CAVEATS ON PROTOCOLS FOR CHEMICAL YIELDS GREATER THAN 100%

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Quality Systems for Radiochemical Testing

- DoD/DOE Quality Systems Manual[1]
  - Tolerance limits for chemical yields, 30% - %110
- ANSI/ANS 41.5 Verification and Validation of Radiological Data[2]
  - Requires qualification of results with greater than 110% chemical yield
- MARLAP[3]
  - Suggests a yield estimate that is much greater than 100% cannot be accurate and indicates a problem

[1]Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, July 2013.
[2]ANSI/ANS-41.5-2012 “Verification and Validation of Radiological Data for Use in Waste Management and Environmental Remediation, February 2012.
[3]NUREG-1576, EPA 402-B-04-001B, NTIS PB2004-105421, Multi-Agency Radiological Laboratory Analytical Protocols Manual, July 2004

Chemical Yields Greater than 100%

- Actual chemical recovery cannot be greater than 100%.
- It has been reasoned that application of a recovery factor greater than 100% creates a negative bias on the results.
- This reasoning has led some labs and projects to a protocol of not applying recovery corrections to reported values when the chemical recovery is greater than 100% but otherwise meets the acceptance criteria.
- This practice deserves re-examination.

Typical causes of excessive chemical yields

- Interferences with the carrier or tracer used
  - For interferences, the protocol of not applying a recovery correction is appropriate.
- Efficiency or geometry differences that are different from the initial calibration
  - For detector efficiency or geometry differences when carriers are used the protocol of not applying a recovery correction is appropriate.
  - For detector efficiency or geometry differences when isotopic tracers are used, as in alpha spectroscopy, the results may be more accurate if the recovery factor is applied.

Quantitation Using Tracers

- Efficiency is assumed to be the same for the tracer and the analyte and the efficiency term cancels out in the calculation.
  - $DPM = \frac{CPM\ sample}{\cancel{efficiency}} \times \frac{DPM\ tracer}{CPM\ tracer/\cancel{efficiency}}$
- Quantitation is based on the tracer standard value and the efficiency is used only for the purpose of determining chemical yield.
- Application of the chemical recovery factor will eliminate any biases introduced from efficiency drifts or geometry differences.

