

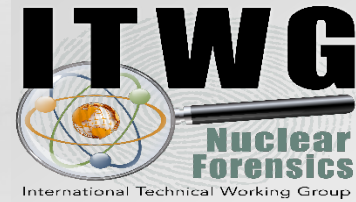
# CMX-6 Data Review Meeting – Day 1

**4<sup>th</sup> – 6<sup>th</sup> June 2019**  
**Warsaw, Poland**

**Jon Schwantes**  
**Olivia Marsden**

**Pacific Northwest National Laboratory, Richland, Washington, USA**  
**AWE, Aldermaston, UK**

# Welcome to CMX-6 DRM



## ▶ Welcome-

- Institute of Nuclear Chemistry and Technology, Warsaw, Poland

## ▶ Venue information

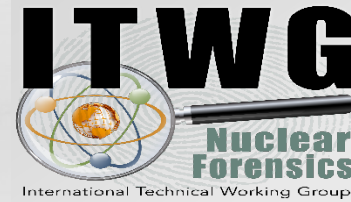
# Data Review Meeting Overview

- ▶ **Day 1**
  - **Welcome and introductions**
  - **24 hour report results**
  
- ▶ **Day 2**
  - **1 week report results**
  - **Introduction to 2 month report results**
  - **DRM evening dinner**
  
- ▶ **Day 3**
  - **2 month report results**
  - **Reveal/backstory**
  - **Lessons learned and future exercises**
  - **CMX-6 after action report schedule**
  - **Close out**

# Background

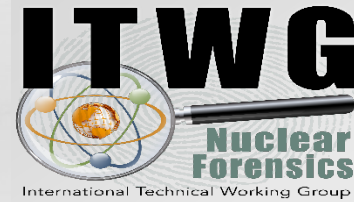
- ▶ **6<sup>th</sup> Collaborative Materials Exercise (CMX-6)**
  - **1999-2000 Pu Oxide Powder, Round Robin 1**
  - **2000-2002 HEU Powder, Round Robin 2**
  - **2009-2010 HEU Metal, Round Robin 3**
  - **2014-2015 LEU pellets and powder, CMX-4**
  - **2016-2017 LEU pellets, theoretical injects, CMX-5**
  
- ▶ **Goal of CMX's:**
  - ...to improve international Technical Nuclear Forensics capabilities, cooperation, and communication between practitioners through the discovery, development, and sharing of best practices**

# ITWG Exercise Philosophy



- ▶ **Each Laboratory's results are held in confidence**
- ▶ **A summary of exercise results is published (AAR-After Action Report)**
- ▶ **Uses “real world” samples – not reference materials**
- ▶ **Scenario based exercises with reporting times balancing the needs of the investigation with the limitations of methodologies**
- ▶ **Designed to target questions of both a (1) legal and (2) national security nature**
  1. Is the material radioactive? Dangerous? LEU? HEU? Illegal to possess?
  2. Can we identify the origin? Can we include or exclude it from other materials?
- ▶ **Use the Graded Decision Framework to accurately communicate exercise results**

# Benefits of participating in CM exercises



- ▶ **Put the lab capability into practice**
- ▶ **Utilise different techniques to answer questions that may be asked when nuclear material is seized by law enforcement agencies**
- ▶ **Compare results to other laboratories (although NOT a proficiency test)**
- ▶ **Exchange information on nuclear forensics with other laboratories**

## CMX-6 Participant Statistics

- ▶ **Congratulations!!! You are all part of the largest Collaborative Materials Exercise in the 24 year history of the ITWG**
  
- ▶ **Sixth Collaborative Materials Exercise (CMX-6)**
  - **22** laboratories will have completed the exercise plus one virtual participation
  
  - Round Robin 1 (**RR1**), **6** participating laboratories
  - Round Robin 2 (**RR2**), **10** participating laboratories
  - Round Robin 3 (**RR3**), **9** participating laboratories
  - Collaborative Materials Exercise 4 (**CMX-4**), **16** participating laboratories
  - Collaborative Materials Exercise 5 (**CMX-5**), **20** participating laboratories



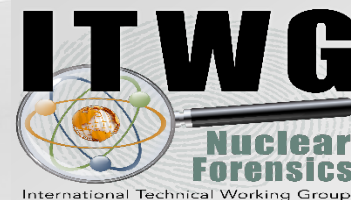
# Caveats & Assumptions for CMX-6



- ▶ **CMX-6 is the 4<sup>th</sup> consecutive “paired comparison” exercise**
  - Supporting technical comparisons without the need for a NF Library
  
- ▶ **It is assumed that all participating laboratories have a satisfactory Quality Control and Quality Assurance program**
  
- ▶ **Exercise materials used:**
  - Have well known process history and laboratory analysis, but are not “certified” materials (when possible, mean values of all CMX-6 laboratory results are provided to illustrate consensus answers)
  - CMX-6 has a conventional forensics part of the exercise (not included since RR2)

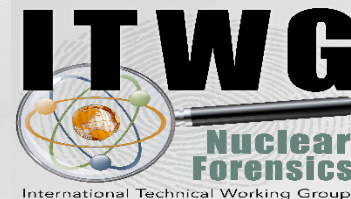


## Purpose of CMX-6



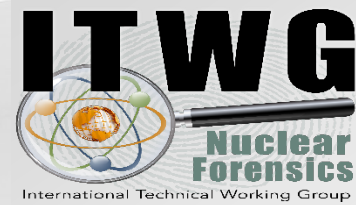
- ▶ **Improve TNF tools and best practices**
- ▶ **Assist labs to develop new and improve existing TNF capabilities**
- ▶ **Enhance decision making process by optimizing the ITWG Graded Decision Framework (GDF)**
- ▶ **Emphasize the utility of Group Inclusion/Exclusion (GIE) decisions related to TNF evaluations**
- ▶ **Address questions of both legal and National Security nature**

# Ground Rules for CMX-6



- ▶ **Technical Learning Experience / not a performance test**
  - ITWG is not a governing body and does not have the “right answer”
  - Our job is to facilitate a discussion about best practices
- ▶ **Data Review Meeting is open to only participants or persons that have helped facilitate the exercise.**
- ▶ **All meeting discussions are to be held in confidence and not shared outside of this community**
- ▶ **Individual data points will be referenced using the code name for that laboratory at all times.**
- ▶ **Meeting participants are asked to refrain from references to data in a way that may divulge the identity of laboratories other than their own**

# Participant Introductions



- ▶ **Name**
- ▶ **Country**
- ▶ **ITWG exercises experience**
- ▶ **What was your lab hoping to gain from participation?**





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## Operation Celestial Skónis



6<sup>th</sup> Collaborative Materials  
Exercise (CMX-6) of the  
Nuclear Forensics International  
Technical Working Group  
(ITWG)

**Jon Schwantes & Olivia Marsden**  
Co-Chairs ETG, ITWG



## 20 years of Materials Exercises

- 1999-2000 Pu Oxide Powder, RR1 (6)
- 2000-2002 HEU Powder, RR2 (10)
- 2009-2010 HEU Metal, RR3/CMX-3 (9)
- 2014-2015 LEU pellets and powder, CMX-4 (16)
- 2016-2017 LEU pellets, CMX-5 (20)
- 2018-2019 PuF<sub>4</sub> powder, Ce & DU Metal, CMX-6 (23)



## Background



## Purpose:

...to improve international Technical Nuclear Forensics capabilities, cooperation, and communication between practitioners through the discovery, development, and sharing of best practices

## Goal:

Evaluate the state of practice and identify emerging technologies



## CMX-6 Participants

- 23 participants
- 19 shipped standard exercise materials
- 3 shipped exercise materials w/o Pu
- 1 virtual participant\*

- Australia
- Azerbaijan
- Brazil\*
- Canada
- France
- Germany
- European Commission
- Hungary-U
- Israel-U
- Japan-U
- Kazakhstan
- Korea
- Moldova
- Poland
- Romania
- Russia
- Singapore
- South Africa
- Sweden
- Switzerland
- UK
- Ukraine
- USA



## Ground Rules / Design

- Each Laboratory's results held in confidence
- Summary of exercise results is published
- Uses "real world" samples (i.e., not PT)
- Scenario based exercises with real-world reporting
- Designed to target questions of both a (1) legal and (2) national security nature
  - Is the material radioactive? Dangerous? LEU? HEU? Illegal to possess?
  - Can we identify the origin? Can we include or exclude it from other materials?

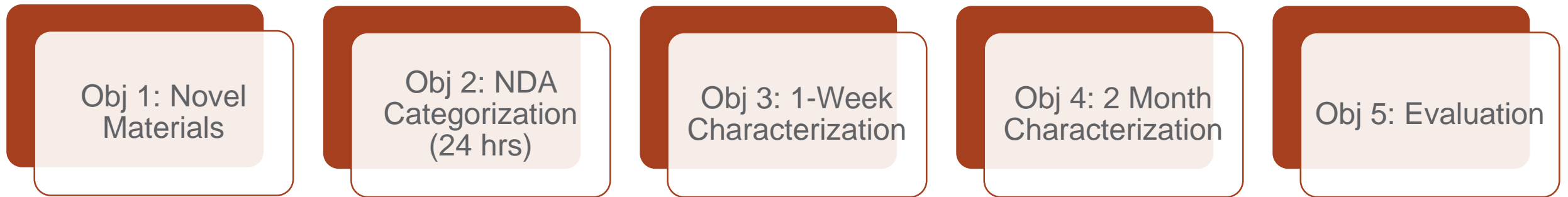
### Key CMX-6 Design Features

- Include Traditional Forensics
- Utilize novel materials
- Blind receipt



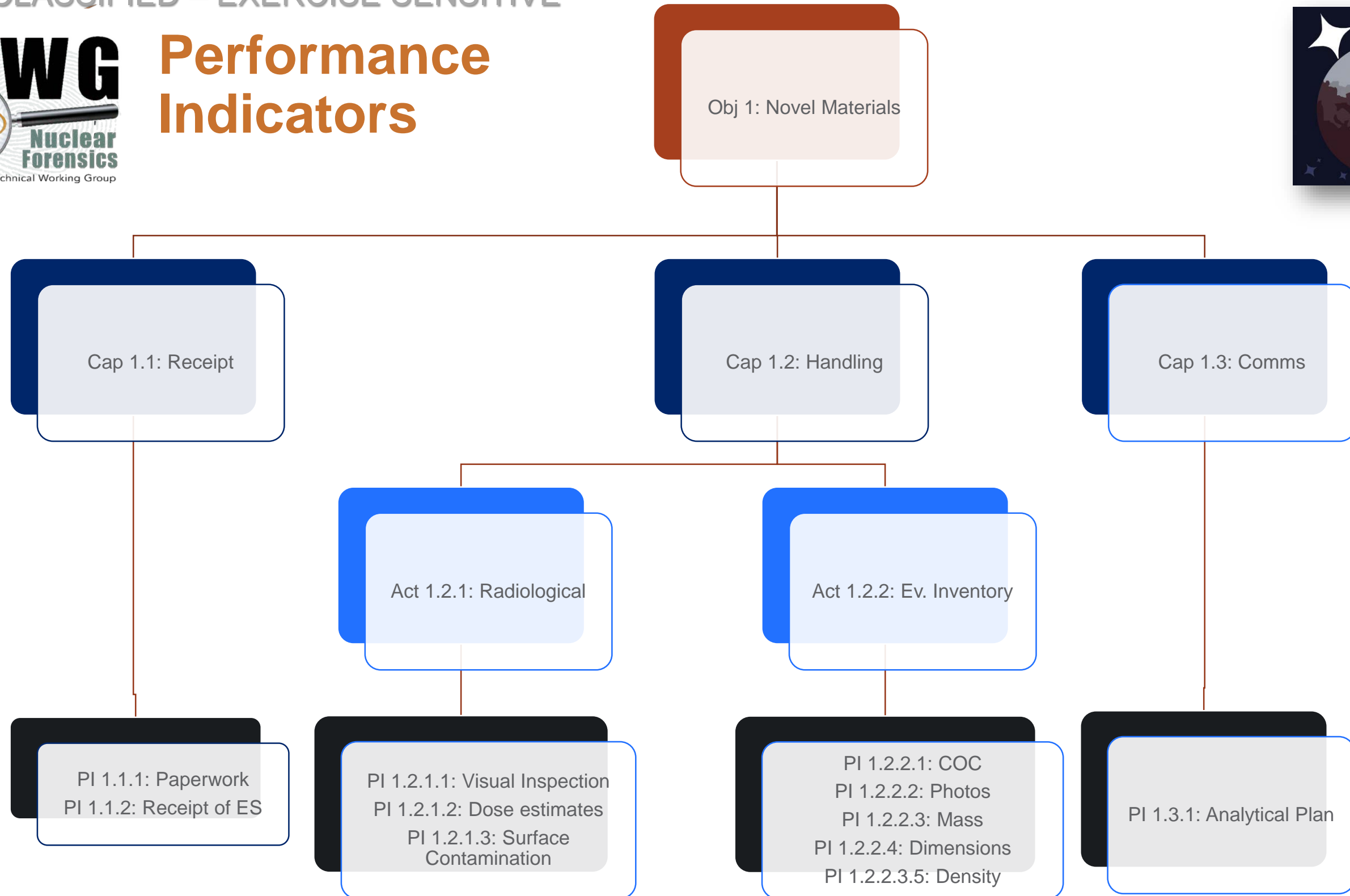


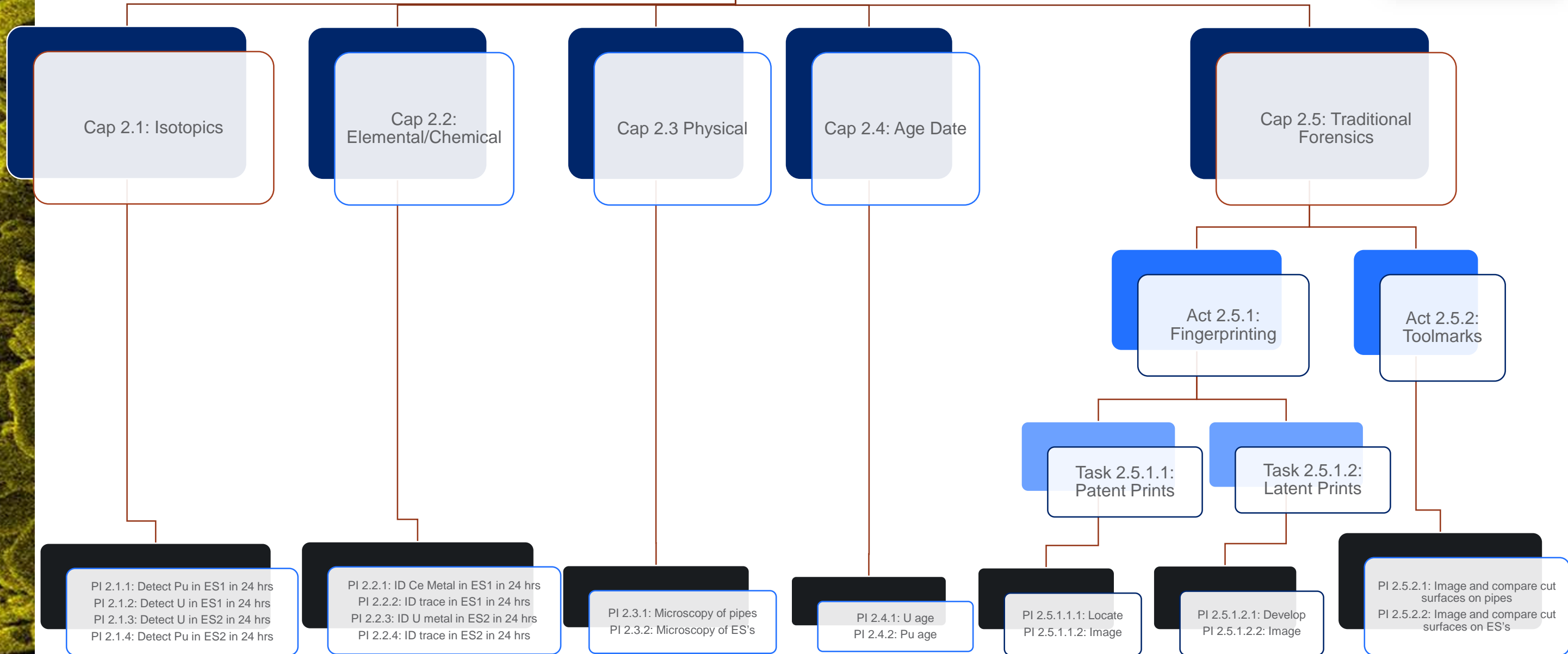
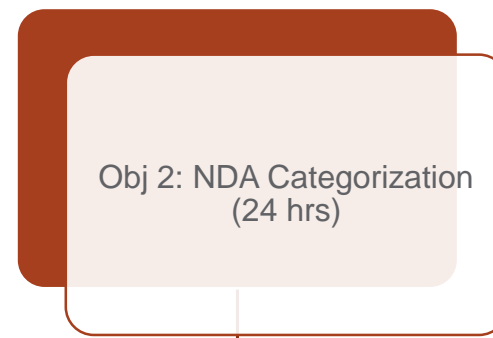
# Exercise Objectives





# Performance Indicators





## Purpose of the Data Review Meeting

- Opportunity to view your results relative to the community of results
- Please pay special attention to the results attributed to your lab:
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  - If either of these are not the case, please let me or Olivia know so we can correct this in the After Action Report





## Operation Celestial Skónis



Inject 1 – 24 Hour Preliminary Report



Case No. 52521, Sample ID: ES-1

- On August 3, 2018, workers at Greene's Metal Recycling call health department after a shipment of scrap metal alarm their radiation detectors
- Health department confirms alarm, isolates radioactive material (~15cm metal pipe) and notifies Central Police. Pipe and contents are taken into custody



Case No. 52521, Sample  
ID: ES-2

## Operation Celestial Skónis



Inject 1 – 24 Hour Preliminary  
Report



- On August 4, 2018, after a search by authorities of 6 metal foundries that contributed scrap to Greene’s Recycling, recovered 31 additional pipe sections that were radioactive.





## Authorities requested lab assistance to:

### Operation Celestial Skónis



- (1) Inventory evidence and conduct basic physical measurements
- (2) Identify any potential traditional forensic evidence that might need to be processed
- (3) Categorize radioactive items without (significantly) destroying any of the evidence
- (4) Develop an analytical plan for the purpose of determining if ES-1 and ES-2 are related in any way

Inject 1 – 24 Hour Preliminary Report





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**Thank you**



## Laboratory Presentations – 24 Hours

TIME	TOPIC	Responsible
11:45 – 12:05 pm	24-hour report results	Azerbaijan
12:05 – 12:25 pm	24-hour report results	Ukraine
12:25 – 1:25 pm	Lunch	
1:25 – 1:45 pm	24-hour report results	Germany
1:45 – 2:05 pm	24-hour report results	Russia
2:05 – 2:20 pm	Break	
2:20 – 2:40 pm	Novel Methodologies in 24 hours - sample receipt / managing contamination	Hungary
2:40 – 3:00 pm	Novel Methodologies in 24 hours - sample receipt / managing contamination	Romania





## Operation Celestial Skónis



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**Jon Schwantes & Olivia Marsden**  
Co-Chairs ETG, ITWG

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## Summary of 24 Hour Reporting



# 24 Hour Reporting

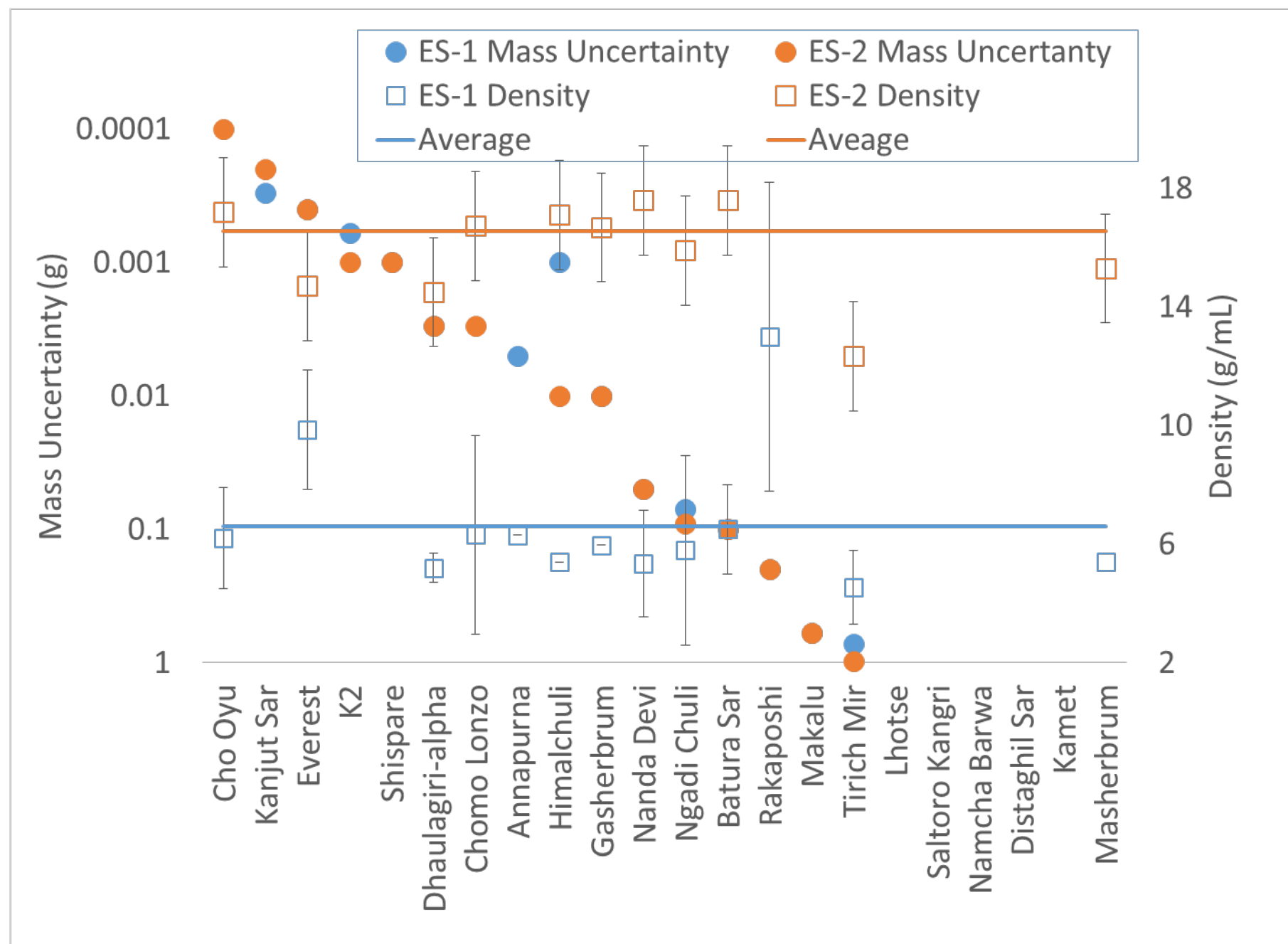
- Summary of 24 Hour Analyses
  - Basic Physical Measurements
    - ✓ Mass uncertainties
    - ✓ Density Estimates
  - Trace Elements
  - Categorization – Isotopic Analyses
    - ✓ U isotopes
    - ✓ Pu & Am isotopes
    - ✓ Other
- Graded Decision Framework
- Notable Efforts
- Discussion: Lessons Learned

## Purpose of the Data Review Meeting

- Opportunity to view your results relative to the community of results
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  - If either of these are not the case, please let me or Olivia know so we can correct this in the After Action Report

