators ANSI/ANS 41.5 (2012).
- It is a good starting point to ensuring competence of Radiochemistry data validators.
- It at least gives minimum requirements as to lab background needed to understand and properly handle radiochemistry data.

Validator Laboratory Expertise

We would like to call on DOD Prime Contractors to:

- Check the qualifications of all individuals performing data validation – even from ***major***, established DV companies.
- Insist on at least 3 years of Rad. lab experience or 2 years of stable chemistry experience.
- Insist on at least 5 years combined laboratory and data validation experience – in any discipline.

It is your duty as Prime Contractors to ensure you receive quality data validation services from experienced professionals.

Validator Laboratory Expertise

- Ultimately, the best solution would be to certify radiochemistry data validators.
- Right now, the validation companies are left to regulate themselves.
- As we have seen, some companies put heavy emphasis on radiological laboratory experience.
- But others put no emphasis on it.
- In our view, and in the view of MARLAP and ANSI, these companies cannot correctly perform data validation. But they have been validating DOD radiochemistry data for years.

Validator Laboratory Expertise

- Are there past DOD radiological data that have been incorrectly validated due to the use of unqualified data validators?
- Unfortunately, yes. But there are good solutions.
- We invite the military branches to meet with us to discuss further.

Validata Chemical Services, Inc.

About Us:

- Validata has adheres to the highest standards of expertise in the data validation industry without any enforced standards.
- This is true of radiochemistry and stable chemistry data validation – Our stable chemistry staff has even more experience.
- 25 years successfully performing data validation services.
- Validata is a Small, Disadvantaged, Woman-Owned Business

Contact Information

We welcome you to contact us for the best in data validation, audits and other QA/QC consulting services.

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