When to Apply Tracer Recoveries that are Greater than 100%

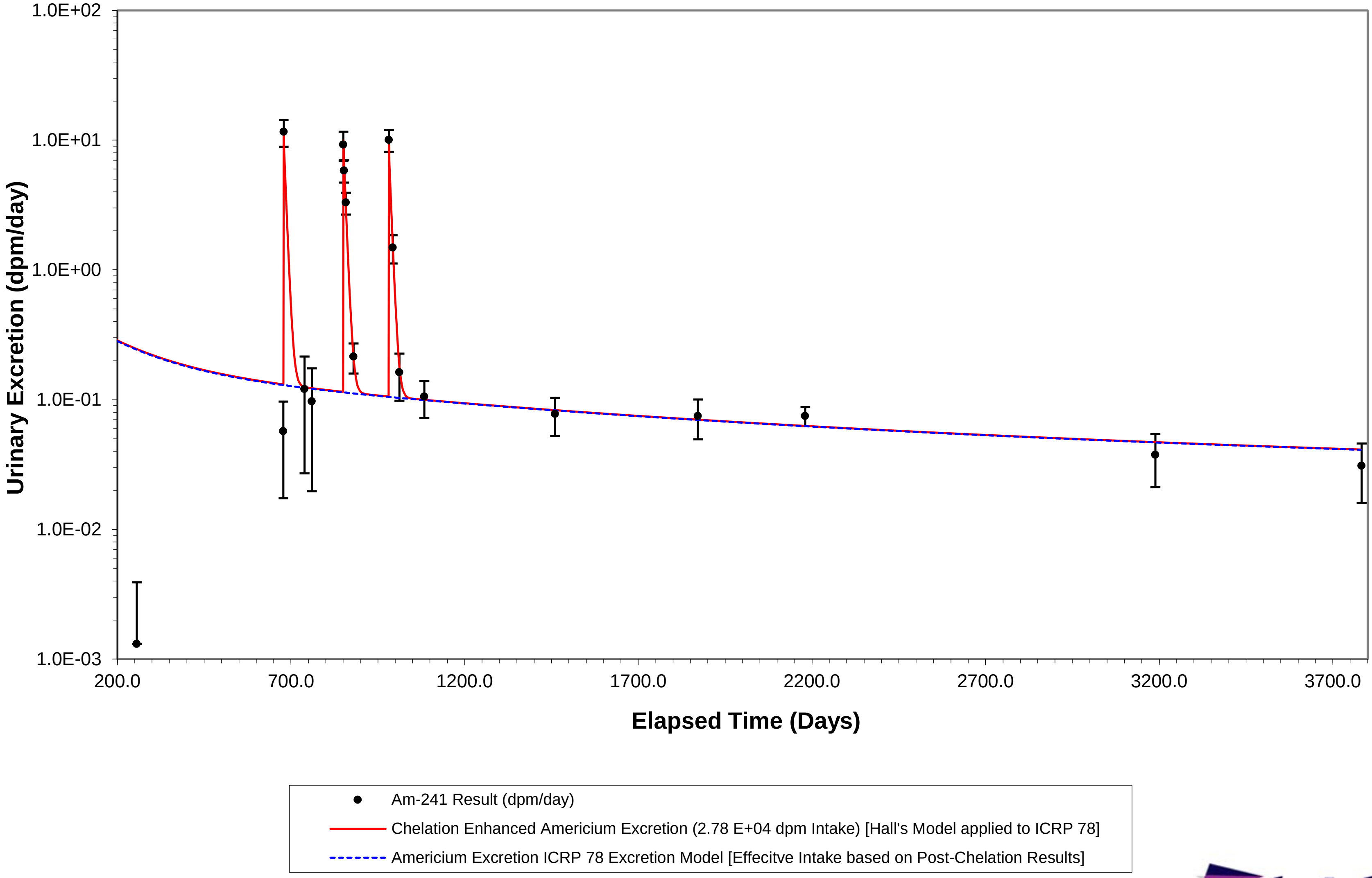
- Evaluate the cause of the problem
  - Don’t just assume it is due to statistical variation
- If it suspected that the tracer or carrier is present in the unspiked sampled, application of the recovery factor should not be applied to the calculation.
- If the review suggests that the efficiency changed since the initial calibration, either due to detector drifts or geometry differences, the recovery factor should be applied to allow the quantitation to based on the tracer value and eliminate the bias.