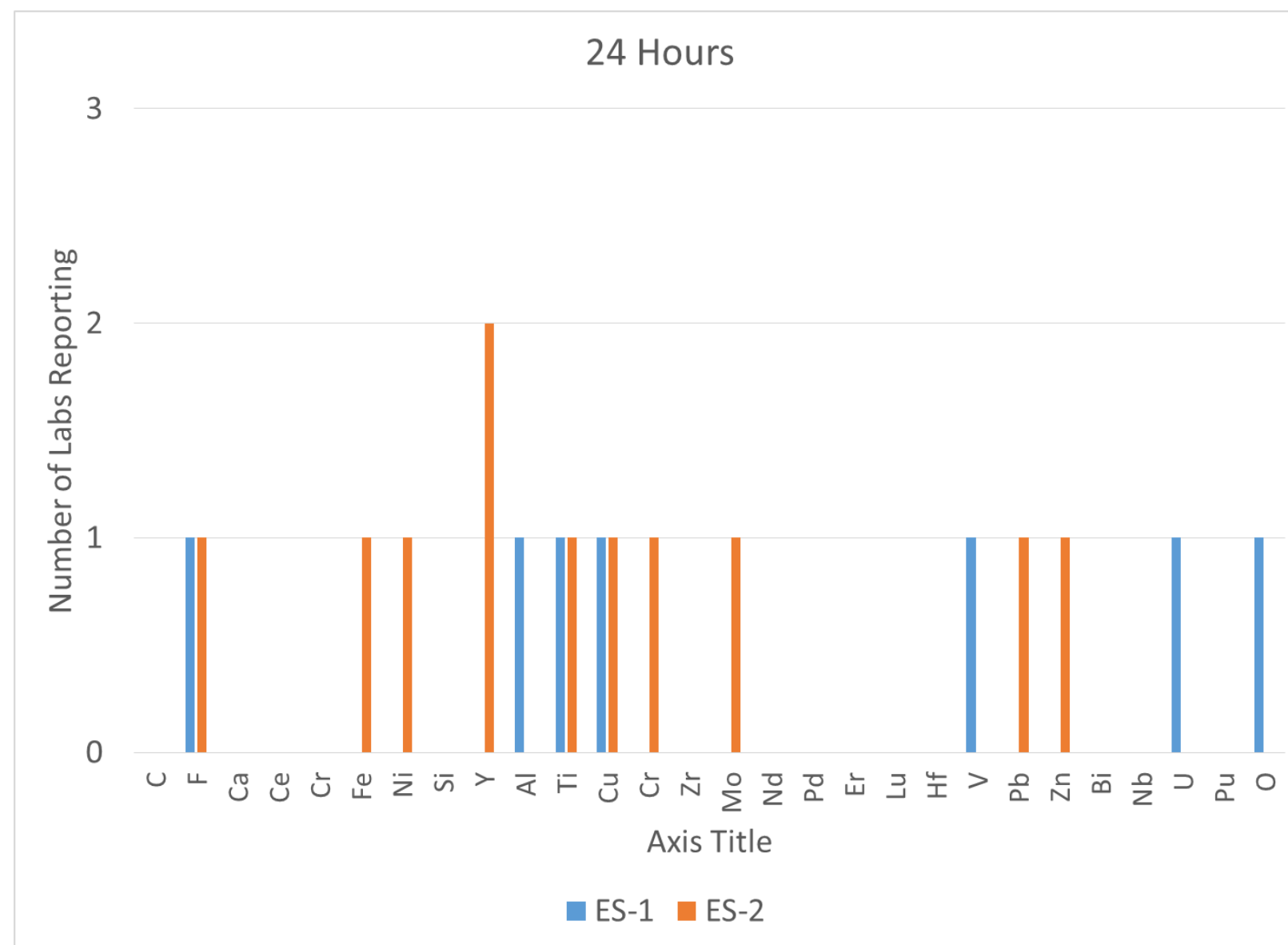
# Comparison of Basic Physical Measurements

- 4 orders of magnitude difference in mass uncertainties
- Uncertainties of density measurement not correlated to mass uncertainty
- Complicated geometry (estimate of volume) likely drives density uncertainty
- All but 2 labs reported results in 24 hrs



# Trace Elements

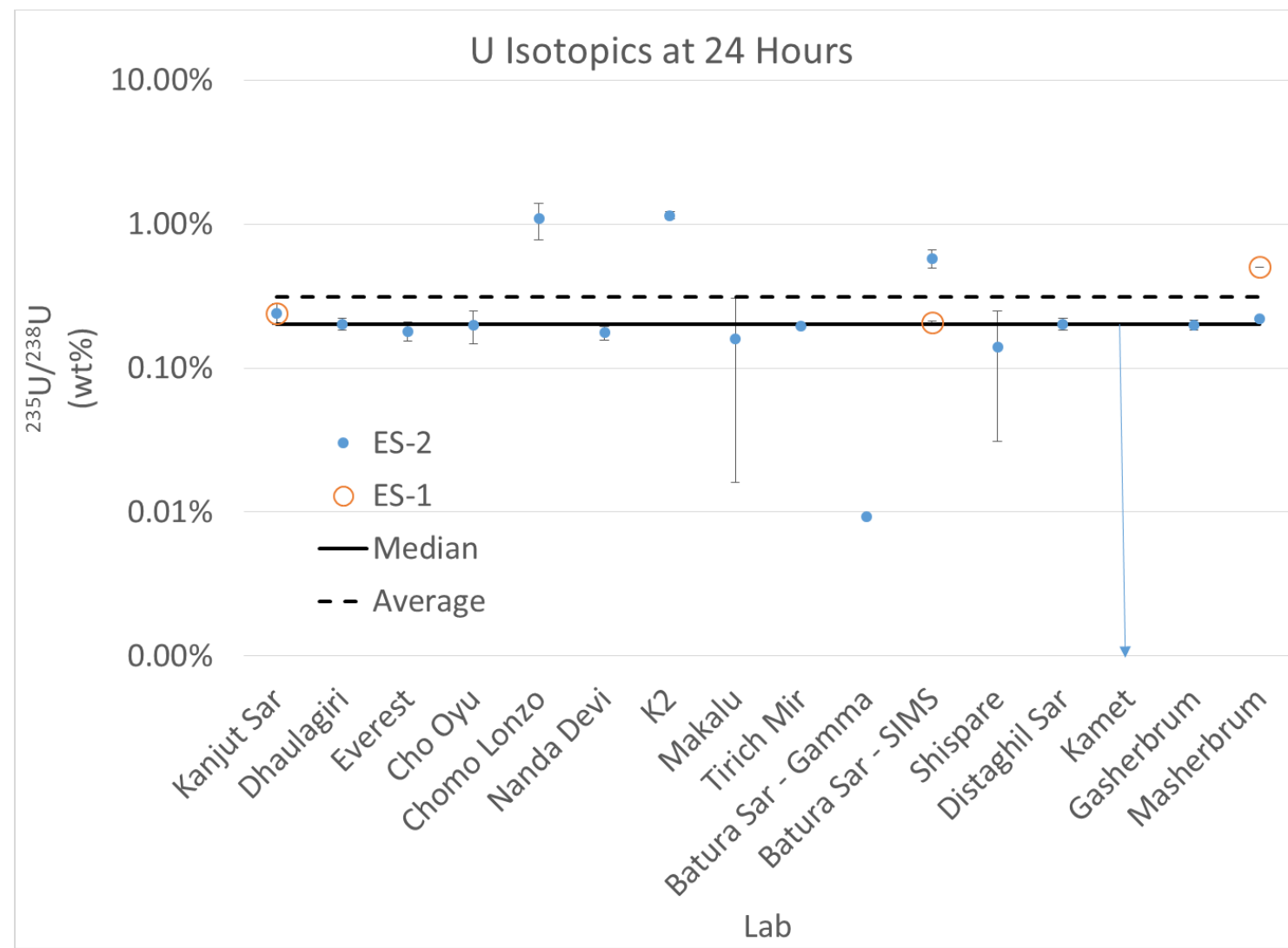
- 7 labs reported trace elements in the first 24 hrs
- 6 used XRF, 1 used SEM-EDX
- Pipe
  - Makalu, Nanda Devi, Tirich Mir, Kamet-Gamma
- Ingots
  - Himalchuli, Tirich Mir, Masherbru,
  - Batura Sar - SEM-EDX
  - Gasherbram – X-ray ID of Ce via HPGe





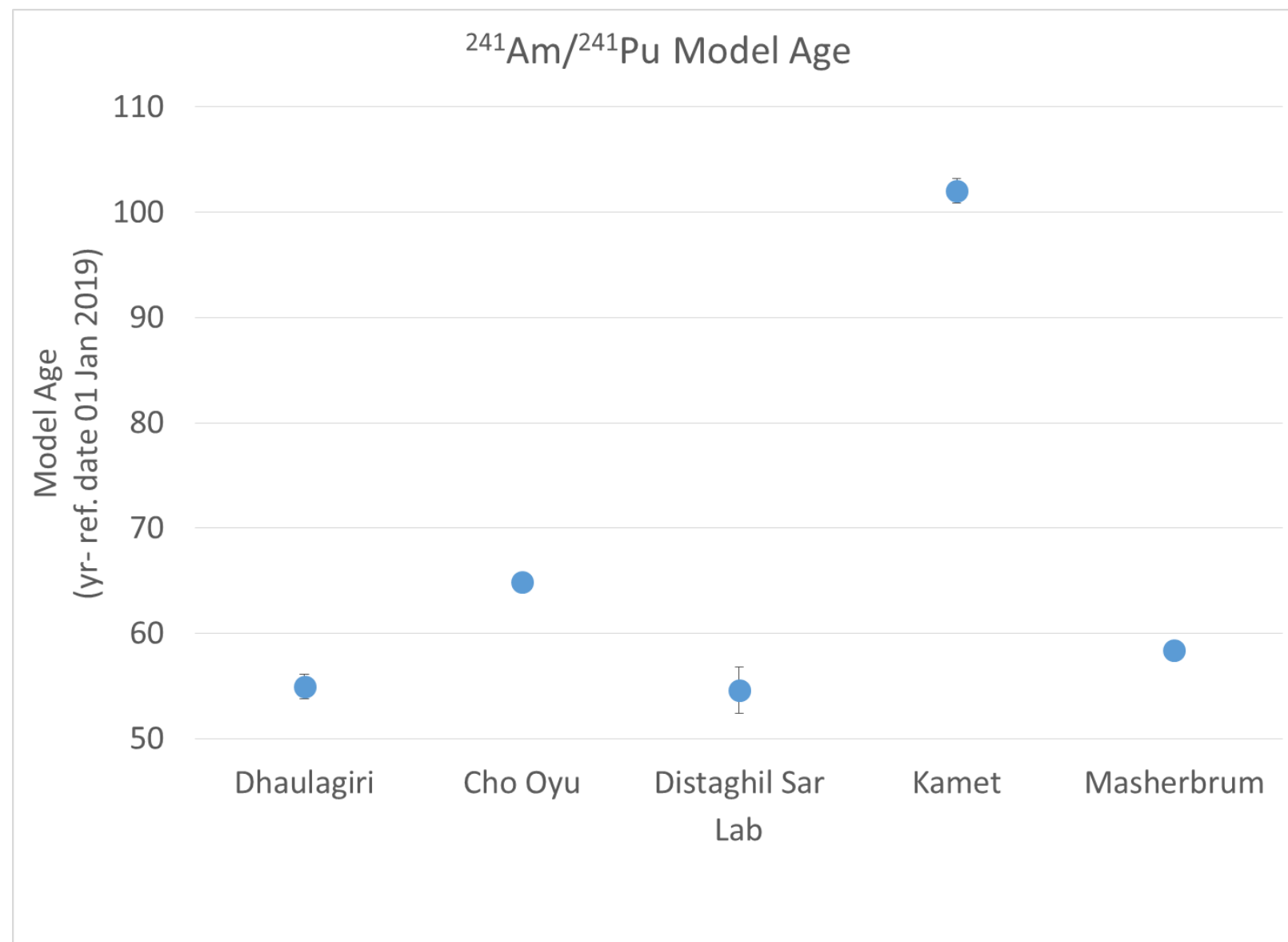
# Uranium Isotopics at 24 hrs

- 6 labs detected U in ES-1
- 3 labs reported  $^{235}\text{U}/^{238}\text{U}$  ratios in ES-1
- All but 1 lab detected U in ES-2
- 16 labs reporting  $^{235}\text{U}/^{238}\text{U}$  ratios
- 1 of three labs show  $^{235}\text{U}/^{238}\text{U}$  ratios in ES-1 and ES-2 consistent with each other
- HPGe, 1xSIMS



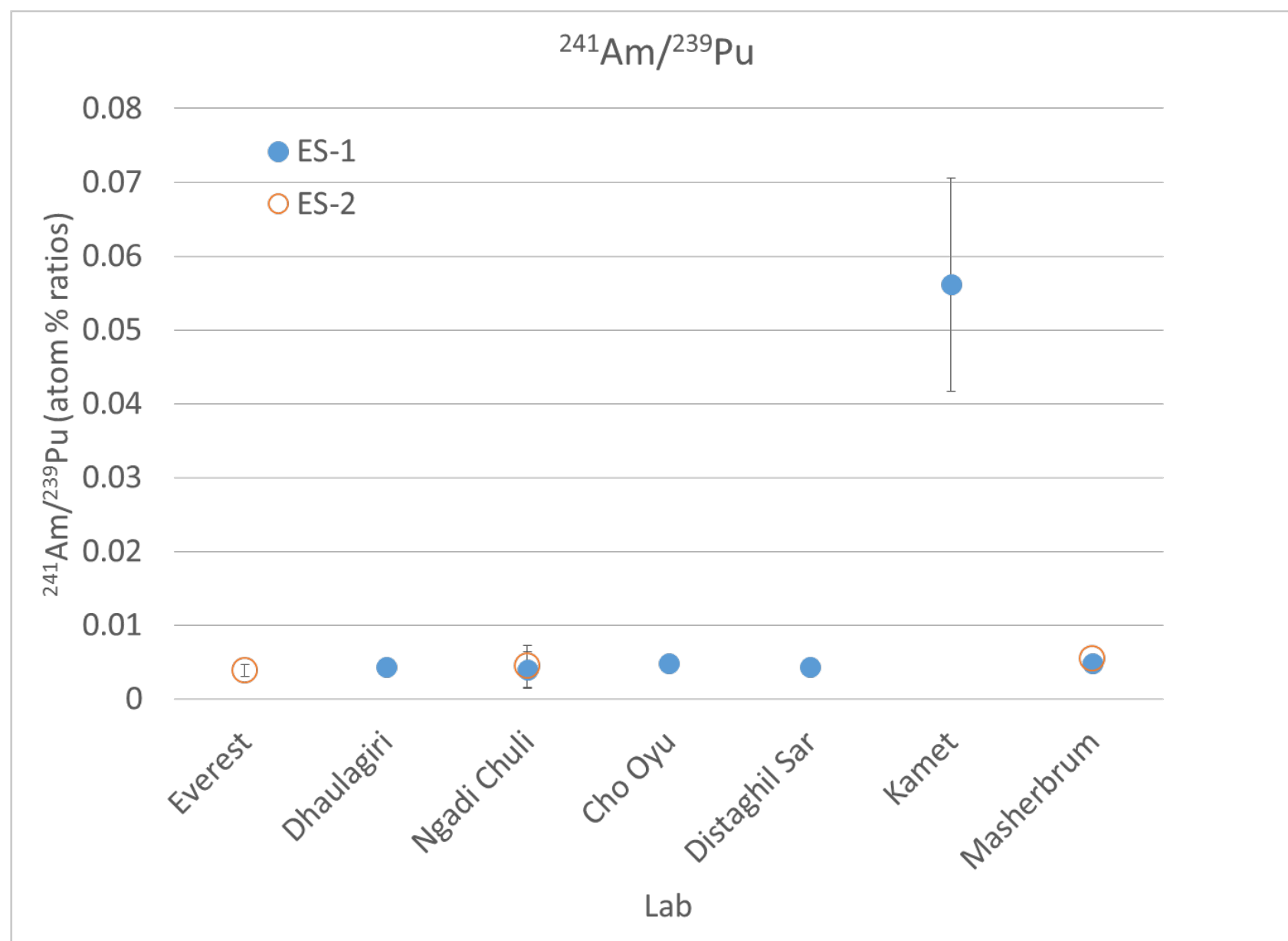
# Pu & Am Detection at 24 Hours

- 17 of the 20 labs received samples containing Pu
- Of those 17:
  - 13 detected Pu in ES-1
  - 7 detected Pu in ES-2
  - 13 detected  $^{241}\text{Am}$  in ES-1
  - 12 detected  $^{241}\text{Am}$  in ES-2
  - 5 labs reported model age
    - ✓ 2 of 5 were consistent with one another



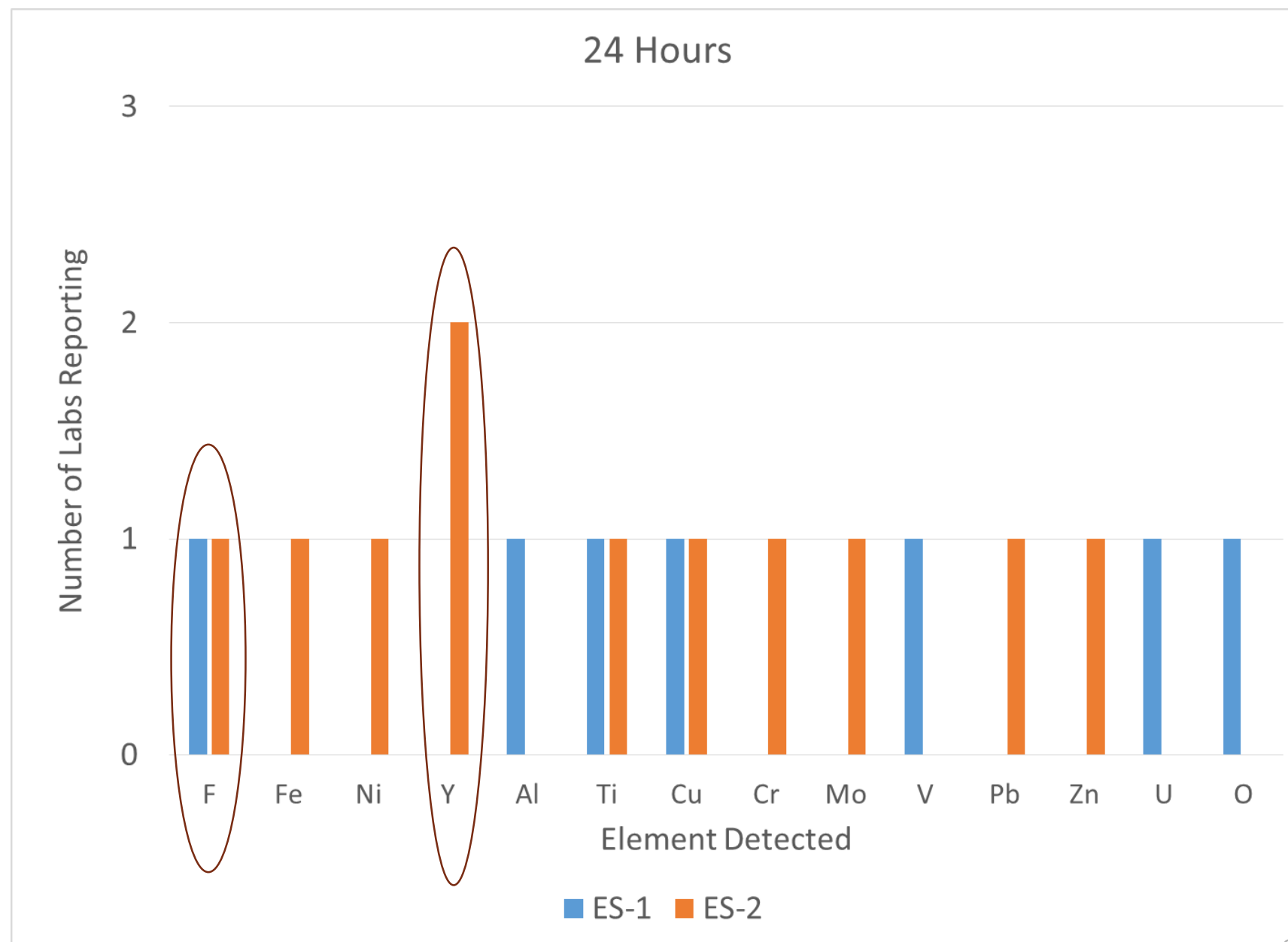
## Pu & Am Detection at 24 Hours

- 17 of the 20 labs received samples containing Pu
- Of those 17:
  - 13 detected Pu in ES-1
  - 7 detected Pu in ES-2
  - 13 detected  $^{241}\text{Am}$  in ES-1
  - 12 detected  $^{241}\text{Am}$  in ES-2
  - 6 labs reported  $^{239}\text{Pu}/^{241}\text{Am}$  ratios for ES-1
  - 3 labs reported  $^{239}\text{Pu}/^{241}\text{Am}$  ratios for ES-2
  - 2 labs reported ratios for both
    - ES-1 and ES-2 consistent



## Notable Efforts in 24 Hours

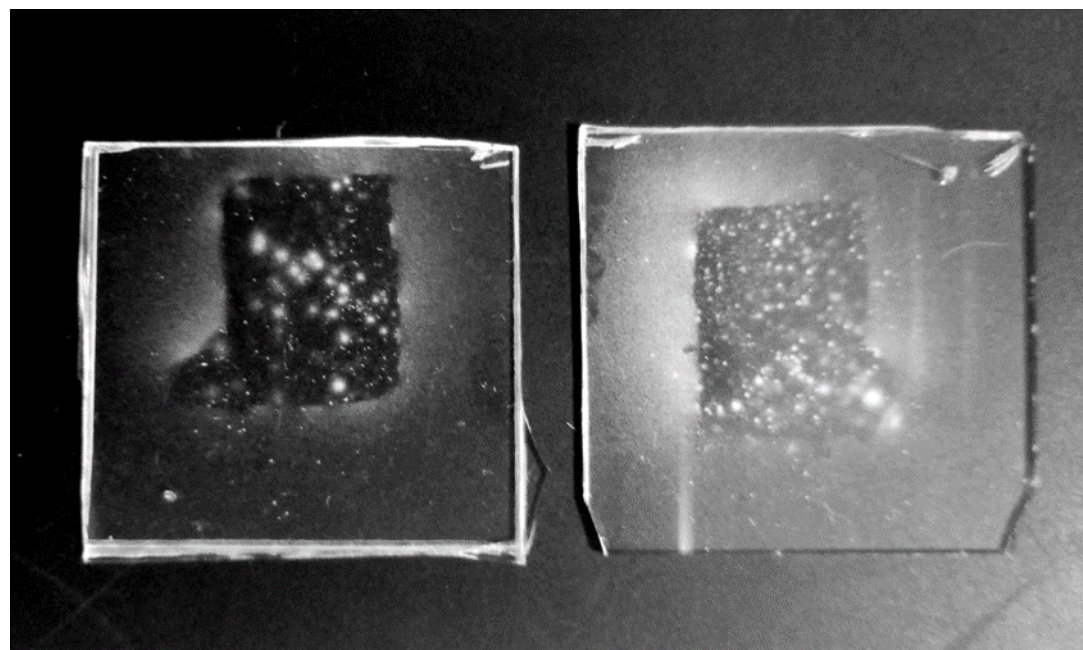
- Chomo Lonzo and others – contamination control
- Batura Sar, Masherbrum & Tirich Mir  
 - Detection of important trace contaminants (F, Y)
- Gasherbram – X-ray ID of Ce via HPGe



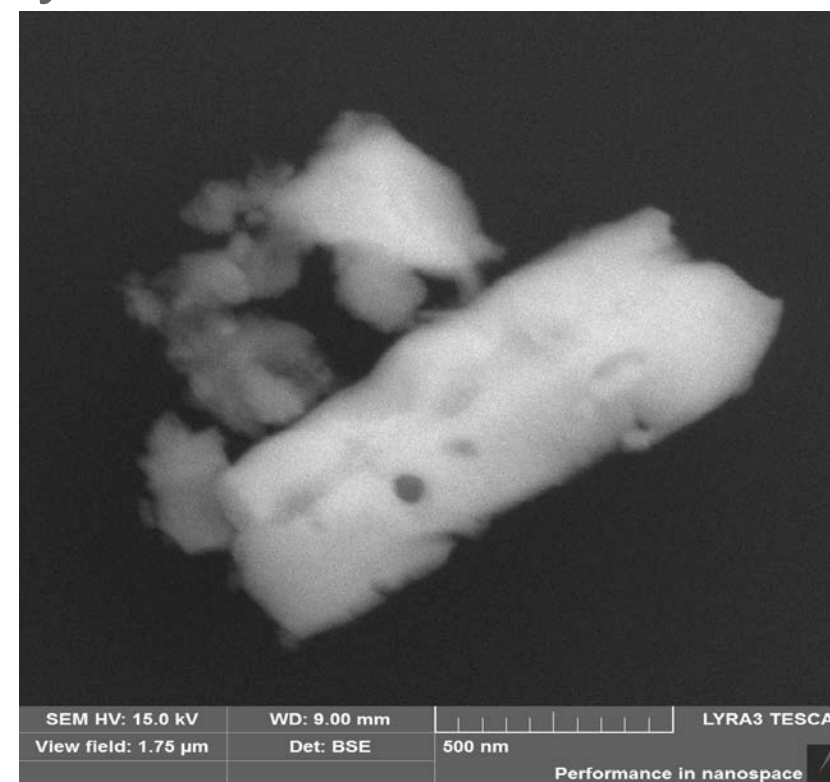


## Notable Efforts in 24 Hours

- Batura Sar – alpha track, SEM and SIMS analysis at 24 hrs!
  - Detection of U contamination on Ce



ES-1 alpha-tracks over sample ES-1 (Batura Sar).