Detector Efficiency Drifts

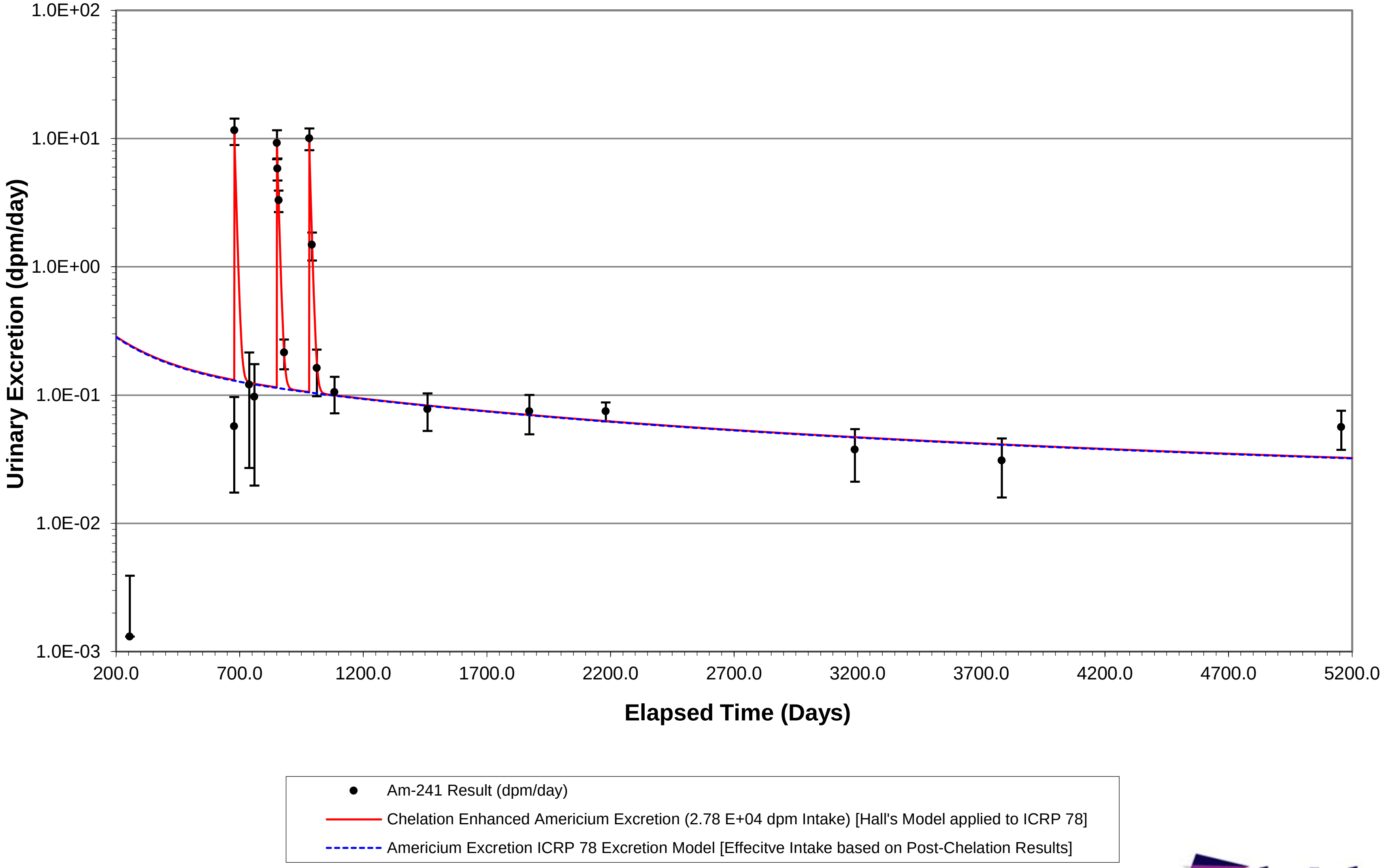
> Sometimes not identified by the laboratory because of their use of control charts to monitor instrument performance rather than the use of tolerance charts and limits.
  - Control Charts revise the limits based on the new mean and standard deviation for each check run.

> Tolerance limits relative to the <u>initial calibration</u> value should always be used to keep the actual efficiency in tolerance to the calibration value and based on Measurement Quality Objectives (MQOs).

Example:. Americium Urinary Excretion Fit with ICRP 78 Model



Example: Americium Urinary Excretion Fit with ICRP 78 Model



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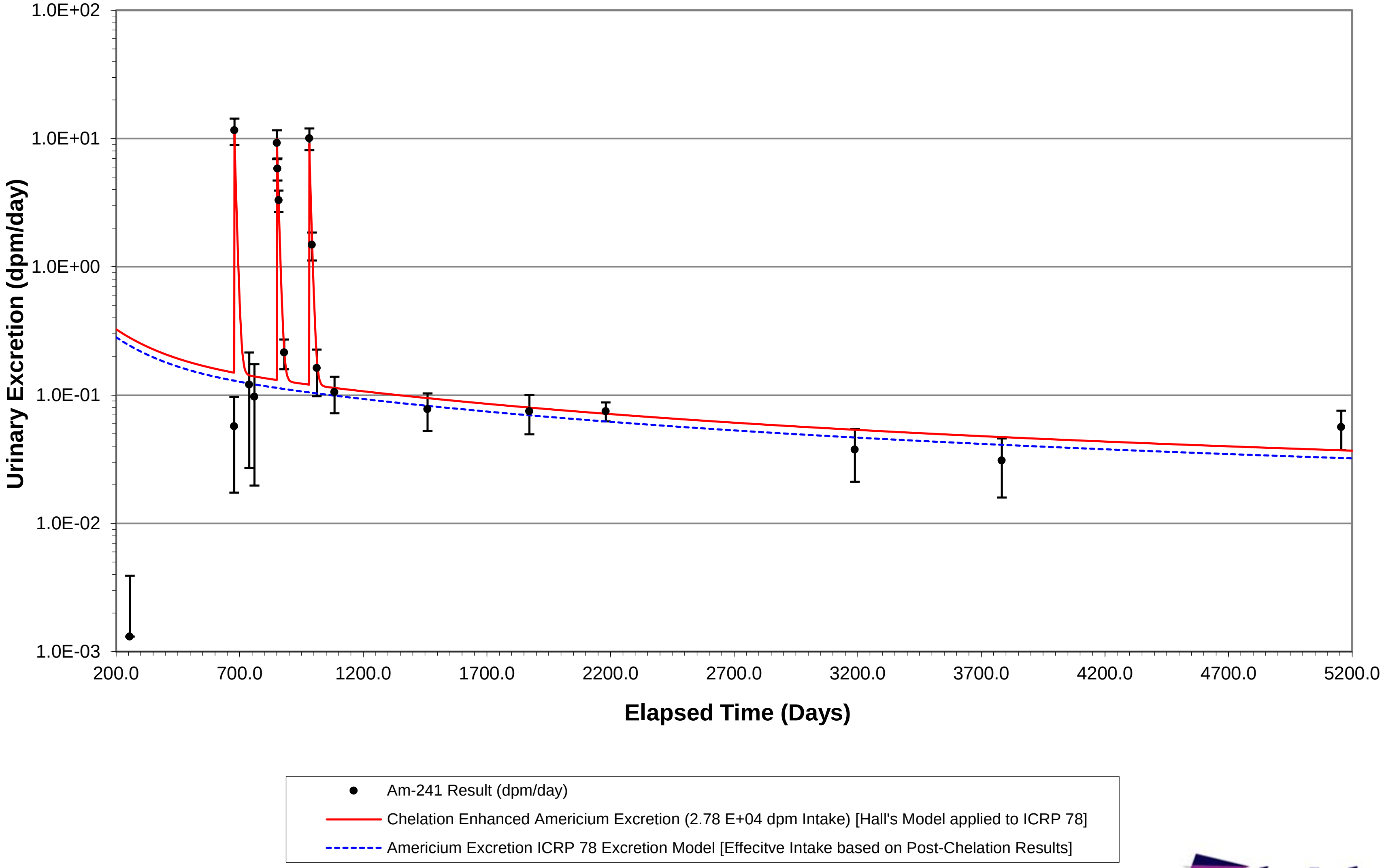

Real Life Example – Am-241 Bioassay

> AMERICIUM 241 5.64E-02 +- 1.9E-02 dpm/sa 104%

- "In the initial count, tracers recovered high for the sample, blank and LCS. The batch was recounted and results are within acceptance limits."

Comparison of Reported Results to Recovery Corrected Results

	Detector	Tracer Yield	Quoted Efficiency	Tracer Calculated Efficiency	Calculated Result (dpm)	Expected Result (dpm)	Quoted LCS Recovery	Calculated LCS Recovery
Sample 1	ALP73		0.185	0.215	0.0485			
Tracer		116.03%	0.185	0.215				
LCS	ALP78		0.282	0.331	0.0871	0.104	98%	83.72%
Tracer		117.26%	0.282	0.331				
Sample 1 R	ALP441		0.319	0.333	0.0541			
Tracer		104.33%	0.319	0.333				
LCS R	ALP443		0.311	0.357	0.0851	0.104	94%	81.89%
Tracer		114.83%	0.311	0.357				

Calibration Efficiency vs Check Efficiency

	Detector	Quoted Efficiency	Past 3 Month Average Efficiency Check	% Difference	Tracer Yield
Sample 1	ALP73	0.185	0.212	15%	116%
LCS	ALP78	0.282	0.327	16%	117%
Sample 1 R	ALP441	0.319	0.331	4%	104%
LCS R	ALP443	0.311	0.339	9%	115%