ES-1 Pu-containing particle. Approximate elemental composition: Pu - 80.2%, O - 11.3%, F - 6.6%, Ce - 1.5%, Al - 0.4%.

## Notable Efforts in 24 Hours

- Rakaposhi – detection of  $^{22}\text{Na}$



- Alpha energy barrier for this reaction is  $\sim 5\text{MeV}$ ...suggesting the presence of an element in fluoride form (e.g., Pu, but not U) that emits an alpha particle with an energy  $>5\text{MeV}$
- Dhaulagiri, Cho Oyu, Kamet, Masherbrum - Pu age determination
- Everest, Ngadi Chuli, Masherbrum - Group Inclusion / Exclusion using  $^{241}\text{Am}/^{239}\text{Pu}$
- Cho Oyo, 24 hour analysis of Pu by alpha spectrometry
- Gasherbrum, segregation of traditional evidence from radioactivity for analysis outside of radiochemical laboratory



# Graded Decision Framework

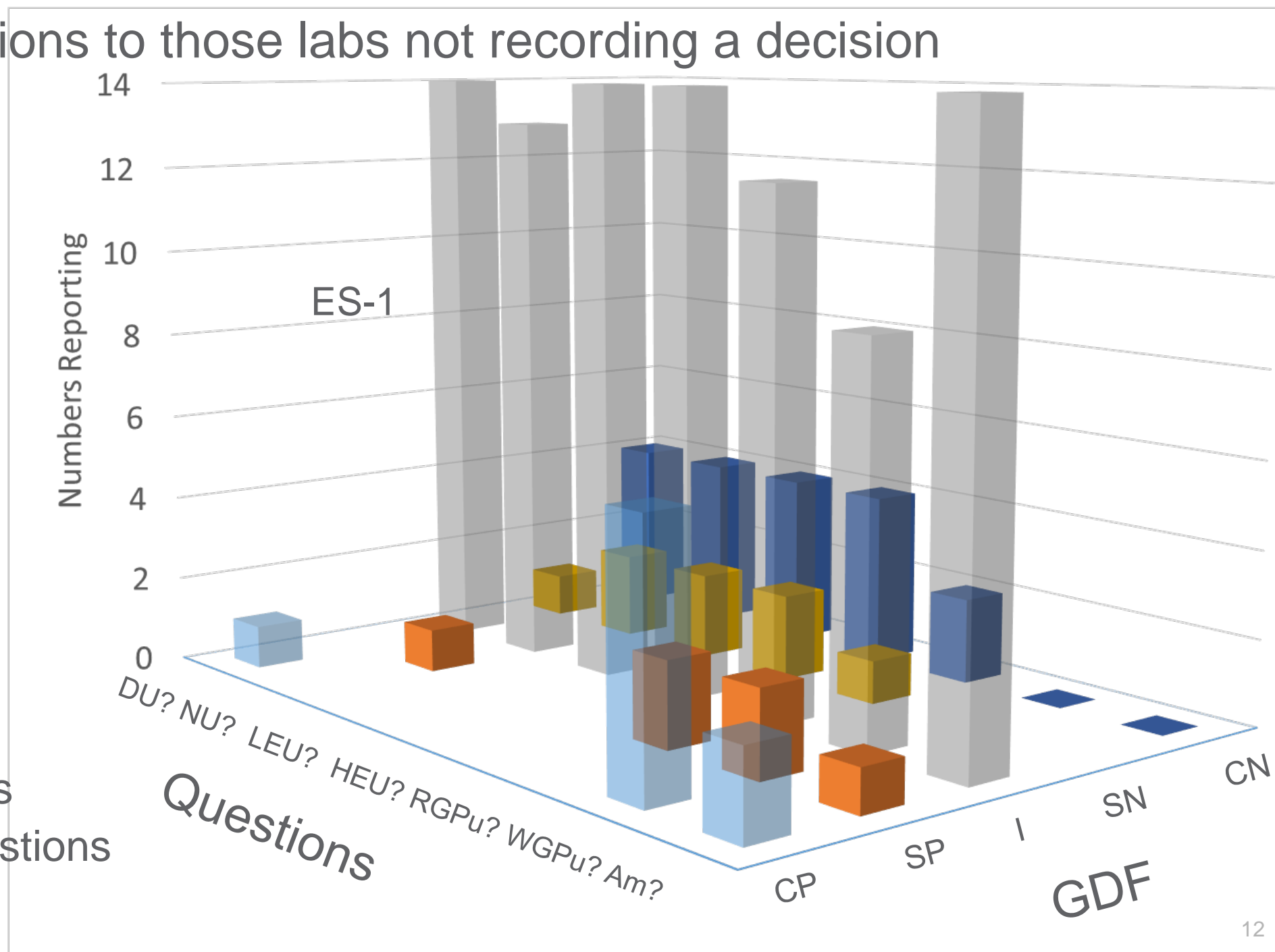
- Ratio of Conclusive Decisions to those labs not recording a decision for ES-1:

- DU? – 5/14
- NU? – 4/13
- LEU? – 4/14
- HEU? – 4/14
- RGPu? – 2/12
- WGPu? - 6/9
- Am? - 2/14

\*NR marked as I

\*20 labs total for U questions

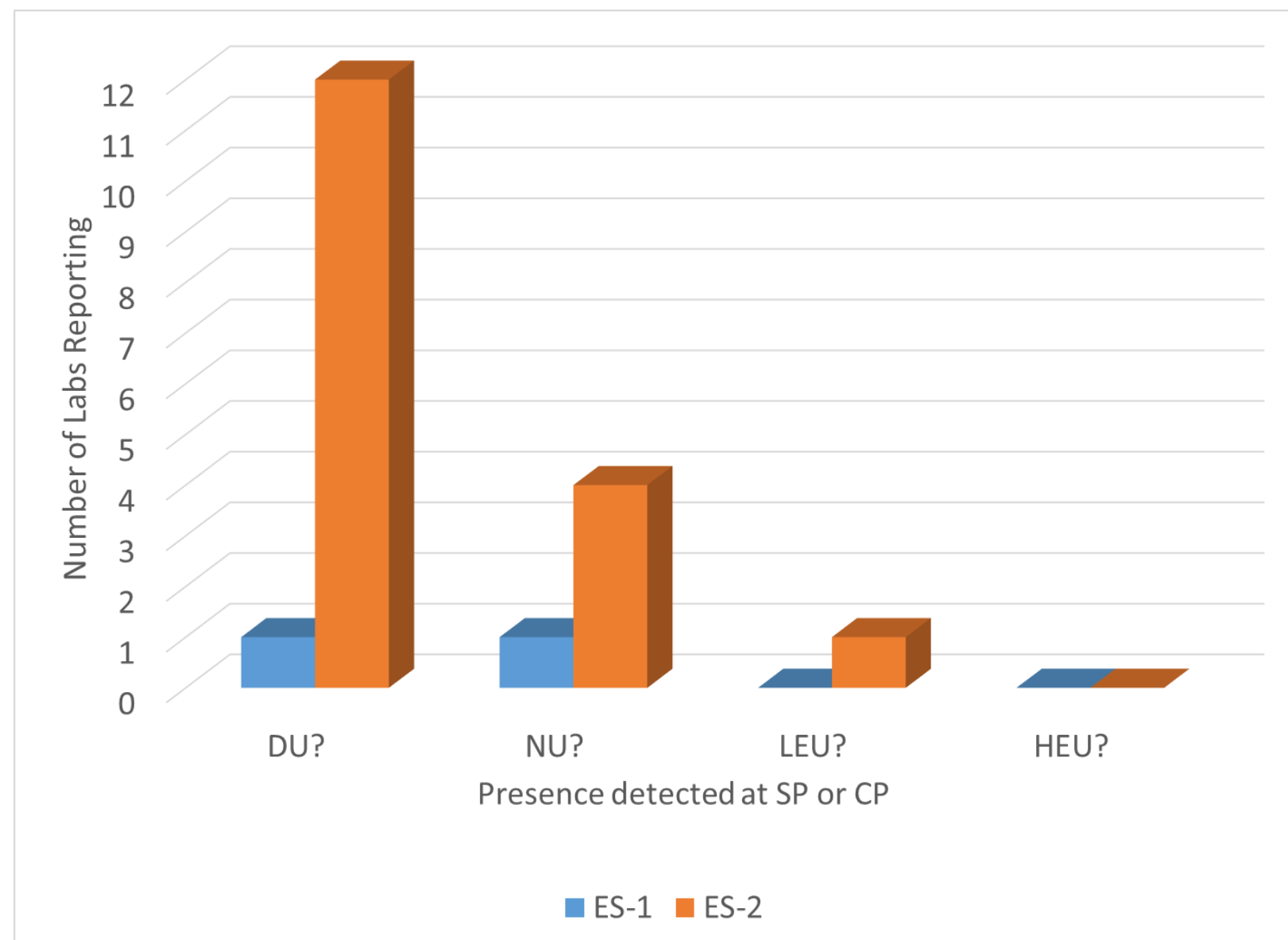
\*17 labs total for Pu/Am questions





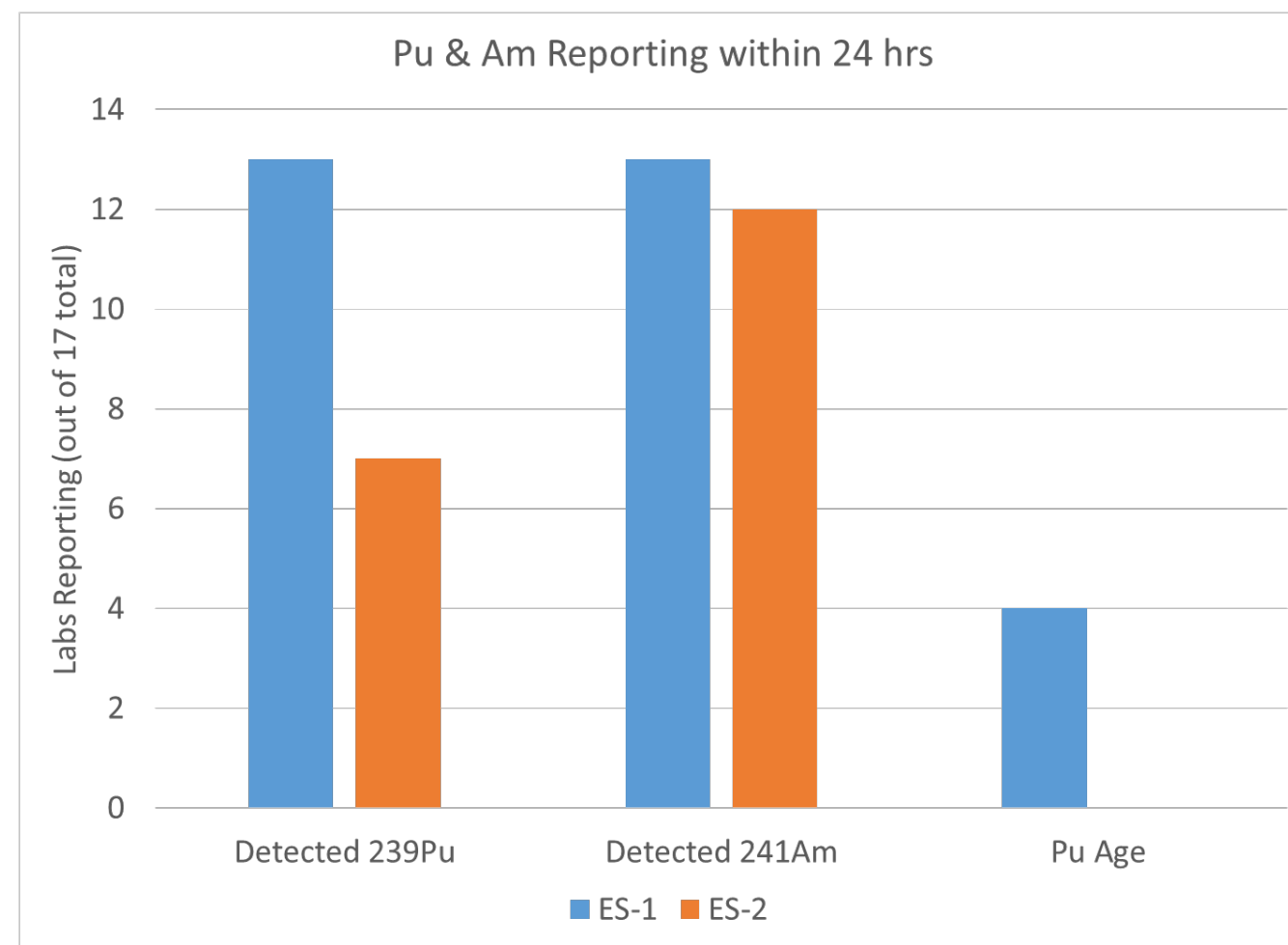
## U Detection at 24 Hours

- 2 labs out of 20 identified DU or NU in ES-1 with SP or CP confidence
- 16 out of 20 identified ES-2 as DU or NU with SP or CP confidence



## Pu & Am Detection at 24 Hours

- 17 of the 20 labs received samples containing Pu
- Of those 17:
  - 13 detected Pu in ES-1
  - 7 detected Pu in ES-2
  - 13 detected  $^{241}\text{Am}$  in ES-1
  - 12 detected  $^{241}\text{Am}$  in ES-2
  - 5 labs reported model age



## Discussion

- Exercise Objectives?
  - Primary Objectives – Novel Materials\*, Traditional Forensics, Blind Receipt
  - General Objectives – Physical characteristics, Phase ID, Trace Elements, Isotopics, Particles, Evaluations at 24 hours, 1 week, 2 months?
  - Inject location (with samples)?
- Sample shipping / receipt?
  - Issues with packaging?
  - Issues with “Blind” receipt?
- Exercise Timing?
  - Every 2 years or every 3 years?
  - Time of year still ok? (we currently target the fall as the start of the exercise)
  - Timing of this exercise? / DRM?
- Other comments related to logistics and the first 24 hrs of play?
- Exercise Scenario? – save for discussion after 2 month reporting
- \*Exercise Materials? – save for discussion after 2 month reporting

## Discussion Cont'd

- Exercise Play in the First 24-Hours?
  - Was inject 1 appropriate for this scenario?
    - ✓ Issues with waiting on DA until after the 24-hr report?
  - What methodologies were used?
  - Which ones were useful? Were there any that were not useful?
  - Were there any methodologies you wanted to use but didn't?
  - Was there anything you would do differently in the first 24 hours knowing what you know now?
- Any other comments / Questions related to the first 24 hours of exercise play?





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## Operation Celestial Skónis



## Inject 2 & Introduction to 1-Week Report

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**Jon Schwantes & Olivia Marsden**  
Co-Chairs ETG, ITWG



## Inject 2 & Introduction to 1-Week Report

- Exercise Objectives / Performance Metrics for 1 Week Report
- Inject 2
- 1 Week schedule of presentations



# Exercise Objectives

Obj 1: Novel  
Materials

Obj 2: NDA  
Categorization  
(24 hrs)

Obj 3: 1-Week  
Characterization

Obj 4: 2 Month  
Characterization

Obj 5: Evaluation





Obj 3: 1-Week Characterization

Cap 3.1: Isotopics

Cap 3.2: Elemental/Chemical

Cap 3.3 Physical

Cap 3.4: Age Date

Cap 3.5: Traditional Forensics

Act 3.5.1: Fingerprinting

Act 3.5.2: Toolmarks

Task 3.5.1.1: Patent Prints

Task 3.5.1.2: Latent Prints

PI 3.1.1: Detect Pu in ES1 in 24 hrs  
PI 3.1.2: Detect U in ES1 in 24 hrs  
PI 3.1.3: Detect U in ES2 in 24 hrs  
PI 3.1.4: Detect Pu in ES2 in 24 hrs

PI 3.2.1: ID Ce Metal in ES1 in 24 hrs  
PI 3.2.2: ID trace in ES1 in 24 hrs  
PI 3.2.3: ID U metal in ES2 in 24 hrs  
PI 3.2.4: ID trace in ES2 in 24 hrs

PI 3.3.1: Microscopy of pipes  
PI 3.3.2: Microscopy of ES's

PI 3.4.1: U age  
PI 3.4.2: Pu age

PI 3.5.1.1.1: Locate  
PI 3.5.1.1.2: Image

PI 3.5.1.2.1: Develop  
PI 3.5.1.2.2: Image

PI 3.5.2.1: Image and compare cut surfaces on pipes  
PI 3.5.2.2: Image and compare cut surfaces on ES's



## Operation Celestial Skónis



Inject 2 – 1-Week Preliminary Report

Lead investigator has received your 24 hour report and approved your Analytical Plan

- 1) Proceed with your proposed analyses of ES-1 & ES-2
- 2) Using new or old version of the GDF, determine if the metal pipes associated with ES-1 and ES-2 consistent with one another?
- 3) Determine chemical composition / phase of ES-1
- 4) Determine chemical composition / phase of ES-2
- 5) Using new or old version of the GDF, determine if the radioactive ingots associated with ES-1 and ES-2 are consistent with one another?
- 6) Evaluate any other traditional or nuclear forensic evidence that may link ES-1 and ES-2 using either version of the GDF.

Report all results to the LI within 6 days



**Thank you**



# Laboratory Presentations – 1 Week

<b>9:45 – 10:05 am</b>	1-Week report results	Poland
<b>10:05 – 10:25 am</b>	1-Week report results	Sweden
<b>10:25 – 10:45 am</b>	Break	
<b>10:45 – 11:05 am</b>	1-Week report results (U Only)	Japan
<b>11:05 – 11:25 am</b>	1-Week report results	Singapore
<b>11:25 – 11:45 pm</b>	1-Week report results (U Only)	Israel
<b>11:45 – 12:05 pm</b>	1-Week report results (with focus on comparing GDF-A and GDF-B)	Korea
<b>12:05 – 1:05 pm</b>	Lunch	
<b>1:05 – 1:25 pm</b>	Novel Methodologies 1-Week – traditional forensics	Australia
<b>1:25 – 1:45 pm</b>	Novel Methodologies 1-Week – traditional forensics	JRC





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## Operation Celestial Skónis



## Summary of 1-Week Reporting

6<sup>th</sup> Collaborative Materials  
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Co-Chairs ETG, ITWG



# 1-Week Reporting

- Summary of 1-Week Analyses
  - Traditional Evidence
  - Physical Characterization
  - Phase ID
  - Chemical / Elemental Characterization
    - ✓ Bulk
    - ✓ Surface
  - Isotopic Characterization
    - ✓ Bulk
    - ✓ Surface
- Evaluation
  - Age Dating
  - Pedigree
  - Graded Decision Framework
- Notable Efforts
- Discussion: Lessons Learned



# Purpose of the Data Review Meeting

- Opportunity to view your results relative to the community of results
- Please pay special attention to the results attributed to your lab:
  - Did we capture all of the important data you generated?
  - Is it accurate?
  - If either of these are not the case, please let me or Olivia know so we can correct this in the After Action Report

## Traditional Evidence

- K2 performed fiber analysis of evidence recovered from ES-1
- 5 labs developed fingerprints in 1-week
- 6 labs compared tool marks on the pipes
- Cho and Masherbrum Oyo compared tool marks on ingots
- Rakaposhi compared tool marks on plastic bags



Image of fiber found on ES-1 processed by K2



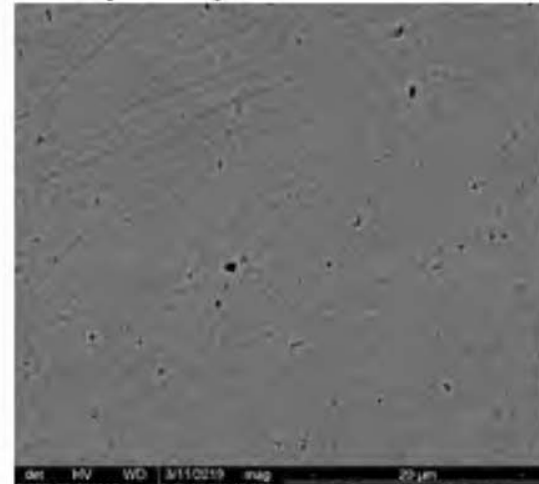
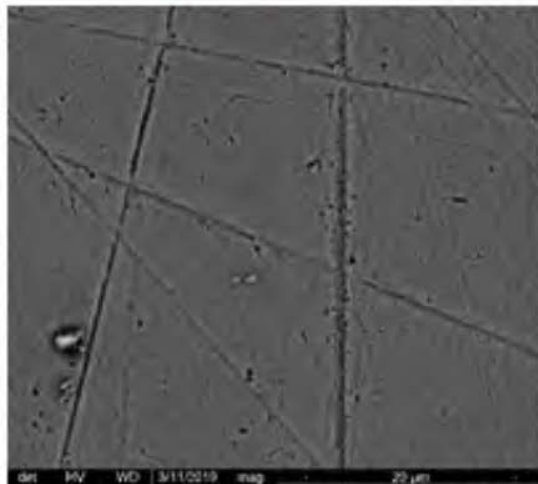
# Physical Measurements

- Optical & Electron Microscopies utilized extensively.
  - 6 labs reported OM results in 1 Week report
  - 9 labs reported SEM results in 1 week

ES-1 pipe

ES-2 pipe

vertical surface (BSE)



SEM images of ES-1 and ES-2 showing remnants of polishing (Masherbrum).