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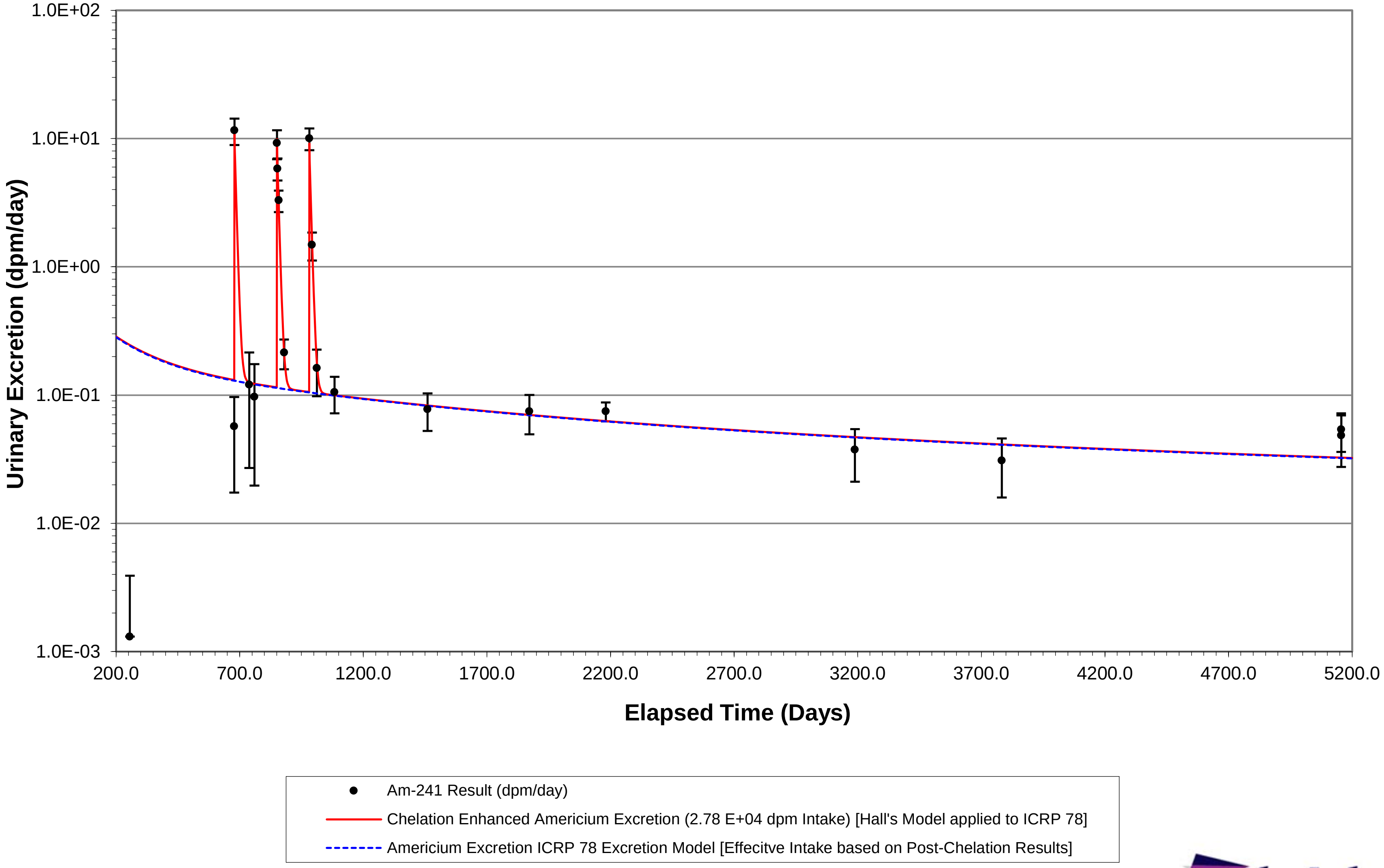
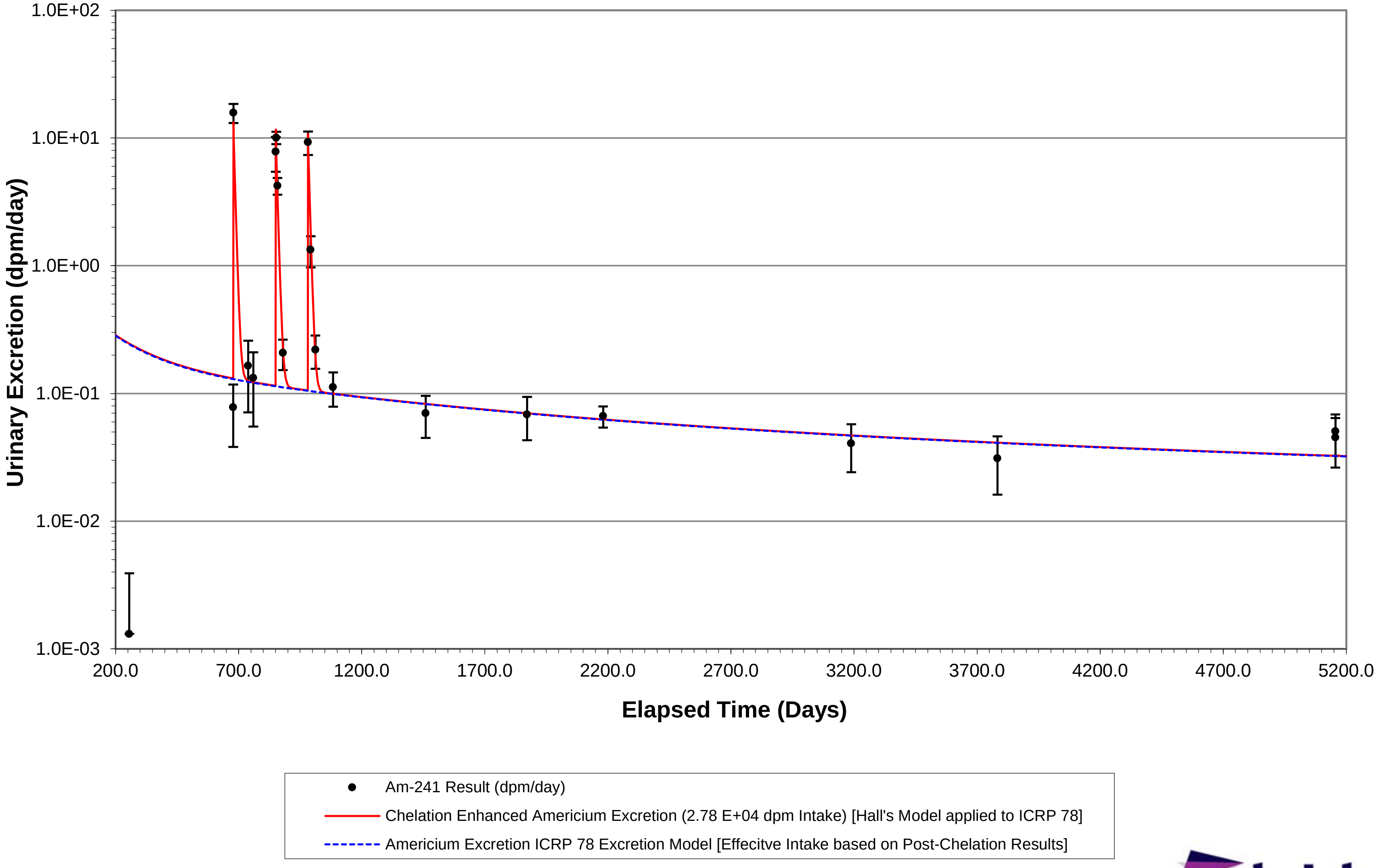
Example. Americium Urinary Excretion Fit with ICRP 78 Model



Example. Americium Urinary Excretion Fit with ICRP 78 Model Volume Normalized



Conclusions / Recommendations

- Evaluate the cause of tracer recoveries greater than 100%
- Do not apply recovery correction when the cause is due to the tracer or carrier is present in the unspiked sampled.
- Apply recovery correction when the cause is either due to detector drifts or geometry differences.
- Use Tolerance Limits and Charts rather than Control Limits and Charts for instrument performance monitoring.

Thank You

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