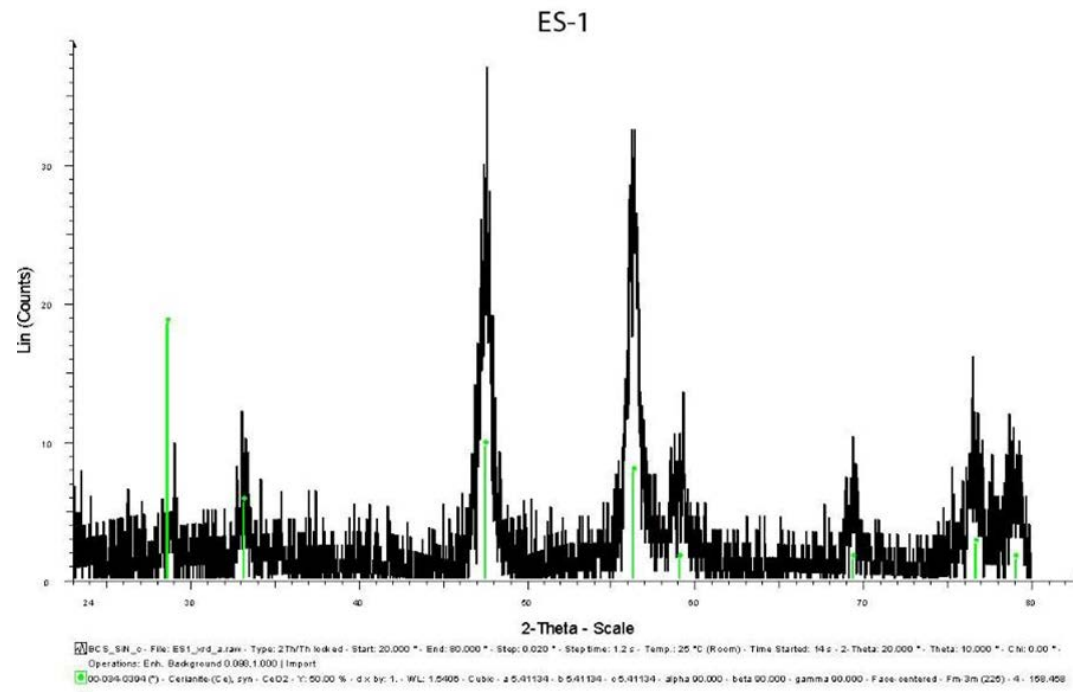


Digital optical microscope image of ES-1 (Cho Oyu).

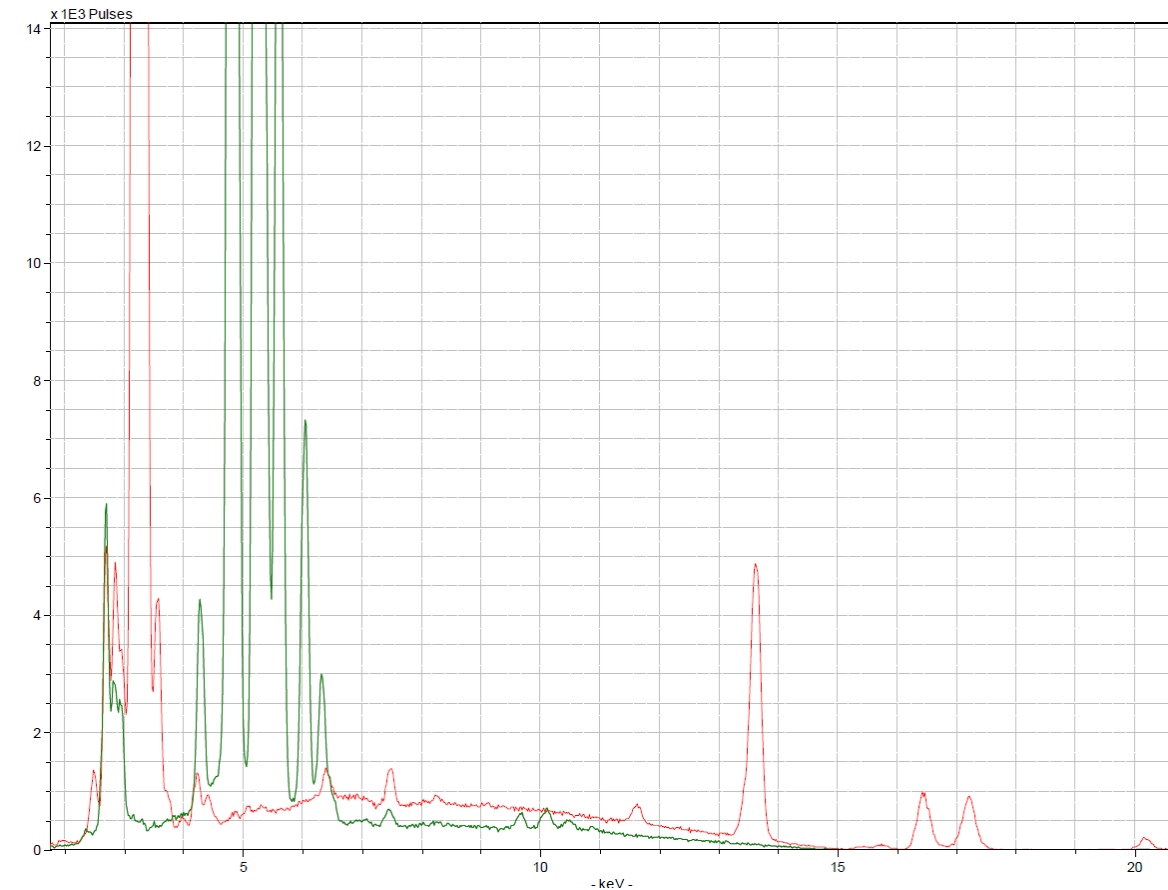


## Phase ID of Ingots

- Chomo Lonzo, Batura Sar, Rakaposhi and Annapurna evaluated phase of ingots with pXRD
- 7 labs used alternative means (Gamma spec, XRF, IR, SEM-EDX) to identify phase of ingots



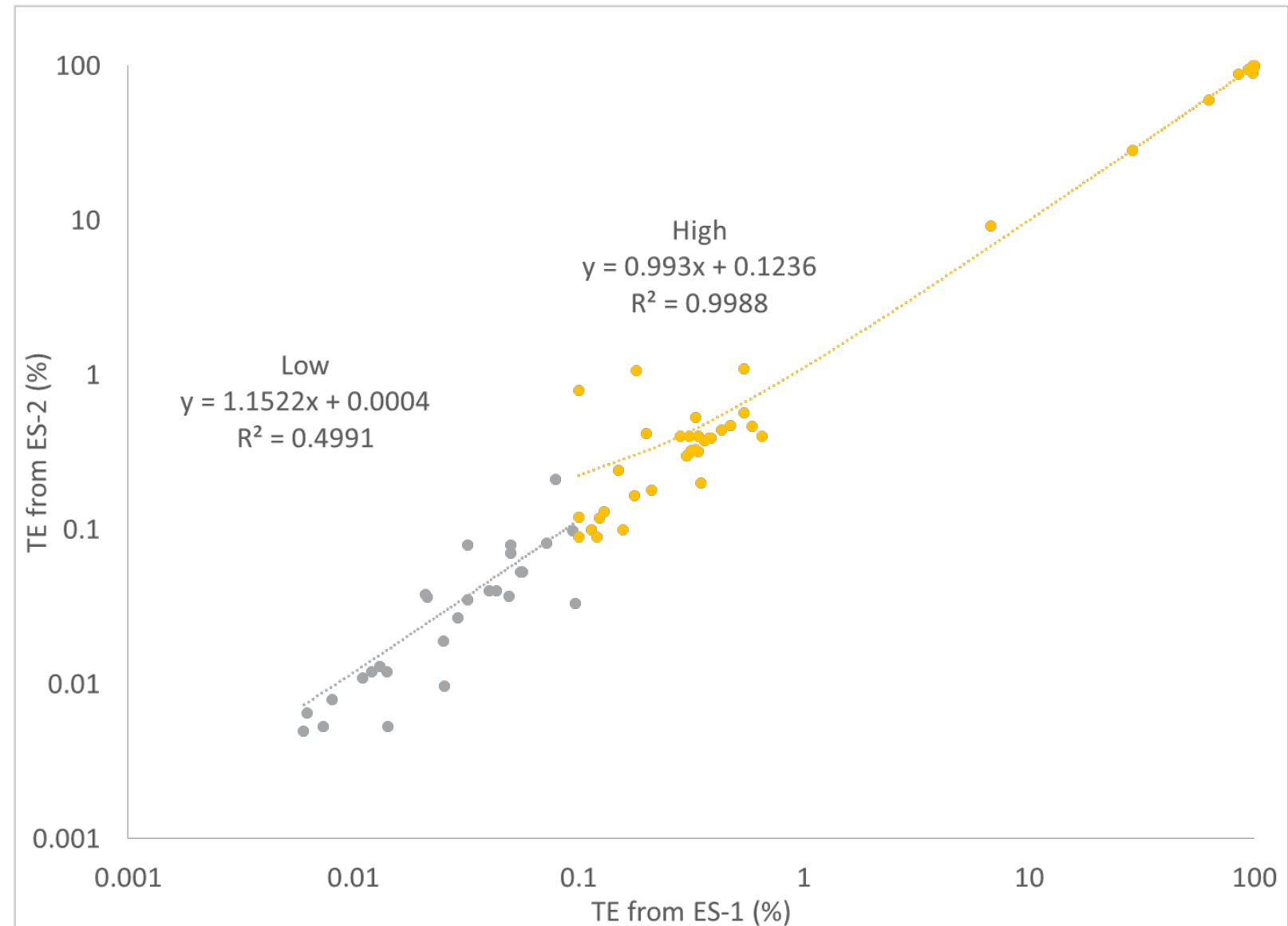
XRD pattern of ES-1 reported by Chomo Lonzo



Comparison of XRF spectra from ES-1 and ES-2 by Kanjut Sar

## Trace Elements - bulk

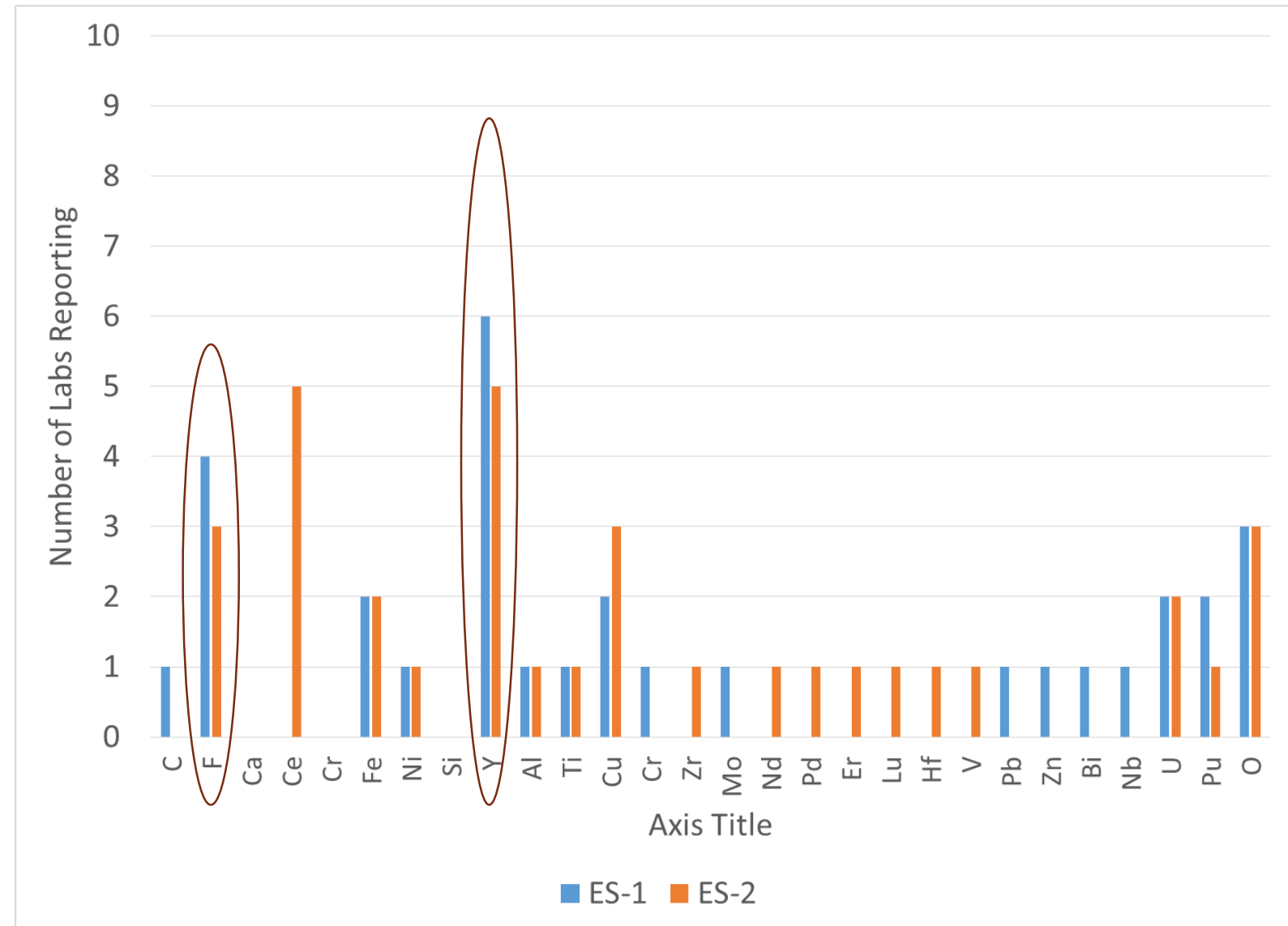
- Good correlation between trace elements found in pipes
- Techniques applied: qICP-MS, SF-ICP-MS, ICP-OES
- Labs reporting values in 1 week: Nanda Devi, Batura Sar, K2, Kamet, Shispare, and Namcha Barwa





# Trace Elements

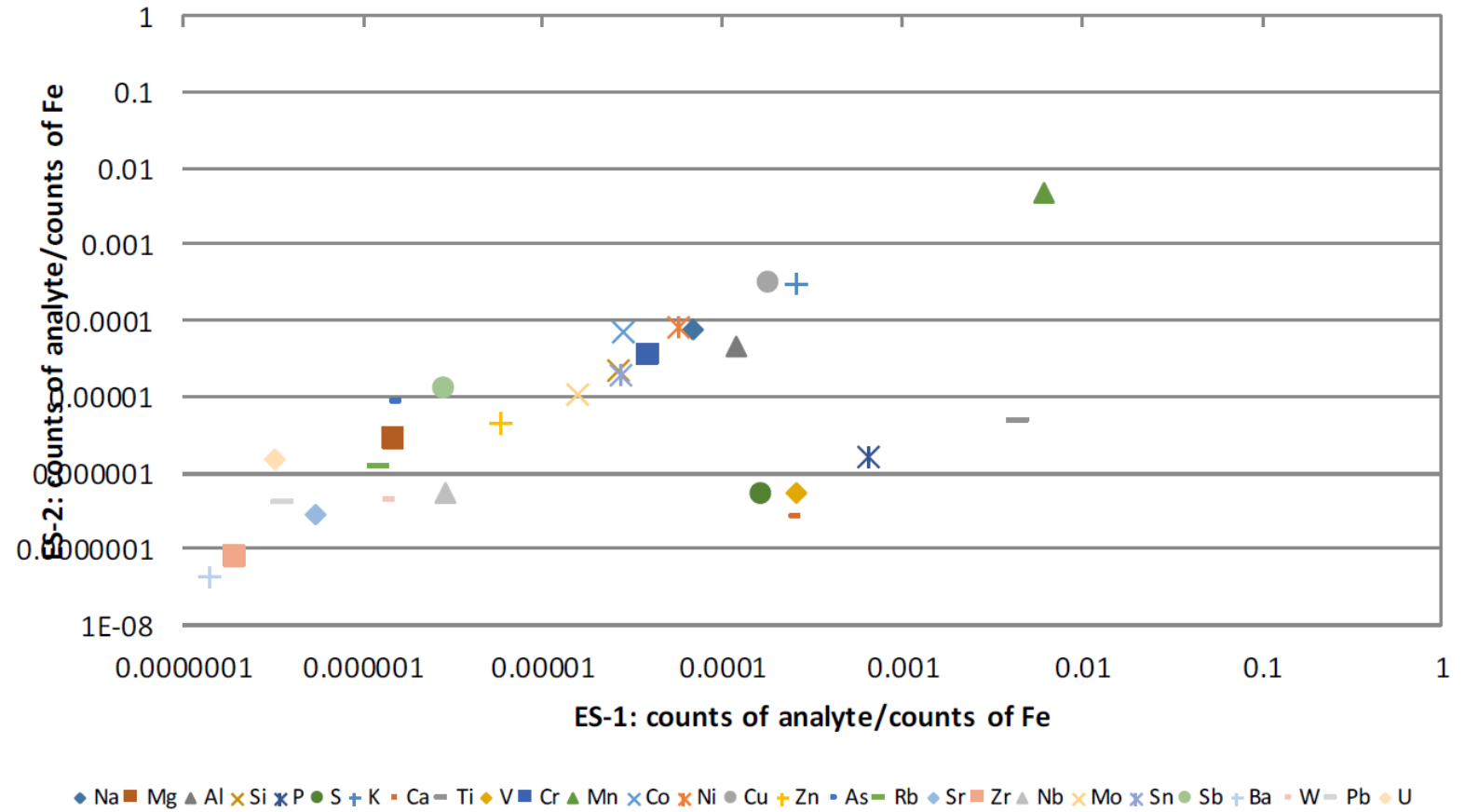
- 13 labs reported trace elements within the first week
  - 3 surface techniques: 6xXRF, 4xSEM, and 1xLA-QQQ-ICP-MS
  - More labs identify presence of significant contaminants



# Trace Elements

- Everest applied LA-QQQ-ICP-MS
  - Quantitative results of surface elemental analysis compared well with bulk analysis

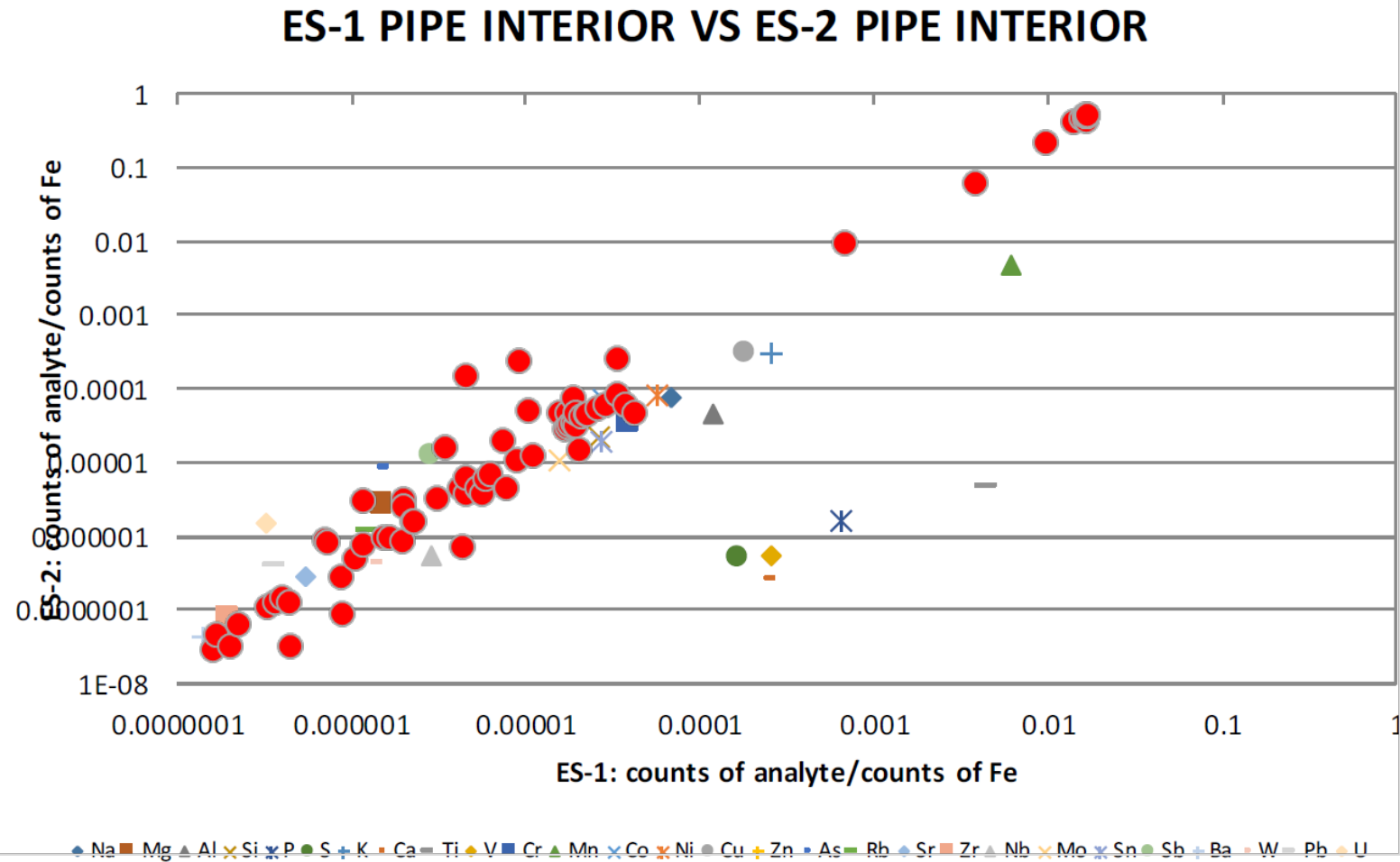
**ES-1 PIPE INTERIOR VS ES-2 PIPE INTERIOR**





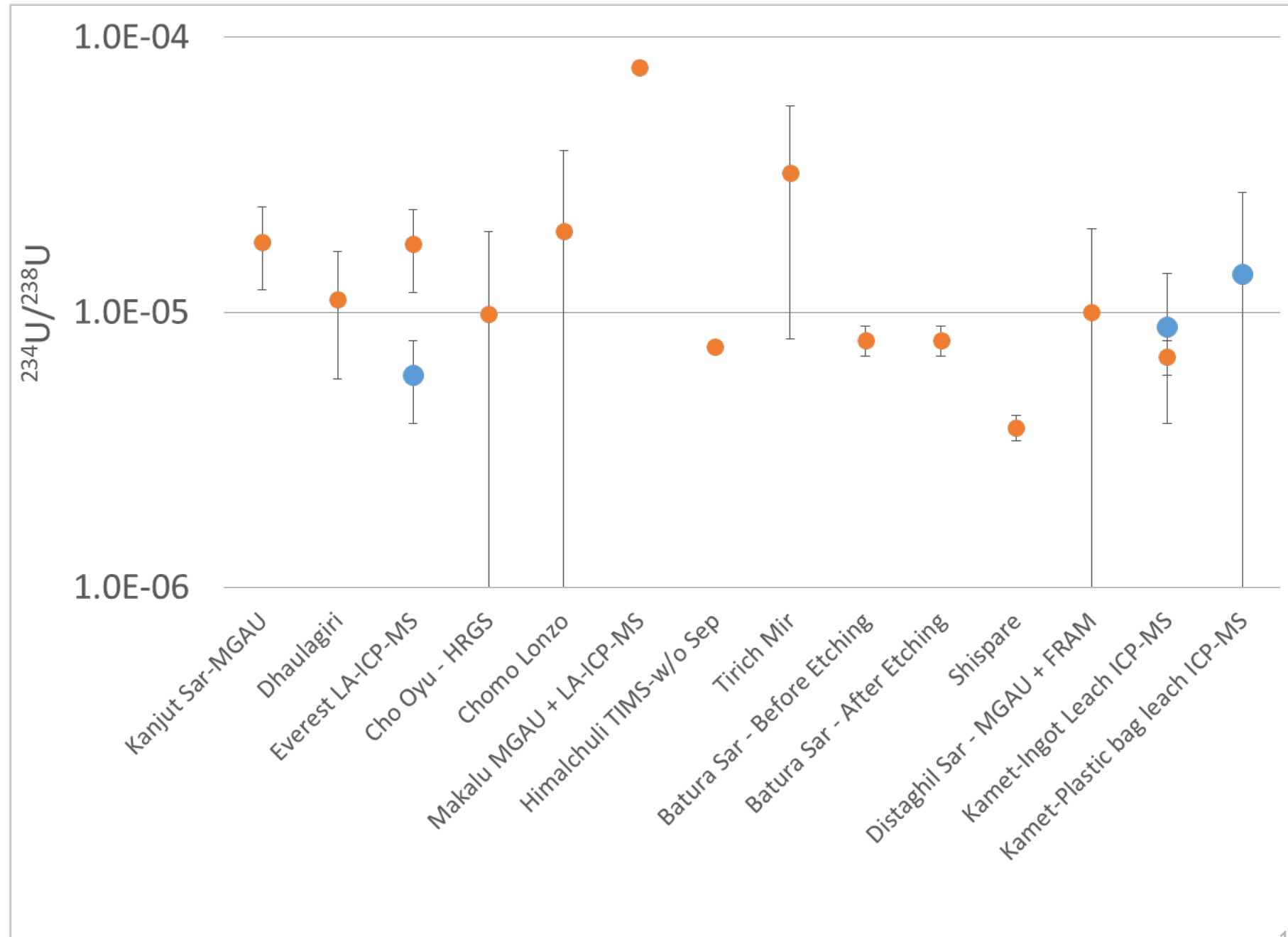
# Trace Elements

- Everest applied LA-QQQ-ICP-MS
  - Quantitative results of surface elemental analysis compared well with bulk analysis



# Uranium Isotopics in 1 Week

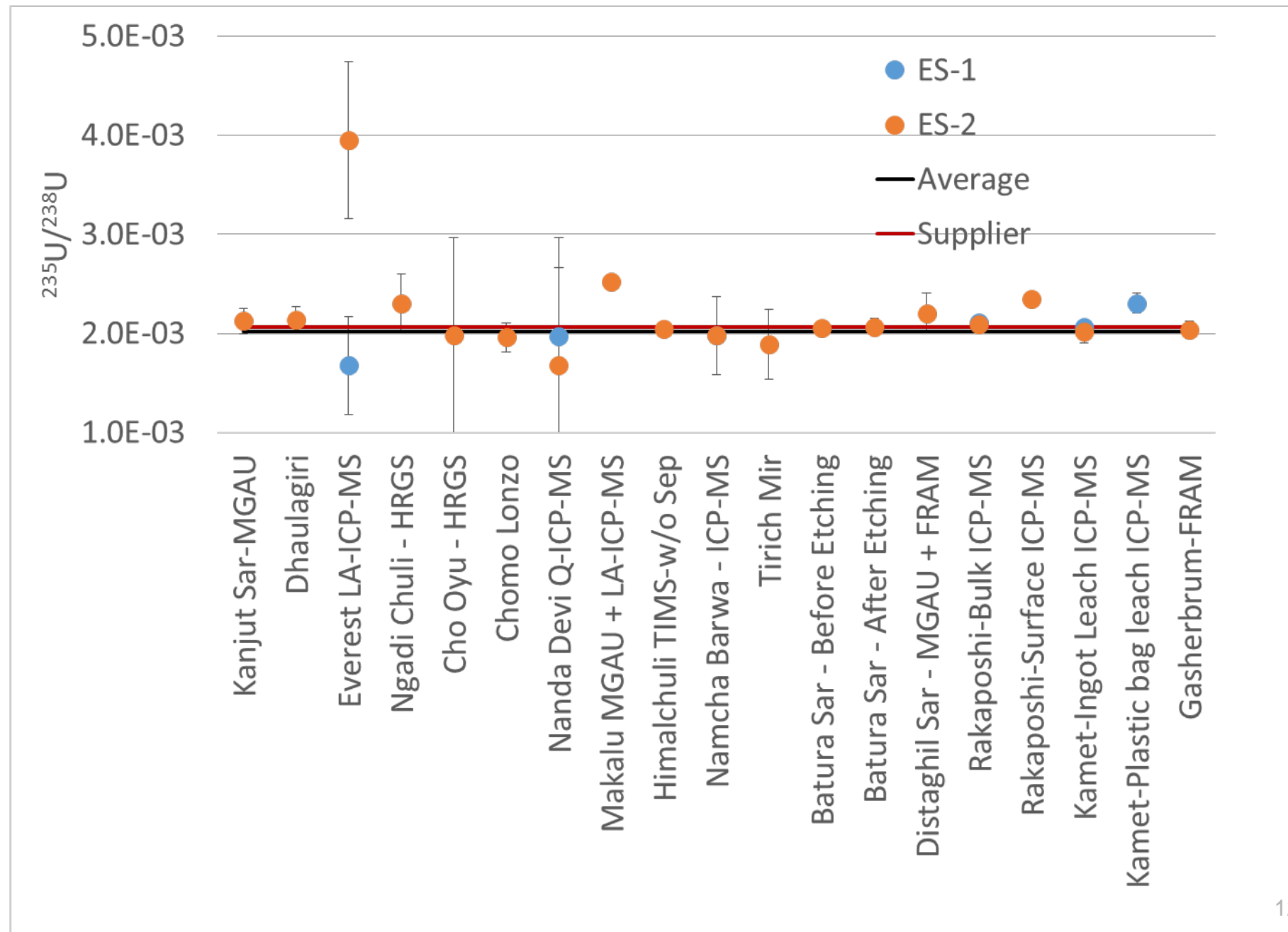
- 14 labs reporting  $^{234}\text{U}/^{238}\text{U}$  ratios for ES-2
- Everest and Kamet reported  $^{234}\text{U}/^{238}\text{U}$  ratios for ES-1
- Kamet shows consistency between ES-1 and ES-2
- Everest shows minor inconsistency between ES-1 and ES-2 using surface technique





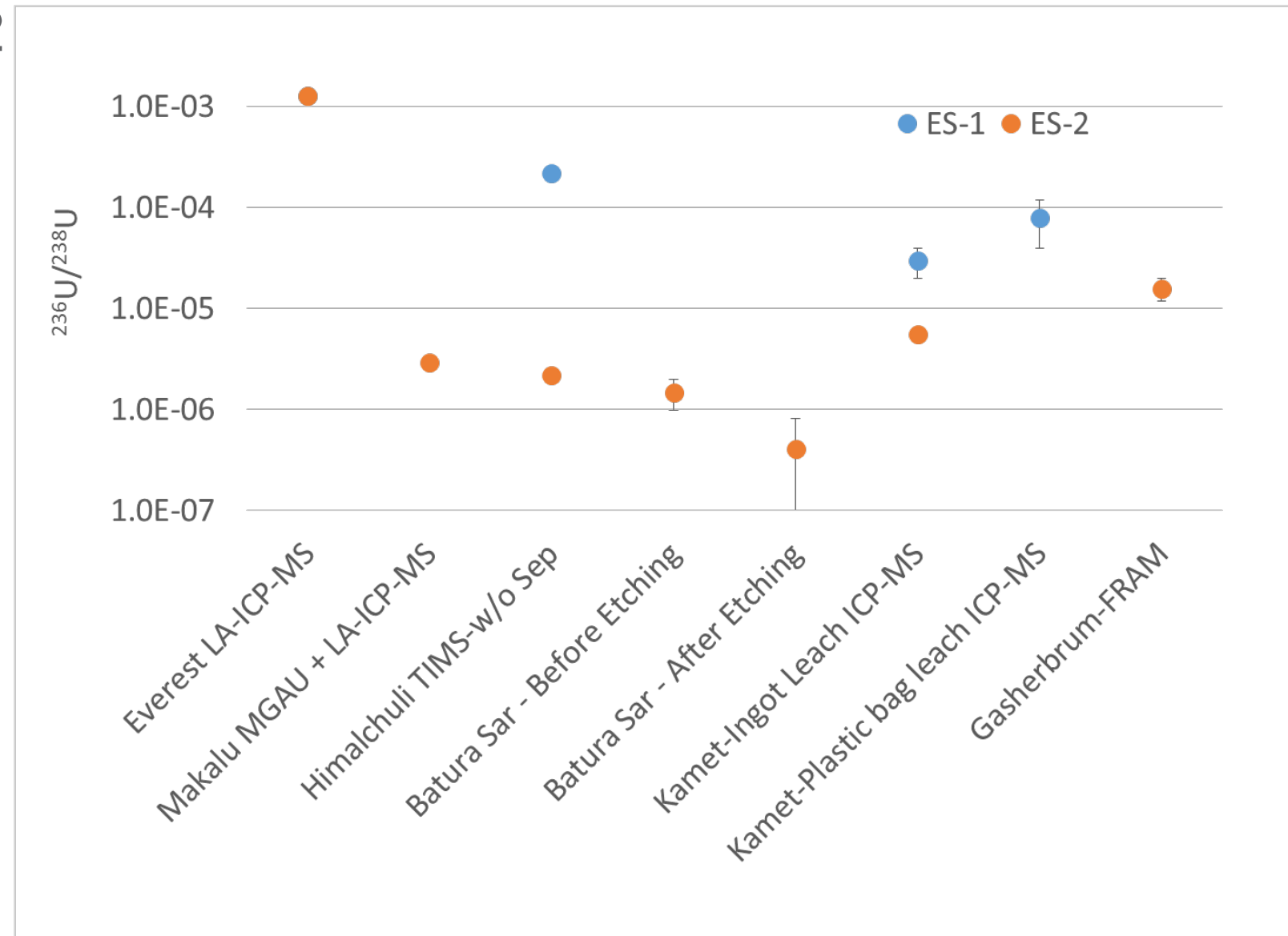
# Uranium Isotopics in 1 Week

- 17 labs reporting  $^{235}\text{U}/^{238}\text{U}$  ratios for ES-2
- Everest, Nanda Devi, Kamet, and Annapurna reported  $^{235}\text{U}/^{238}\text{U}$  ratios in ES-1
- Most labs reporting values for both ES-1 and ES-2 show consistency between samples
- Most lab results consistent with supplier declaration



## Uranium Isotopics in 1 Week

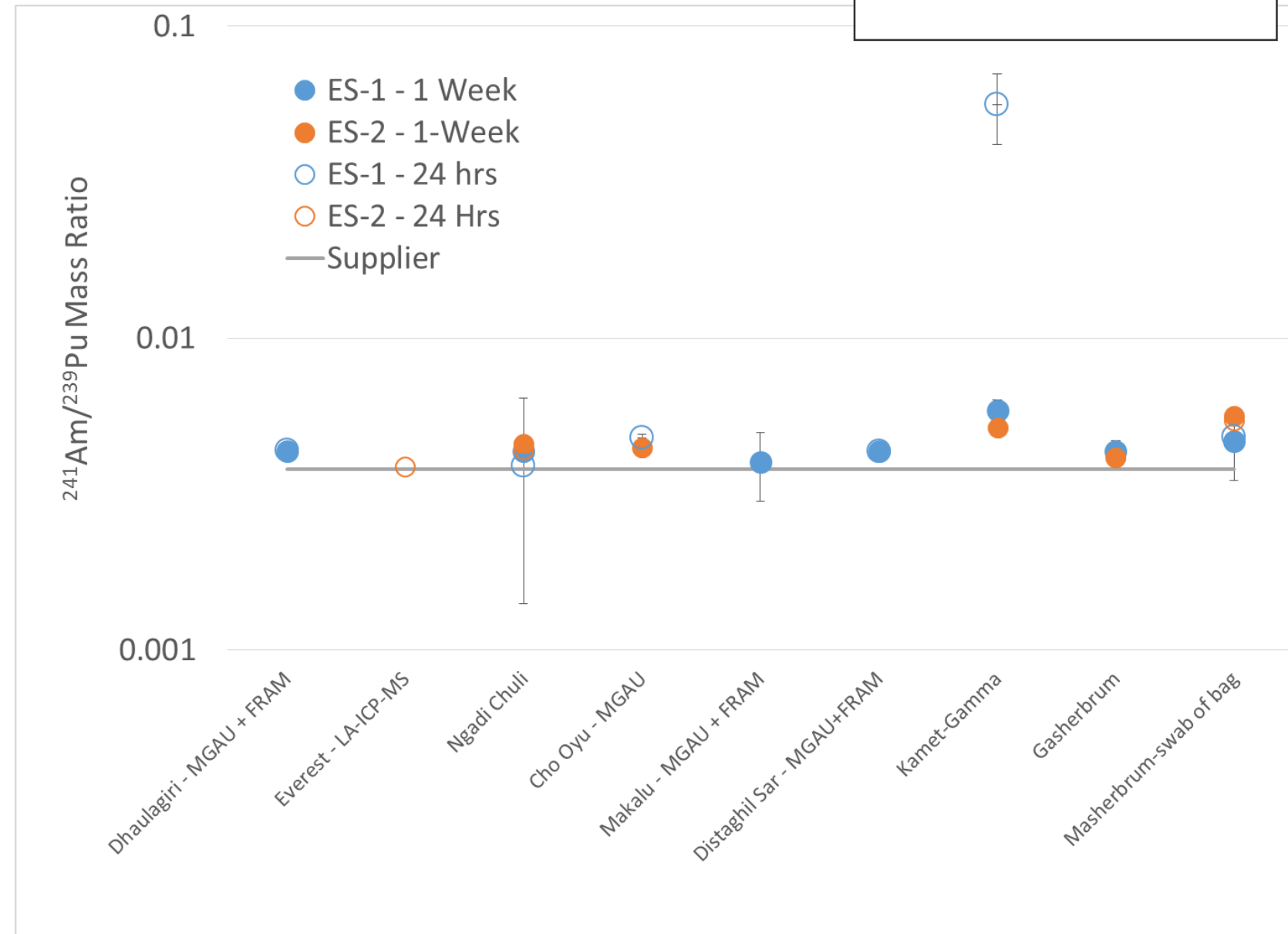
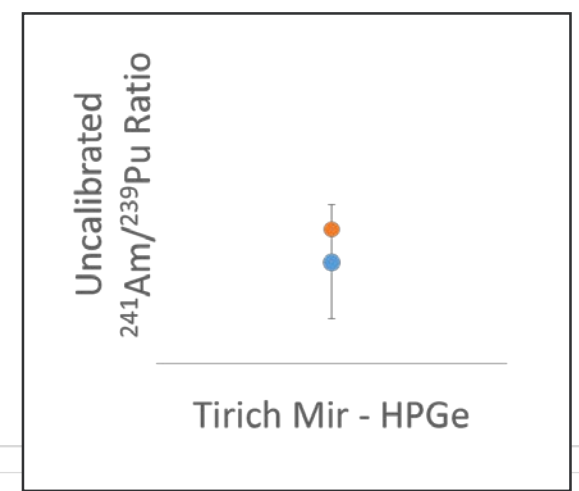
- Himalchuli and Kamet reported  $^{236}\text{U}/^{238}\text{U}$  ratios for ES-1 and ES-2
- Annapurna reported  $^{236}\text{U}/^{238}\text{U}$  ratios for ES-1
- Everest, Makalu, Batura Sar and Shispare reported  $^{236}\text{U}/^{238}\text{U}$  ratios for ES-2
- Large spread in data between labs
- No consistency observed between ES-1 and ES-2
- Excess U from Pu contamination may be to blame??





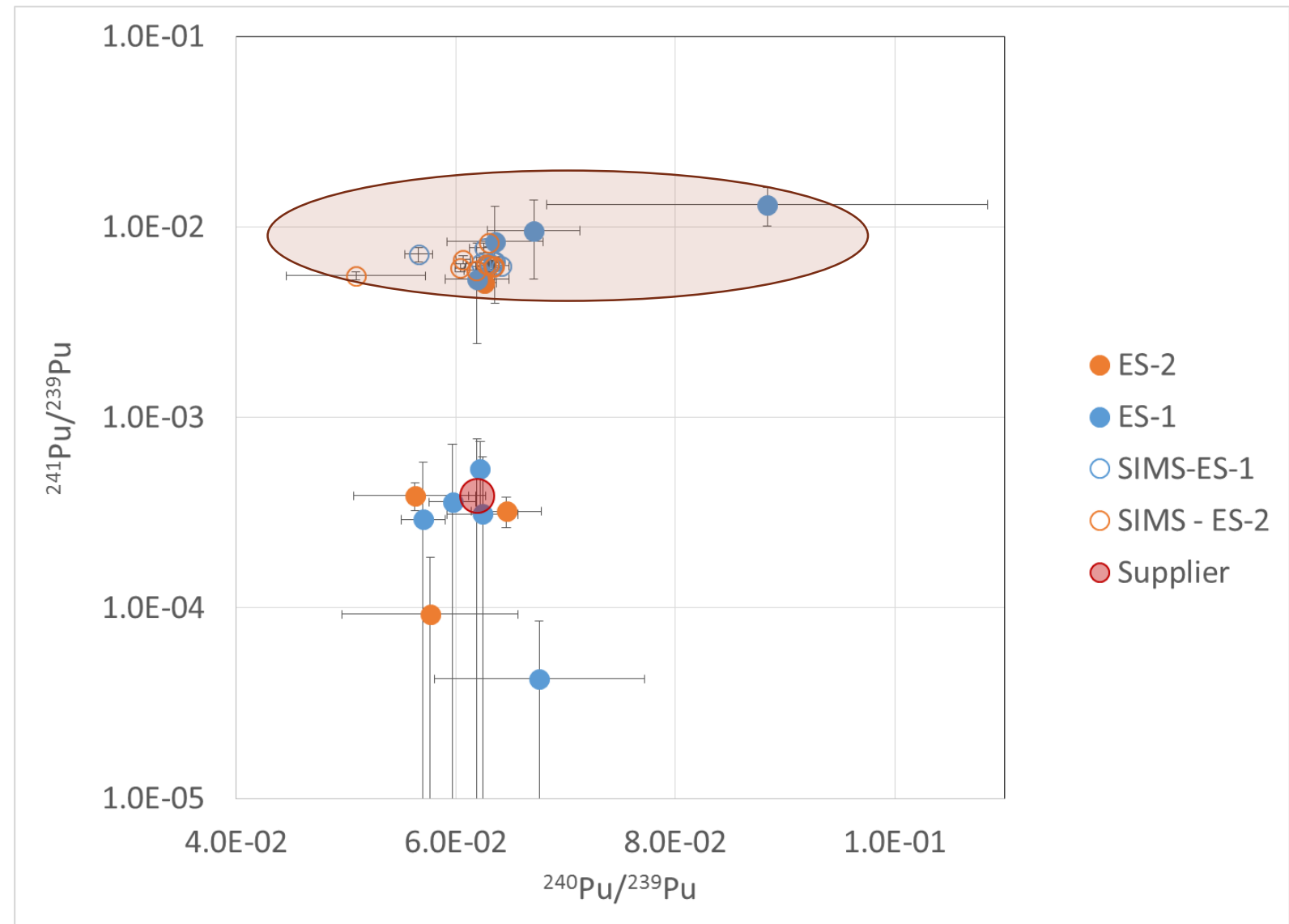
## Pu & Am Detection at 1 Week

- 10 Labs reporting  $^{241}\text{Am}$  &  $^{239}\text{Pu}$  values for ES-1 and ES-2
- Ngadi Chuli, Cho Oyo, Kamet, Gasherbrum, and Masherbrum report values for both ES-1 and ES-2
- 5 out of 6 labs show consistency between ES-1 and ES-2 based on this ratio
- Most values consistent with supplier declarations



## $^{240}\text{Pu}/^{239}\text{Pu}$ vs. $^{241}\text{Pu}/^{239}\text{Pu}$ at 1 Week

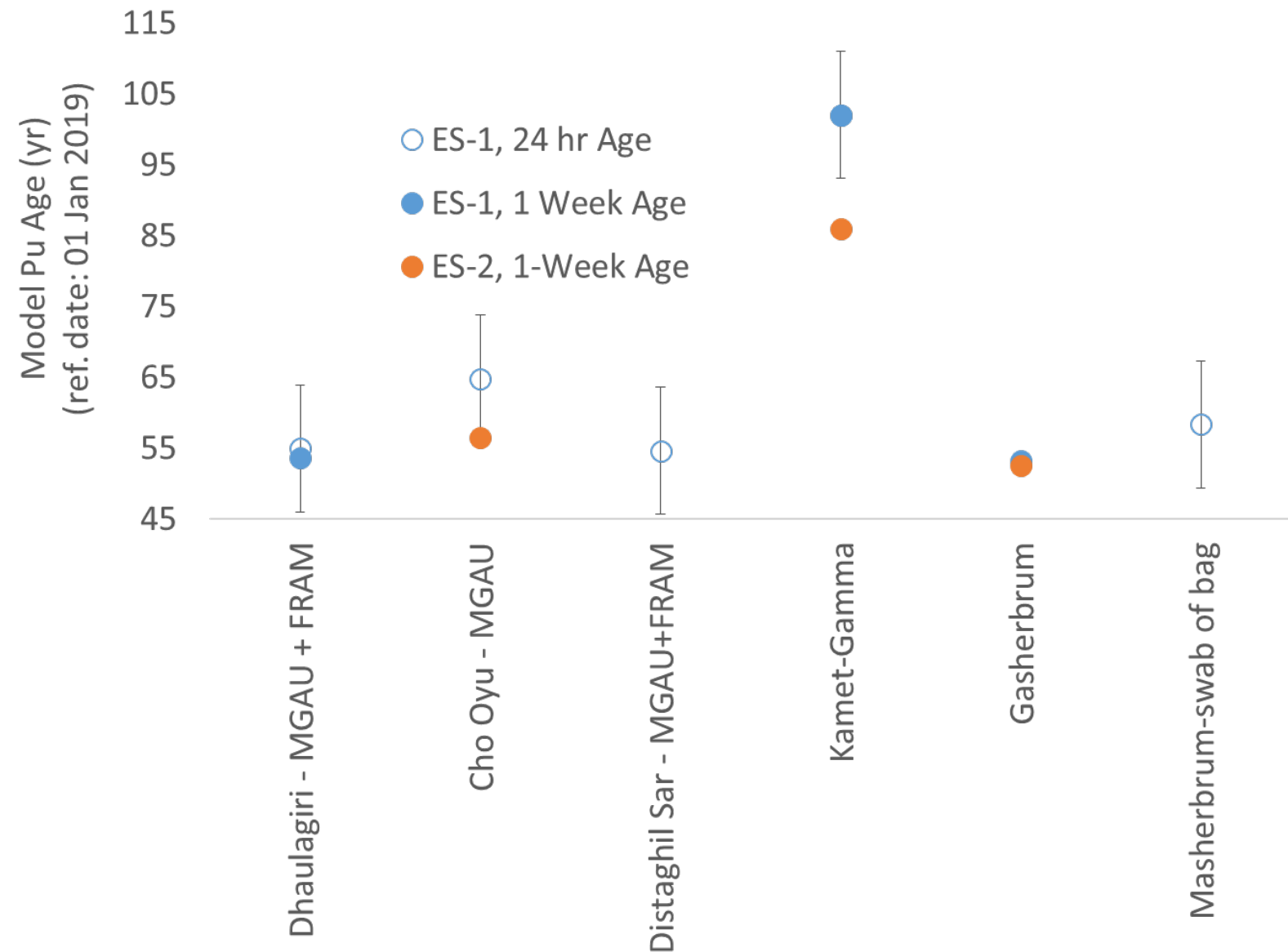
- 11 Labs reporting  $^{241}\text{Am}$  &  $^{239}\text{Pu}$  values for ES-1
- 11 Labs reporting  $^{241}\text{Am}$  &  $^{239}\text{Pu}$  values for ES-2
- Most values consistent with supplier declarations
- SIMS and “rapid” bulk MS methods not employing a separation step expected to be bias high due to isobaric interference with  $^{241}\text{Am}$





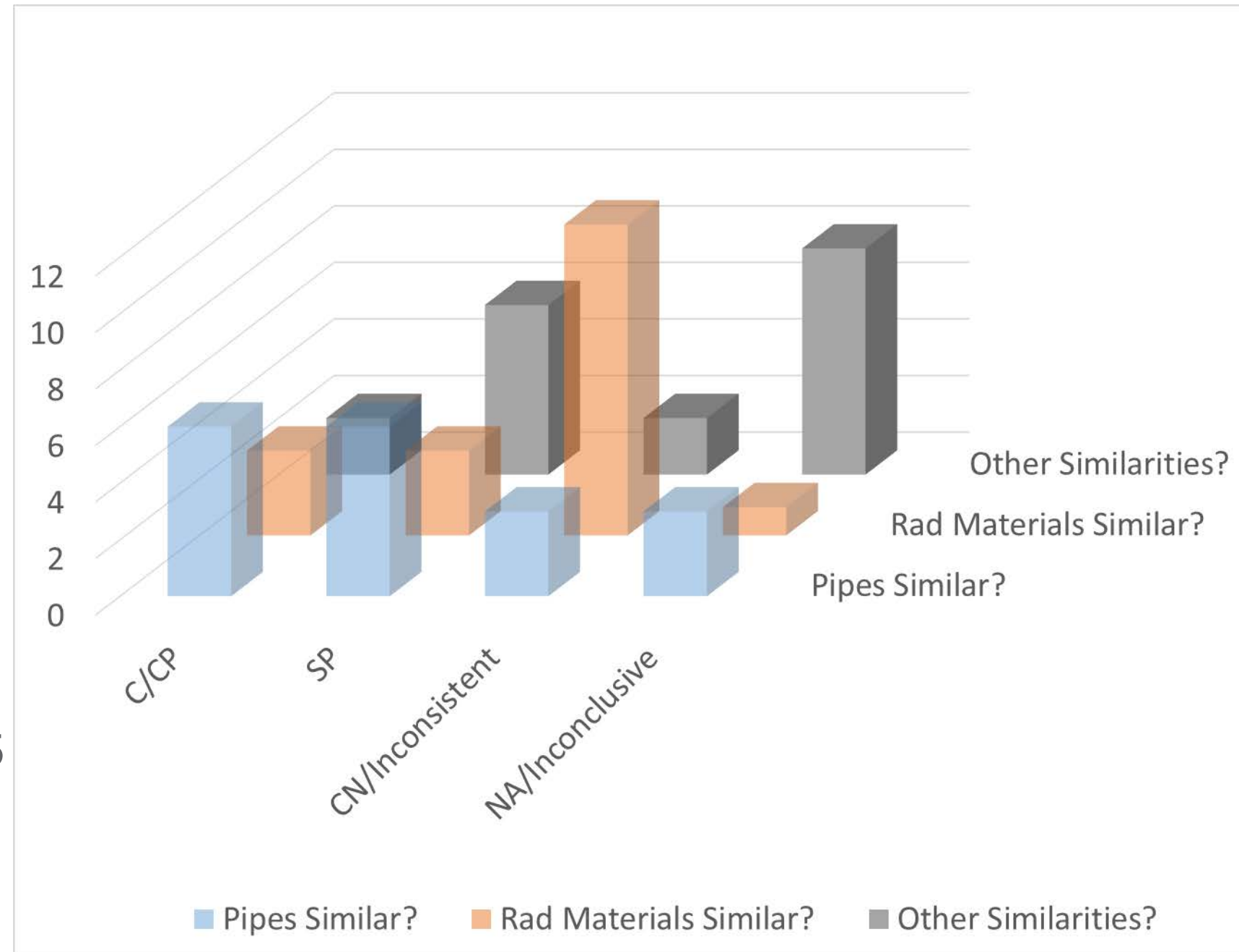
## Evaluation – Model Age of Pu

- Dhaulagiri, Cho Oyo, Kamet, and Gasherbrum reported new estimates of Am/Pu model age
- Gasherbrum and Cho Oyo show consistency in Am/Pu model ages for ES-1 and ES-2



## Evaluation – Graded Decision Framework

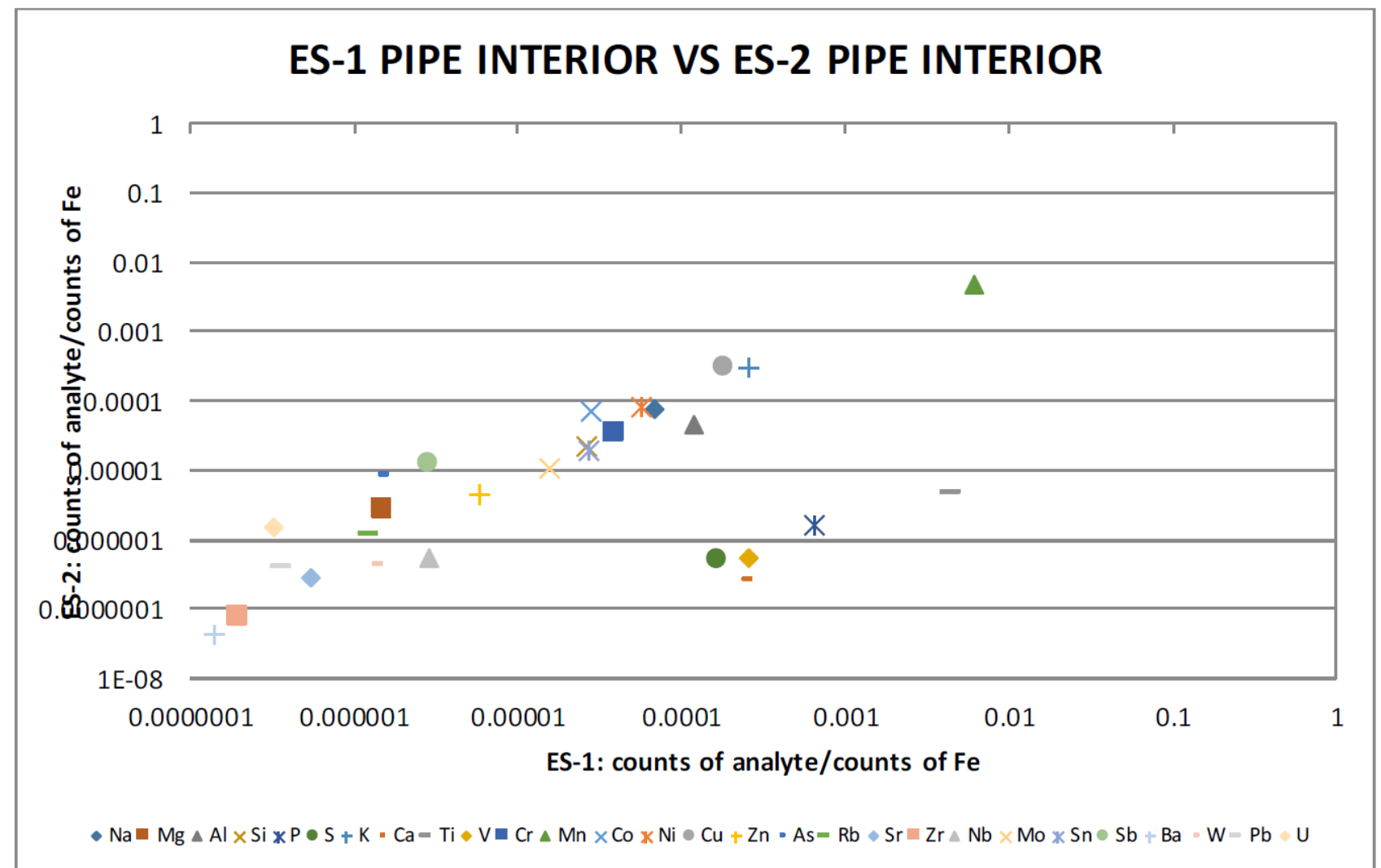
- 18 labs reporting GDF answers
- Indications of some inconsistency with interpretation of question 5 (...rad materials similar?)
  - Those considering radioactive contamination part of material answered CP
  - Those interpreting contamination as “Other evidence” answered CN to Q5 and CP to Q6





## Notable Efforts after 1 Week

- Chomo Lonzo, Batura Sar, Rakaposhi, and Annapurna – application of XRD within 1 Week for phase ID
- Exceptional evaluation of LA-QQQ-ICP-MS for analysis of trace elements, Everest



Trace element pattern comparison in ES-1 and ES-2 pipes (Everest)

## Notable Efforts in 1 Week

- K2 – Fiber analysis
- Rakaposhi – tool marks on bags
- Gasherbrum – detection of  $^{22}\text{Na}$



- Alpha energy barrier for this reaction is ~5MeV...suggesting the presence of Pu in fluoride form
- Tirich Mir & Ngadi Chuli - Group Inclusion / Exclusion using  $^{241}\text{Am}/^{239}\text{Pu}$



# Discussion – 1 Week

## Small Group Discussion

Group 1: Romania, Russia, Ukraine, Azerbaijan

Group 2: Japan, Korea, Singapore, Australia, USA

Group 3: Switzerland, Poland, Sweden, Germany, UK

Group 4: Hungary, Israel, JRC, Canada, France

Questions to discuss (spend 10-15 minutes per question):

- 1) Did you attempt to subsample the ingots for analysis? If so, discuss the techniques that were used to do this.
- 2) Did you attempt to segregate surface contamination from the bulk ingot for isotopic analysis? If so, discuss the different methods that were employed to do this.
- 3) What were the three most useful measurement techniques used during the first week of CMX-6?
- 4) What was the most challenging aspect of producing the 1 week report?
- 5) What would you have changed about your approach to the 1 week report?
- 6) What would you have changed about the execution of the exercise up to the 1-week reporting?





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## Operation Celestial Skónis



Inject 3 & Introduction to  
2-Month (Final) Report

6<sup>th</sup> Collaborative Materials  
Exercise (CMX-6) of the  
Nuclear Forensics International  
Technical Working Group  
(ITWG)

**Jon Schwantes & Olivia Marsden**  
Co-Chairs ETG, ITWG





## Inject 3 & Introduction to Final (2-Month) Report

- Exercise Objectives / Performance Metrics for Final Report
- Inject 3
- Schedule of lab presentations





# Exercise Objectives

Obj 1: Novel  
Materials

Obj 2: NDA  
Categorization  
(24 hrs)

Obj 3: 1-Week  
Characterization

Obj 4: 2 Month  
Characterization

Obj 5: Evaluation



Obj 4: 2 Month Characterization

Cap 4.1: Isotopics

Cap 4.2: Elemental/Chemical

Cap 4.3 Physical

Cap 4.4: Age Date

Cap 4.5: Traditional Forensics

Act 4.5.1: Fingerprinting

Act 4.5.2: Toolmarks

Task 4.5.1.1: Patent Prints

Task 4.5.1.2: Latent Prints

PI 4.1.1: Detect Pu in ES1 in 24 hrs  
 PI 4.1.2: Detect U in ES1 in 24 hrs  
 PI 4.1.3: Detect U in ES2 in 24 hrs  
 PI 4.1.4: Detect Pu in ES2 in 24 hrs

PI 4.2.1: ID Ce Metal in ES1 in 24 hrs  
 PI 4.2.2: ID trace in ES1 in 24 hrs  
 PI 4.2.3: ID U metal in ES2 in 24 hrs  
 PI 4.2.4: ID trace in ES2 in 24 hrs

PI 4.3.1: Microscopy of pipes  
 PI 4.3.2: Microscopy of ES's

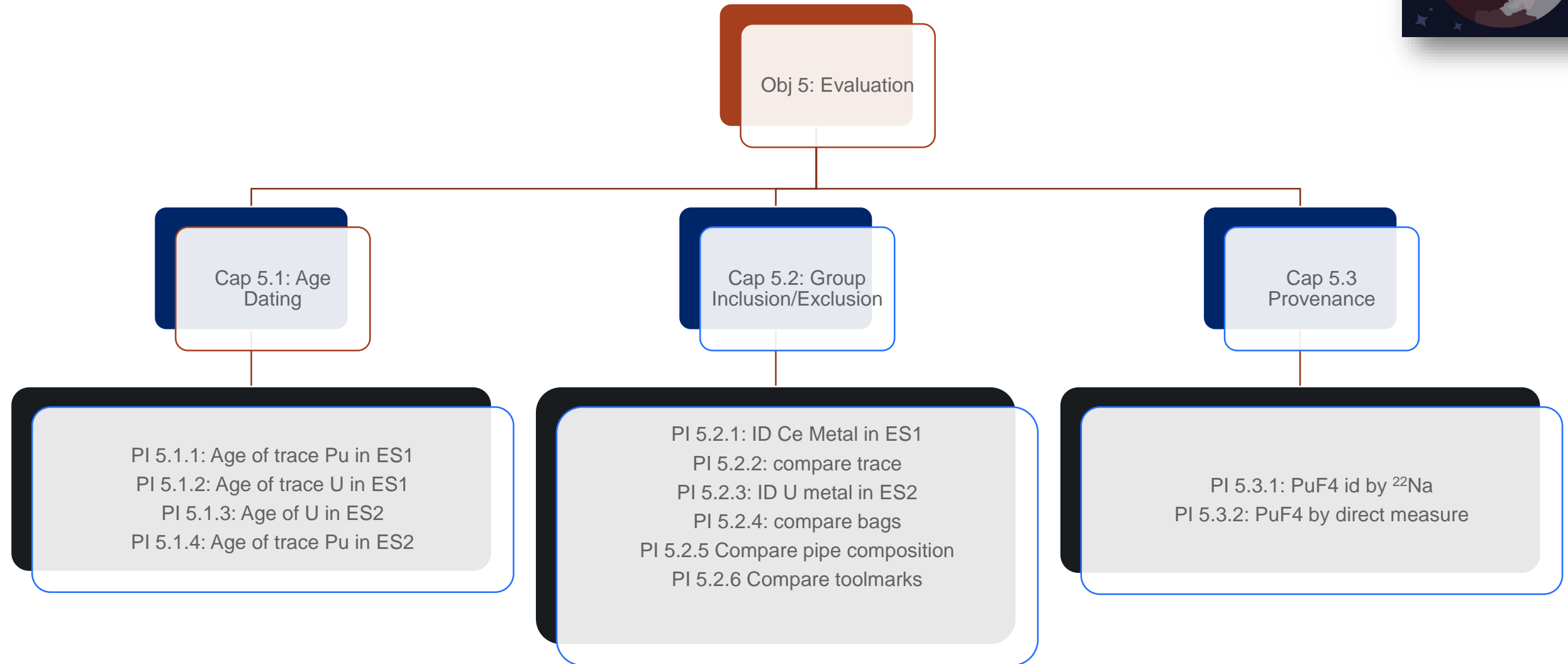
PI 4.4.1: U age  
 PI 4.4.2: Pu age

PI 4.5.1.1.1: Locate  
 PI 4.5.1.1.2: Image

PI 4.5.1.2.1: Develop  
 PI 4.5.1.2.2: Image

PI 4.5.2.1: Image and compare cut surfaces on pipes  
 PI 4.5.2.2: Image and compare cut surfaces on ES's







Case No. 52521, Sample  
ID: ES-3A & ES-3B

## Operation Celestial Skónis

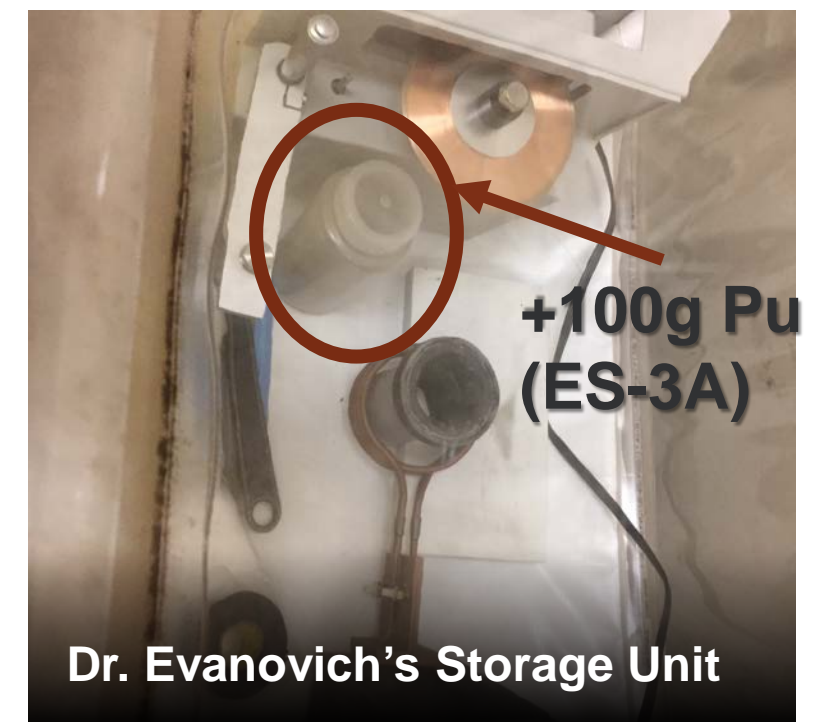


Inject 3 – Final Report

- Dr. Evanovich, staff scientist at Rodesia National Laboratory (RNL), Person Of Interest (POI)
- POI had access to large quantities of SNM located at RNL
- Determine if materials recovered at storage unit are consistent with ES-1 or ES-2 or RNL holdings



Dr. Evanovich's Storage Unit



Dr. Evanovich's Storage Unit





**RNL Holdings**

Sample ID	Atomic Percent				Amount (g)	Date Measured	Chemical Form				
	<sup>235</sup> U	Uncertainty (2-σ)	<sup>238</sup> U	Uncertainty (2-σ)							
RNL-5435	8.500E-01	1.500E-01	8.670E+01	1.500E+00	5.700E-01	8.000E-02	1.100E+01	9.000E-01	1.000E+03	18-Sep-13	Oxide
RNL-789U	1.050E+00	7.000E-02	8.940E+01	1.800E+00	6.900E-01	5.000E-02	8.900E+00	2.000E-01	2.500E+02	2-Jun-06	Oxide
RNL-540G	1.930E-02	2.702E-04	2.245E+00	1.700E-01	2.000E-03	5.000E-04	9.702E+01	8.000E-02	2.412E+03	14-Nov-09	Fluoride
RNL-095W	9.291E-01	6.000E-03	78.96E+00	1.000E-01	7.95E-01	5.00E-02	1.02E+02	1.000E-01	1.000E+03	1-Jun-14	Oxide
RNL-118L	8.640E-01	6.600E-03	6.712E+00	1.000E-01	3.005E-01	3.00E-02	1.000E+01	1.000E-01	1.000E+03	4-Jun-08	Oxide
RNL-992R	bd	bd	2.400E-01	0.000E-02	bd	bd	9.972E+01	6.000E-02	1.000E+03	13-Jun-18	Fluoride
RNL-733Y	9.093E-01	4.000E-03	7.778E+01	1.100E-02	4.600E-02	1.200E-01	2.127E+01	6.400E-02	1.289E+03	4-Aug-11	Metal
RNL-629F	3.430E-02	1.200E-03	2.893E+00	9.300E-02	3.000E-04	5.000E-04	6.708E+01	5.000E-02	1.299E+03	14-Nov-14	Nitrate
RNL-373M	bd	bd	1.900E-01	6.000E-02	bd	bd	9.980E+01	6.000E-02	3.132E+03	8-Apr-11	Oxide

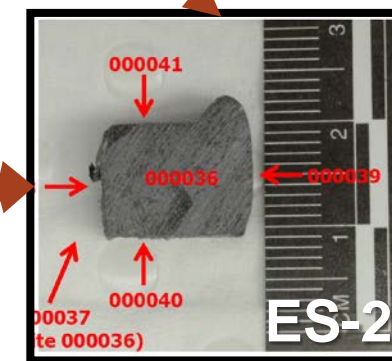
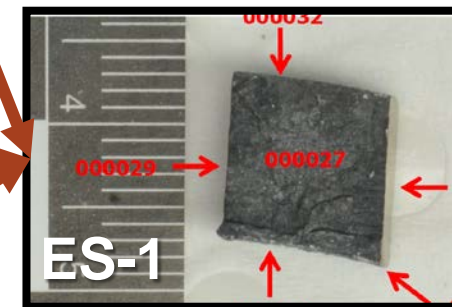
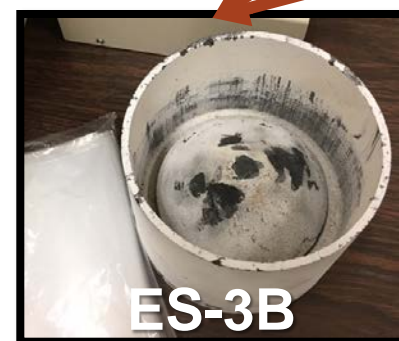
  

Sample ID	Atomic Percent				Amount (g)	Date Measured	Chemical Form	Last Processing Date		
	<sup>239</sup> Pu	Uncertainty (2-σ)	<sup>240</sup> Pu	Uncertainty (2-σ)						
RNL-132D	9.153E+01	4.595E+00	8.210E+00	9.832E-01	3.760E-02	4.920E-03	6.450E+02	3-Mar-11	Metal	Jan-82
RNL-235A	8.120E+01	4.060E+00	1.870E+01	9.348E-01	1.050E-01	5.250E-03	9.230E+02	18-Dec-08	Oxide	Apr-71
RNL-194L	9.340E+01	3.736E+00	6.567E+00	8.340E-01	4.000E-02	5.200E-03	1.400E+02	26-Feb-15	Fluoride	Jul-64
RNL-051H	9.341E+01	4.222E+00	6.698E+00	8.770E-01	4.700E-02	6.300E-03	3.540E+02	27-Nov-14	Oxide	<1956

# Operation Celestial Skónis



Inject 3 – Final Report



Q3?

Q2?

Q1?

Q4?

Q3?

Q4?

Using the Graded Decision Framework, answer questions 1-4.



**Thank you**



# Laboratory Presentations – Final Reporting

<b>9:15 – 9:30 am</b>	Welcome, Schedule & Announcements	Olivia
<b>9:30 – 9:50 am</b>	2-Month report results, U only	Hungary
<b>9:50 – 10:10 am</b>	2-Month report results	Canada
<b>10:10 – 10:30 am</b>	2-Month results	UK
<b>10:30 – 10:50 am</b>	Break	
<b>10:50 – 11:10 am</b>	2-Month report results	Switzerland
<b>11:10 – 11:30 am</b>	Novel Methodologies 2-Month – (Raman/SEM)	France
	Novel Methodologies 2-Month – Radiochronometry	USA





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## Operation Celestial Skónis



6<sup>th</sup> Collaborative Materials  
Exercise (CMX-6) of the  
Nuclear Forensics International  
Technical Working Group  
(ITWG)

**Jon Schwantes & Olivia Marsden**  
Co-Chairs ETG, ITWG

## Summary of 2-Month Reporting





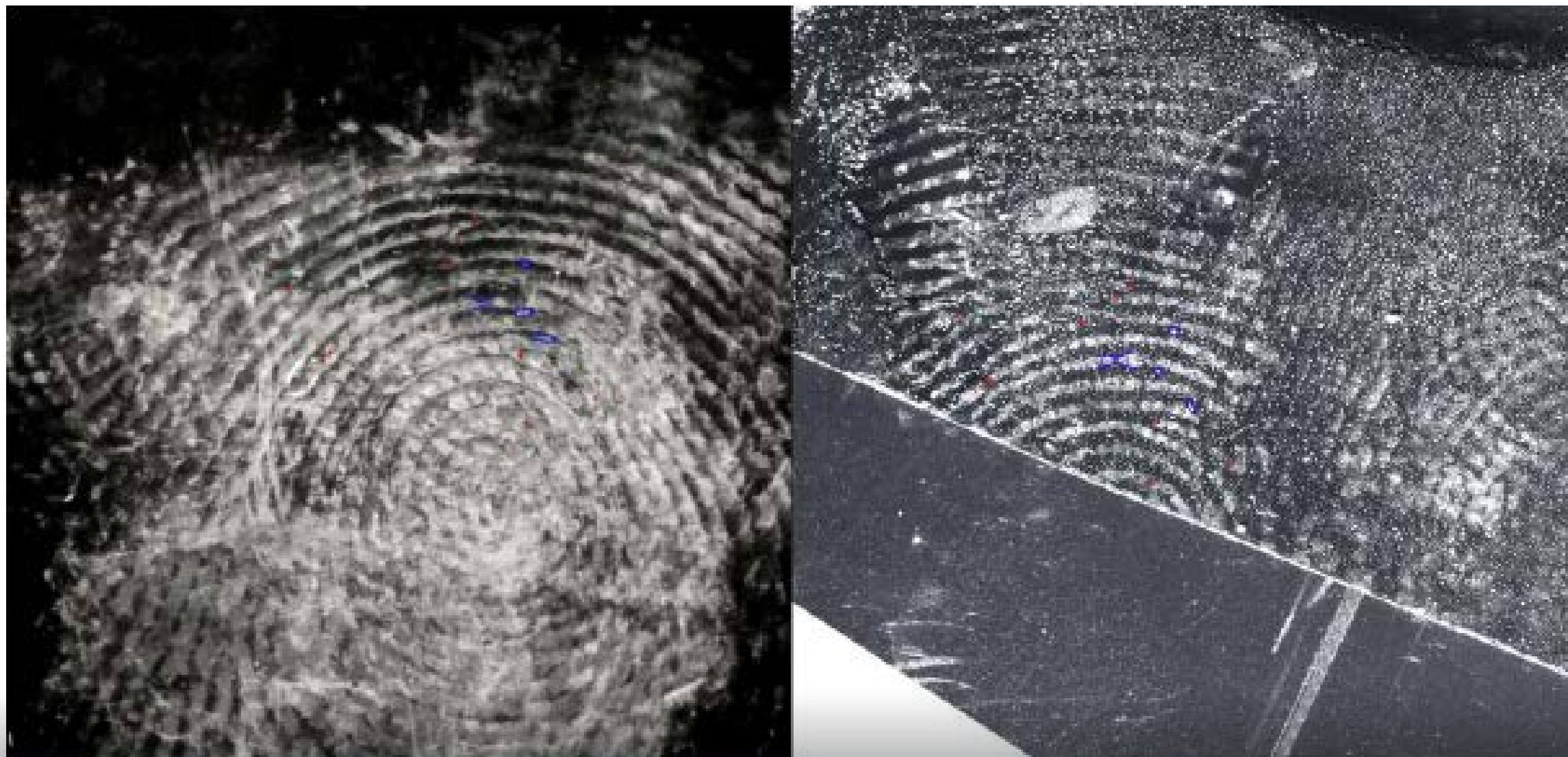
# Traditional Forensics

- 8 labs developed fingerprints during CMX-6
  - Most reported those results in 1 week
- Everest and Rakaposhi performed DNA analysis
- 4 labs performed tool mark analysis on ingots
- 9 labs performed tool marks on pipes
- Kanjut Sar conducted tool mark analysis on plastic bags
- K2 collected fiber and insect evidence

Code	Fingerprints			DNA			tool marks bags			tool marks ingots			tool marks pipes			Fiber			Insect			Worked with LE	
	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn		
Kanjut Sar		■							■		■		■										■
Dhaulagiri																							
Everest	■			■								■											■
Ngadi Chuli																							■
Cho Oyu	■									■													■
Chomo Lonzo			■											■									■
Nanda Devi																							■
K2		■										■			■		■			■			
Makalu		■																					■
Himalchuli																							
Namcha Barwa																							
Tirich Mir													■										■
Batura Sar													■										
Shispare													■										
Distaghil Sar																							
Rakaposhi		■		■																			■
Kamet																							
Annapurna											■												■
Gasherbrum													■										
Masherbrum		■									■		■										■

Summary of the application of traditional forensics during CMX-6

## Matching Latent Prints on ES-1 & ES-2



Successful match by Kanjut Sar of latent fingerprints collected on ES-1 (left) and ES-2 (right). Red markings indicate corresponding features between the two latents. Blue markings indicate ridge shape features of interest to examiner.



# Physical Characterization

- Most labs characterized mass, dimensions, and densities within the first 24 hrs
- 8 labs utilized x-ray radiography, most within 24 hrs
- 9 labs conducted OM, most within 1<sup>st</sup> week of exercise
- 16 labs conducted SEM, most within 1 week
- Kanjut Sar, Annapurna & Masherbrum surface roughness

Code	Mass			Density			Dimensional Analysis			X-ray radiography			Surface Roughness			Optical Microscopy			SEM			
	24 hr	1 wk	2 mth	24 hr	1 wk	2 mth	24 hr	1 wk	2 mth	24 hr	1 wk	2 mth	24 hr	1 wk	2 mth	24 hr	1 wk	2 mth	24 hr	1 wk	2 mth	
Kanjut Sar																						
Dhaulagiri																						
Everest																						
Ngadi Chuli																						
Cho Oyu																						
Chomo Lonzo																						
Nanda Devi																						
K2																						
Makalu																						
Himalchuli																						
Namcha Barwa																						
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Distaghil Sar																						
Rakaposhi																						
Kamet																						
Annapurna																						
Gasherbrum																						
Masherbrum																						

Summary of the application of traditional forensics during CMX-6

## Phase ID / Chemical / Elemental Analysis

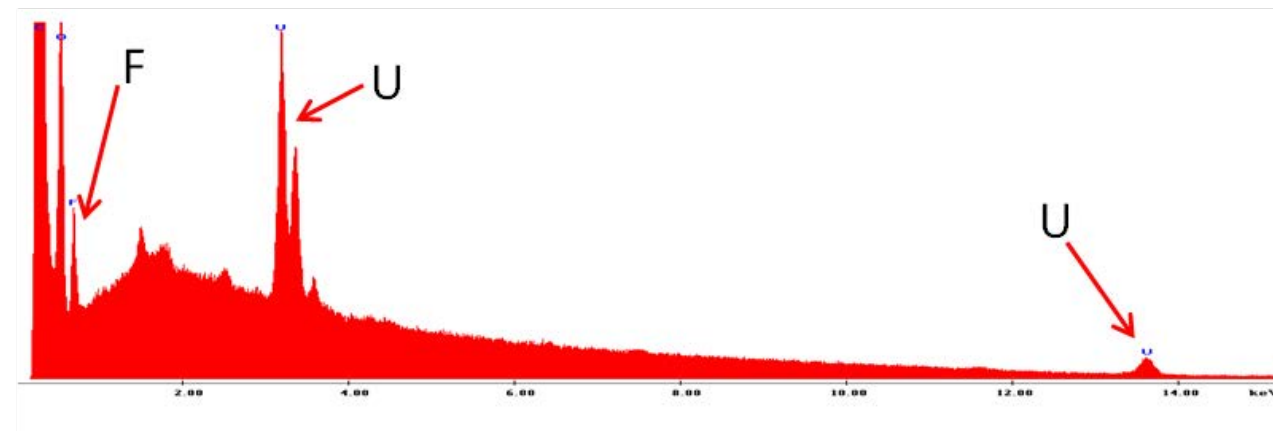
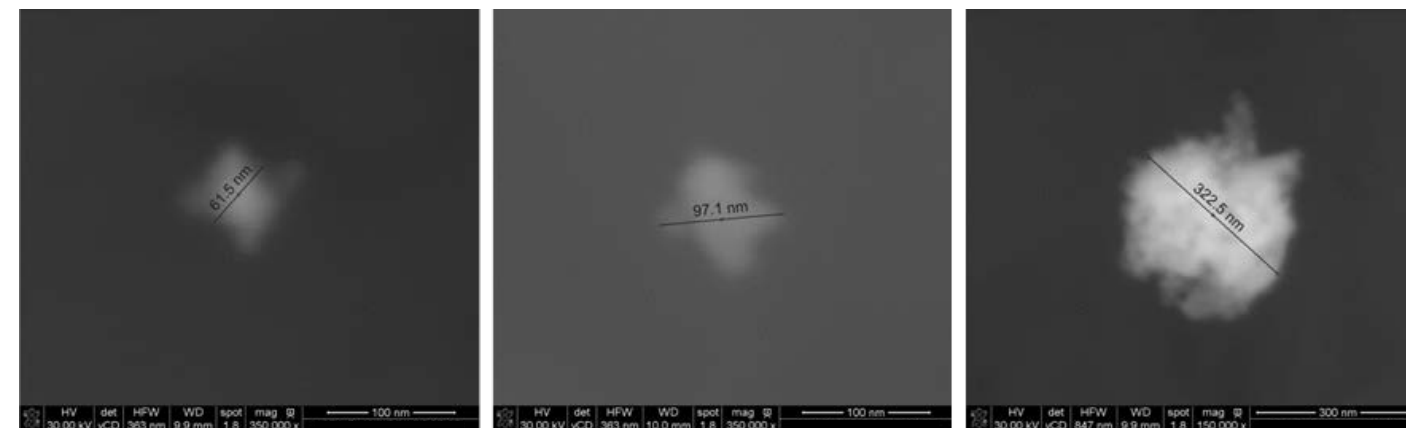
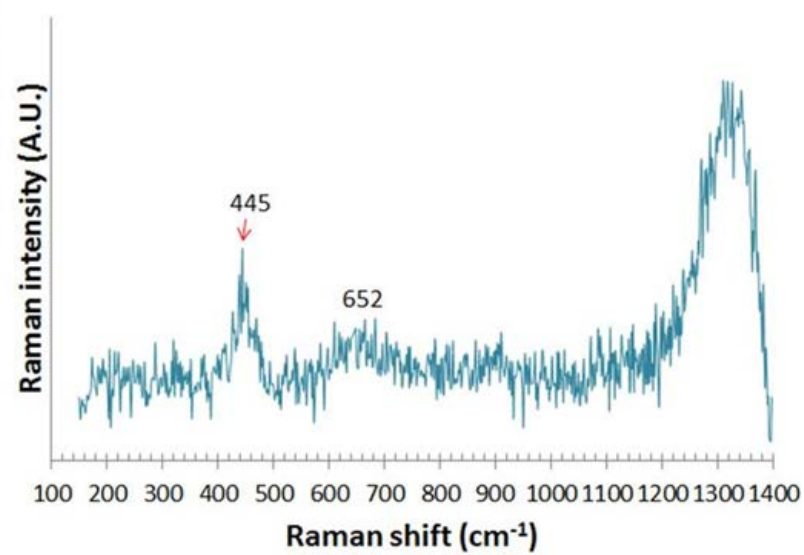
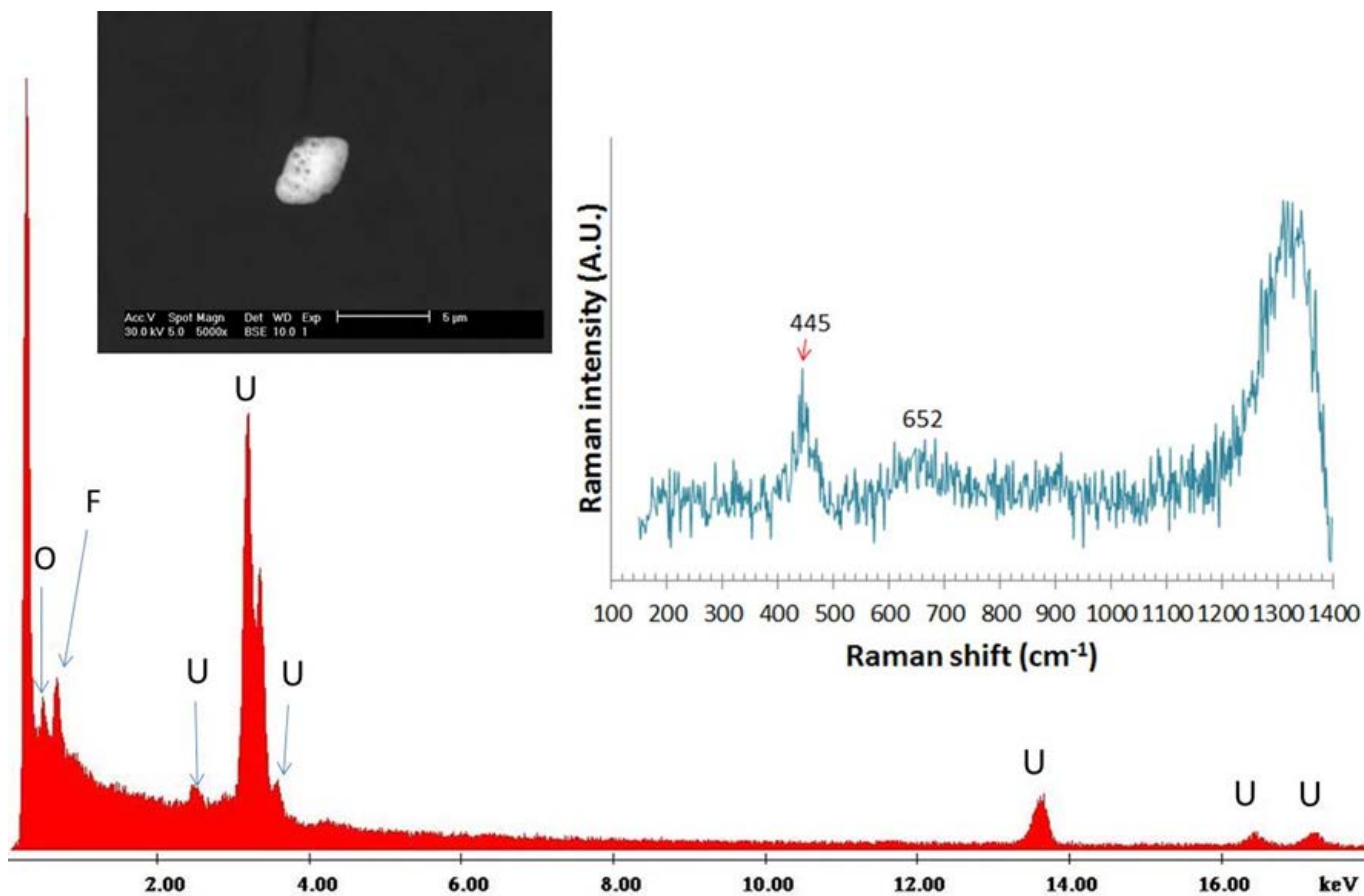
- 7 labs employed pXRD, most in 1 week
- 3 labs used Raman spectroscopy within 2 months
- Masherbrum employed LIBS
- Most labs employed XRF, half of those during 24 hrs

Code	Elemental Surface									Elemental Bulk															
	Powder XRD			Raman			LIBS			XRF			EDX			LA-ICPMS			ICP-OES			ICP-MS			
	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	24 hr	1 wk	2 mn	
Kanjut Sar			■							■															■
Dhaulagiri										■															
Everest			■							■					■										■
Ngadi Chuli						■				■					■										
Cho Oyu										■					■									■	
Chomo Lonzo		■								■															
Nanda Devi						■				■														■	
K2										■															■
Makalu										■															■
Himalchuli										■															
Namcha Barwa															■									■	
Tirich Mir										■					■										■
Batura Sar		■								■					■									■	
Shispare															■										■
Distaghil Sar			■			■				■															■
Rakaposhi		■								■															
Kamet										■										■				■	
Annapurna		■								■		■			■										
Gasherbrum															■										
Masherbrum								■		■					■										

Summary of Phase ID and Trace Element Analysis during CMX-6



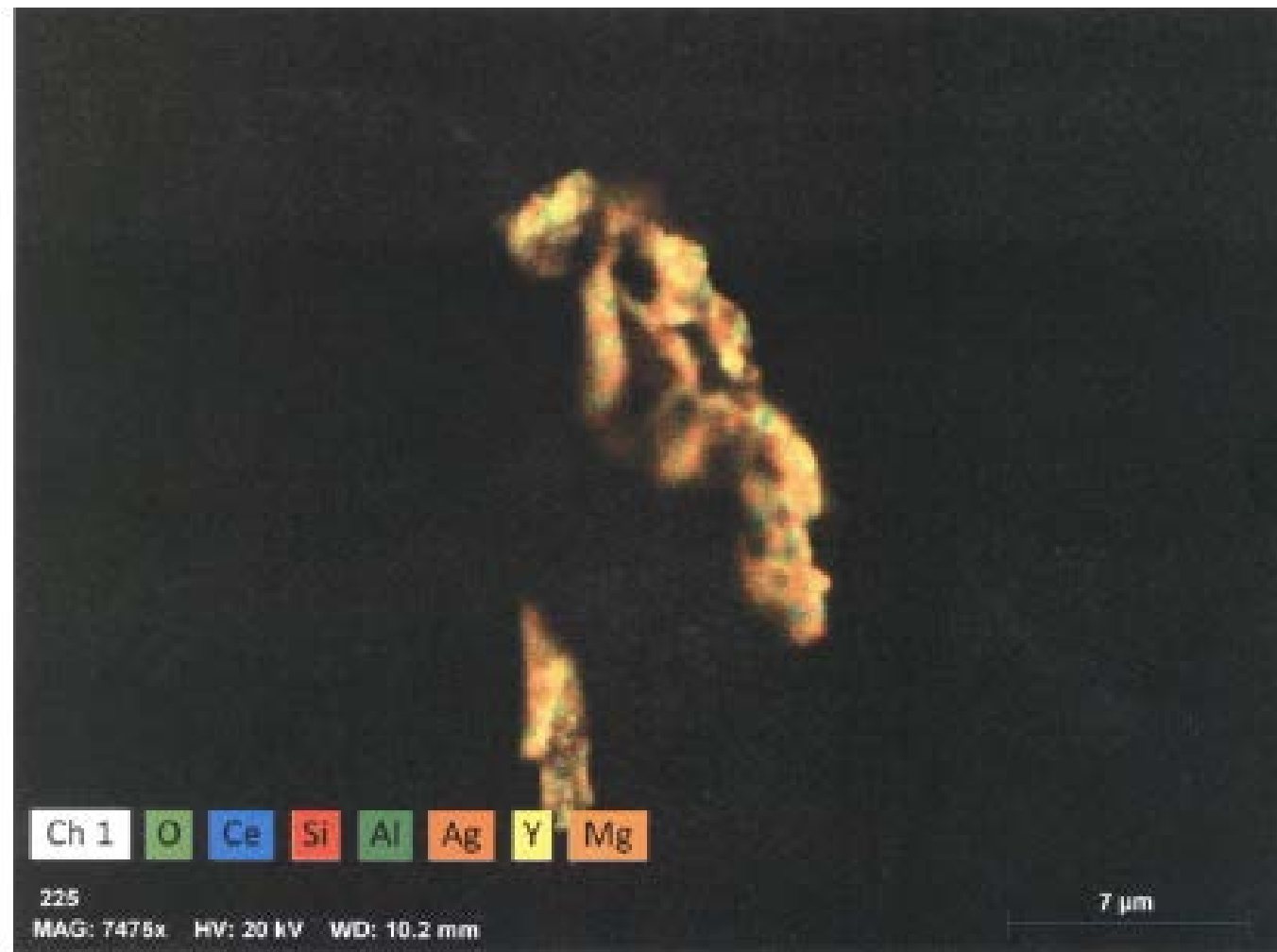
# Outstanding Example by Ngadi Chuli of Physical and Trace Elemental Analysis during CMX-6



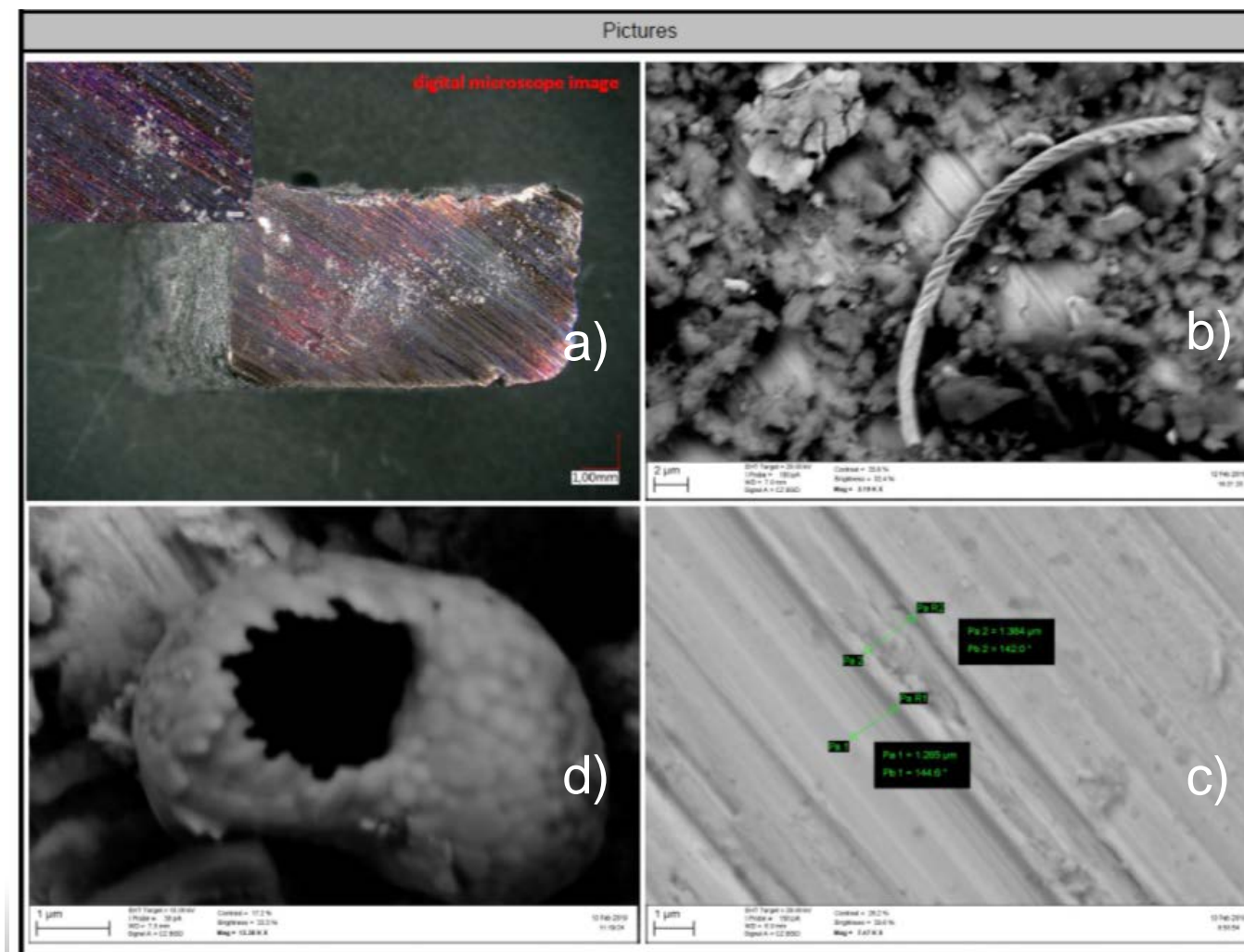
Typical example of results obtained by combined SEM – Raman spectrometry analysis of a UO<sub>2</sub> particle (sample ES-1, particle #4): EDX spectrum (bottom) showing presence of U as the major compound, along with F and O, electronic image (top, left) of the particle and Raman spectrum (top, right) showing the bands at 445 cm<sup>-1</sup> which is characteristic of UO<sub>2</sub>.

Examples of electronic images of very small (60 – 500 nm) U particles sampled on the surface of the sample ES-2 and a typical EDX spectrum obtained for one of these particles. F was detected as a minor constituent in all of the particles. A low abundance of O was also detected in all of these particles.

# Outstanding Example by Nanda Devi and Cho Oyo of Physical and Trace Elemental Analysis



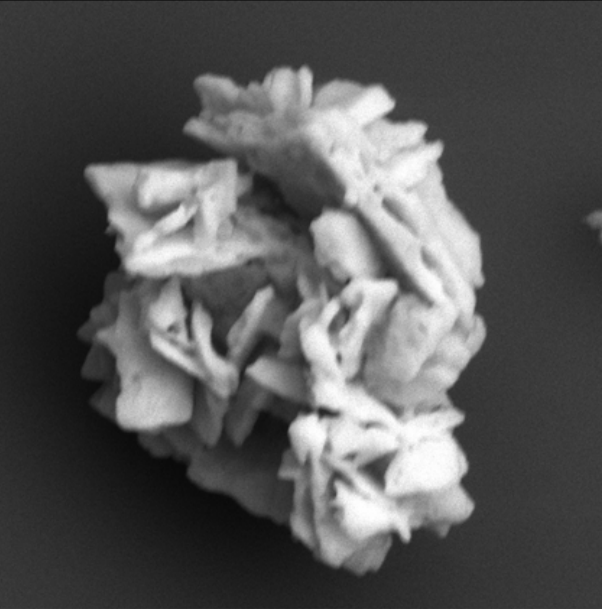
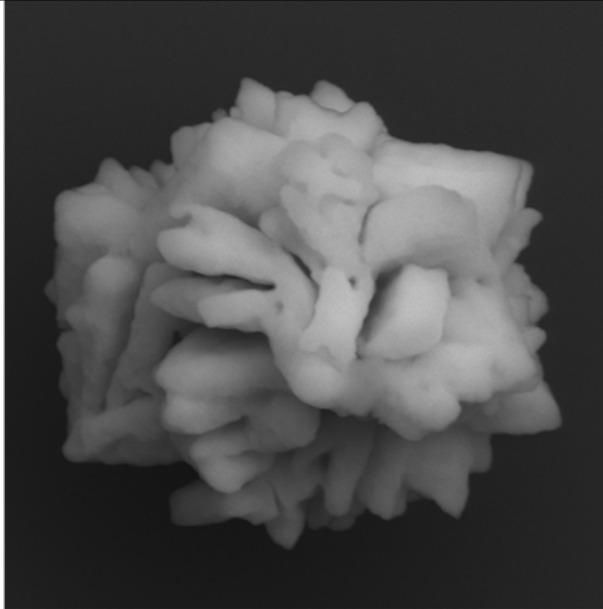
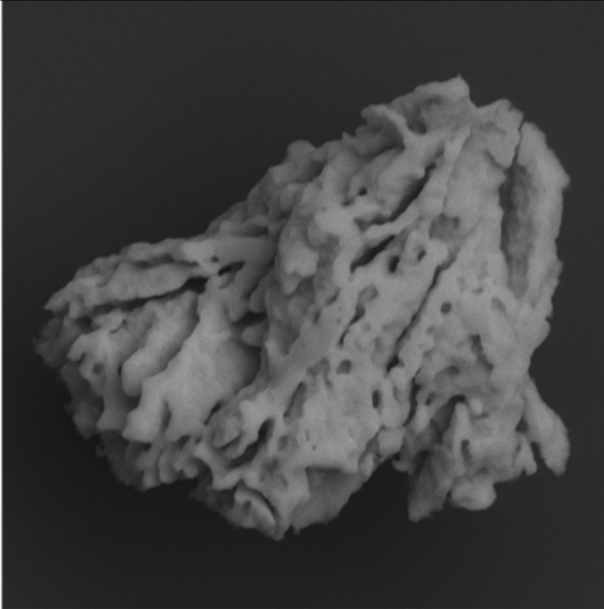
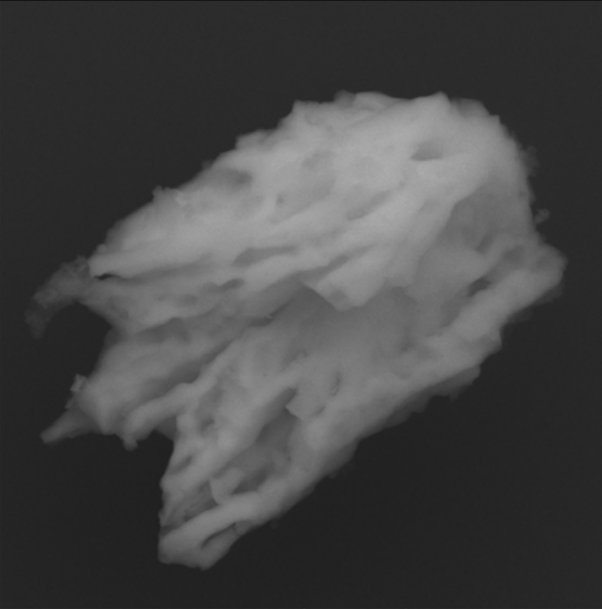
SEM-EDX of ES-1 performed by Nanda Devi using special purpose hermetic cell to seal potentially dispersible radioactive contamination from the instrument during analysis. Analysis shows detection of Al and Y, among other elements.



Tool mark analysis by Cho Oyo of cut edge of pipe. (a) Striations suggest straight cutting tool (band, jigsaw or large diameter circular saw). (b) Possible saw fragment. (c) Saw marks over the diameter of the wire found. (d) Hollow (possibly aerosol) particle consisting of mainly U.



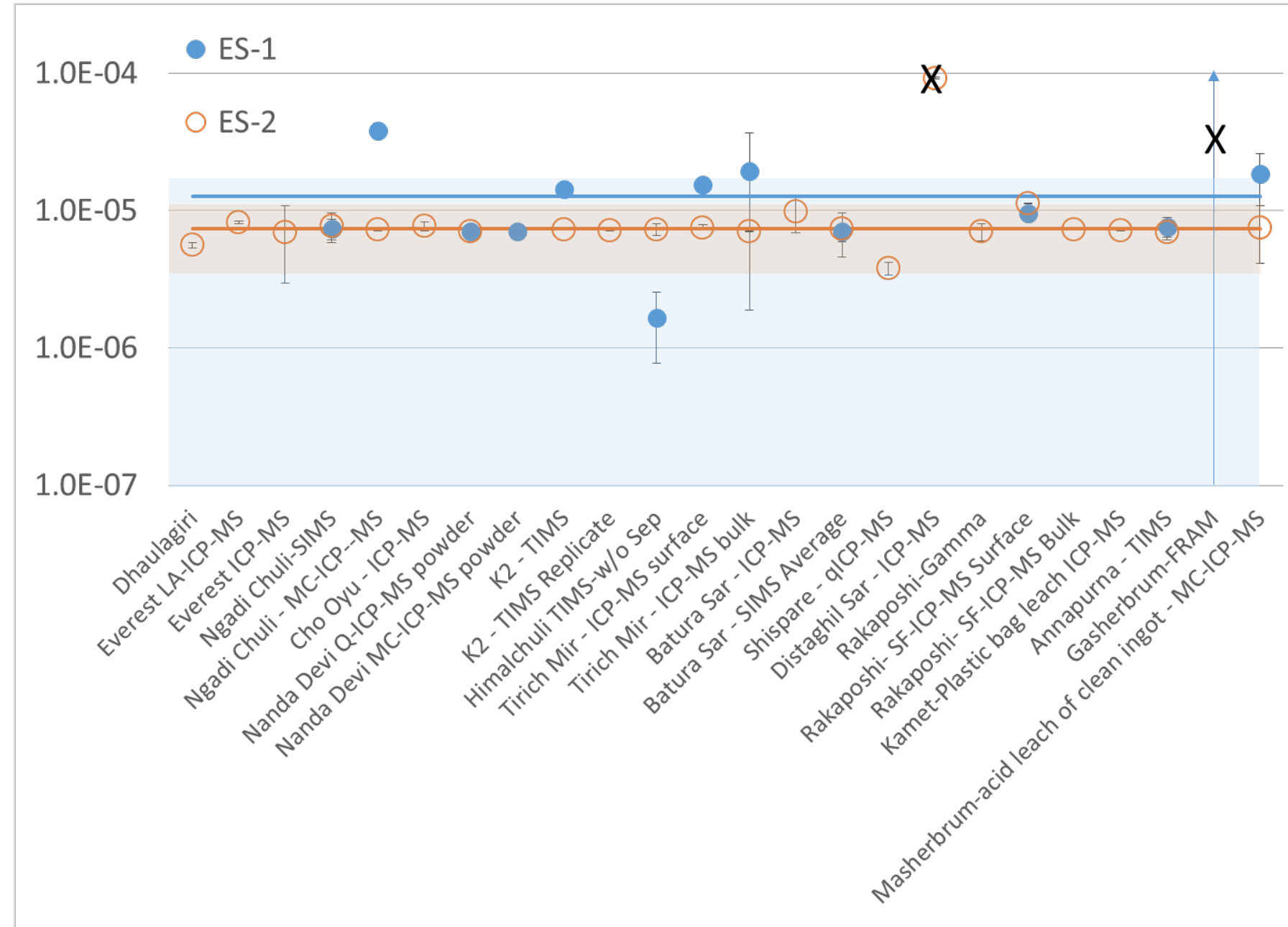
# Outstanding Example by Batura Sar of Physical and Trace Elemental Analysis

ES-1	ES-2	ES-1	ES-2
 <p>SEM HV: 15.0 kV    WD: 9.00 mm    LYRA3 TESCAN        View field: 8.00 μm    Det: BSE    2 μm        Performance in nanospace</p>	 <p>SEM HV: 30.0 kV    WD: 9.00 mm    LYRA3 TESCAN        View field: 9.00 μm    Det: BSE    2 μm        Performance in nanospace</p>	 <p>SEM HV: 15.0 kV    WD: 9.00 mm    LYRA3 TESCAN        View field: 12.0 μm    Det: BSE    2 μm        Performance in nanospace</p>	 <p>SEM HV: 30.0 kV    WD: 9.00 mm    LYRA3 TESCAN        View field: 6.00 μm    Det: BSE    1 μm        Performance in nanospace</p>
<p>Approximate quantification of X-Ray spectrum:        Pu – 83.6%, O – 9.5%, F – 5.1%,        Al, Ni and Fe – less than 1%.</p> <p>Concentration of Pu-239 – (93.518 ± 0.003)%.</p>	<p>Approximate quantification of X-Ray spectrum:        Pu – 82.2%, O – 3.3%, F – 14.0%,        Ni – less than 1%.</p> <p>Concentration of Pu-239 – (93.504 ± 0.005)%.</p>	<p>Approximate quantification of X-Ray spectrum:        Pu – 85.2%, O – 7.0%, F – 5.7%,        Al, Ni, Cr and Fe – less than 1%.</p> <p>Concentration of Pu-239 – (93.473 ± 0.005)%.</p>	<p>Approximate quantification of X-Ray spectrum:        Pu – 82.8%, O – 9.5%, F – 6.0%,        Al, Ni and Fe – less than 1%.</p> <p>Concentration of Pu-239 – (93.545 ± 0.003)%.</p>

Morphological and compositional comparisons of two Pu particles collected from ES-1 and ES-2 (Batura Sar).

## Isotopic Results – $^{234}\text{U}/^{238}\text{U}$

- 12 labs reported values for ES-1
- 20 labs reported values for ES-2
- Of the 11 labs reporting for both ES-1 & ES-2, 5 labs found both to be consistent relative to  $^{234}\text{U}/^{238}\text{U}$  ratio

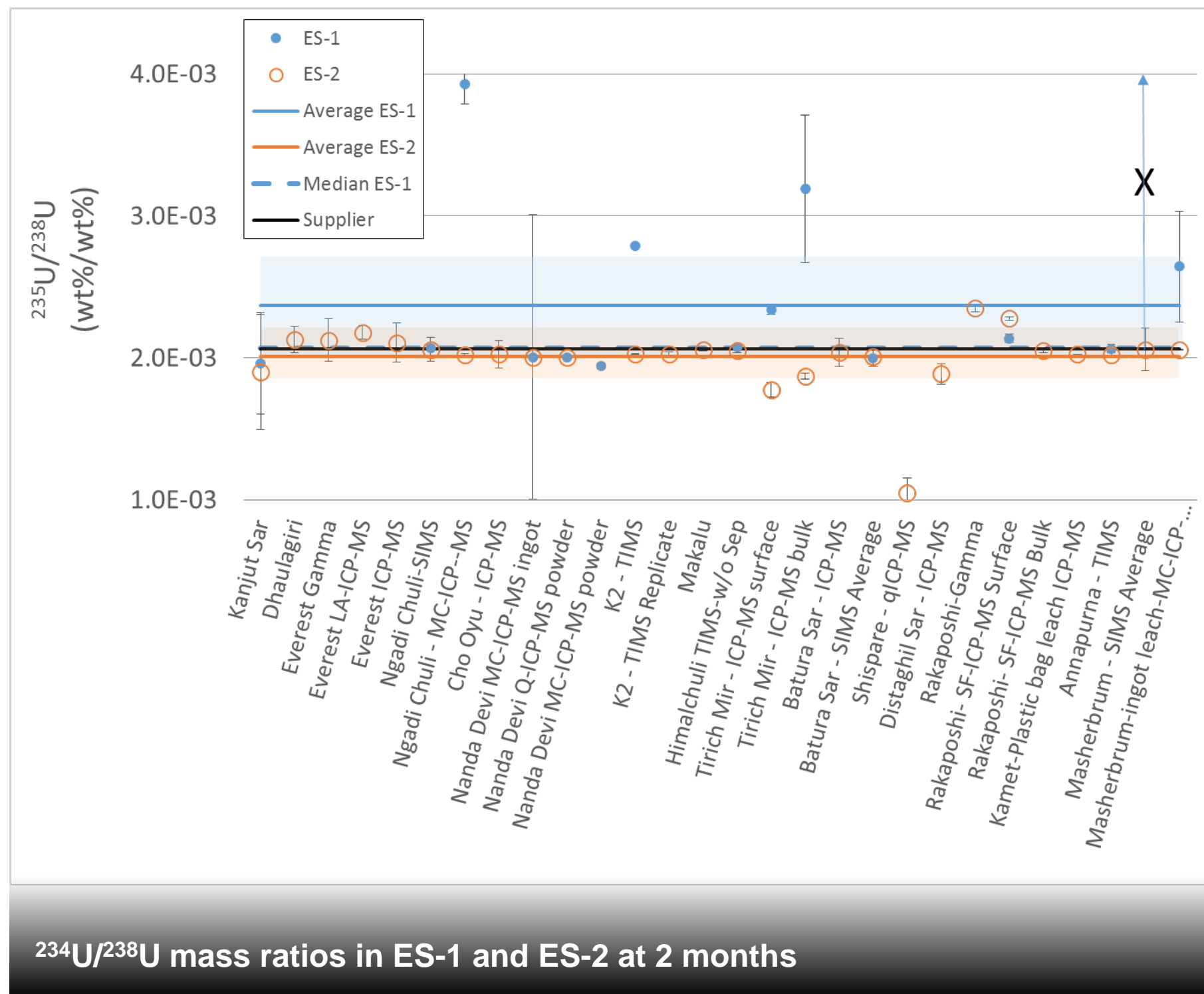


$^{234}\text{U}/^{238}\text{U}$  mass ratios in ES-1 and ES-2 at 2 months



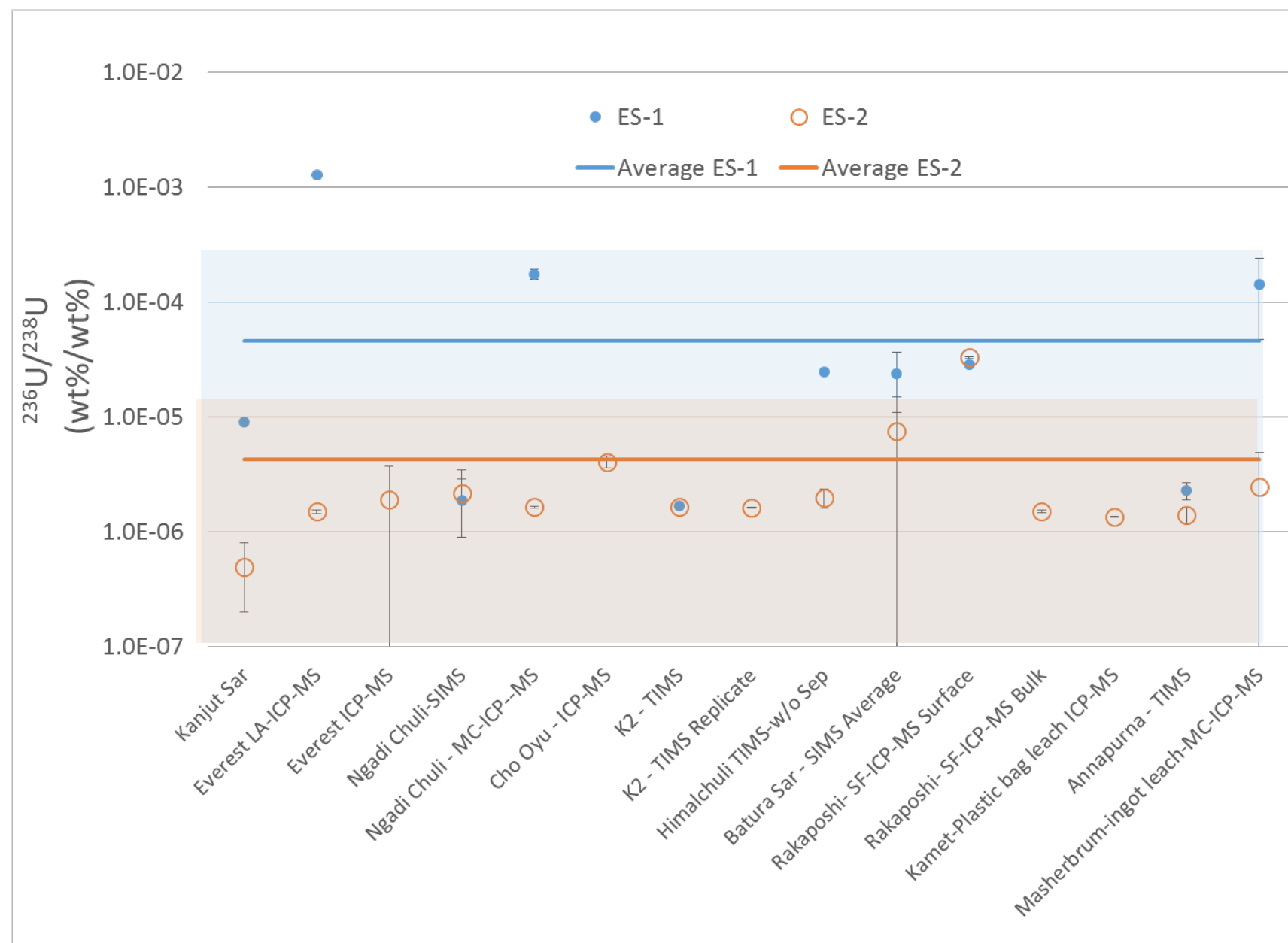
## Isotopic Results – $^{235}\text{U}/^{238}\text{U}$

- 9 labs reported values for ES-1
- 16 labs reported values for ES-2
- Of the 13 labs reporting for both ES-1 & ES-2, 8 labs found both to be consistent relative to  $^{235}/^{238}$  ratio
- With few exceptions, labs consistent with Supplier Declarations



## Isotopic Results – $^{236}\text{U}/^{238}\text{U}$

- 9 labs reported values for ES-1
- 11 labs reported values for ES-2
- Of the 8 labs reporting for both ES-1 & ES-2, 3 labs found both to be consistent relative to  $^{236}\text{U}/^{238}\text{U}$  ratio

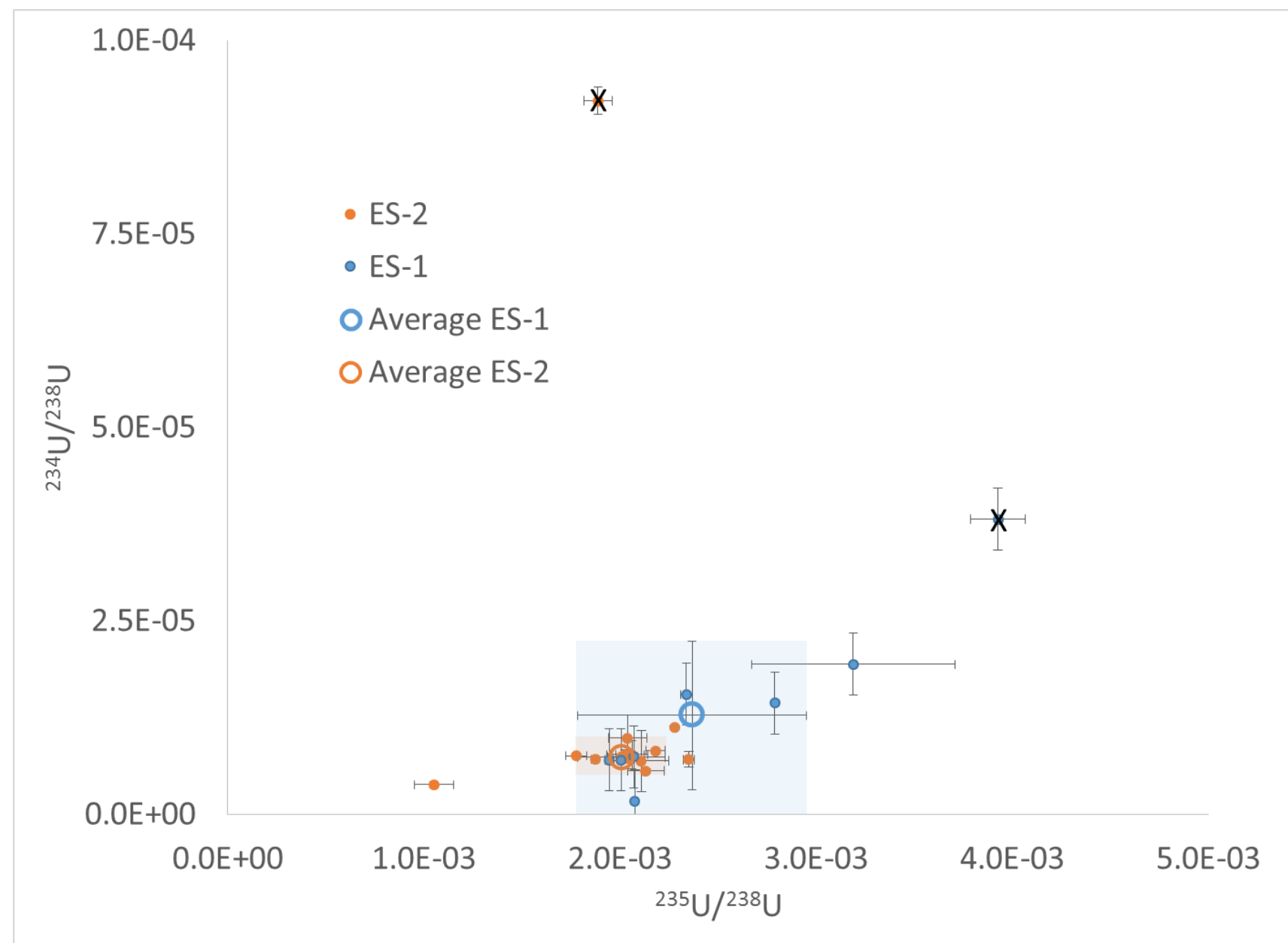


$^{236}\text{U}/^{238}\text{U}$  mass ratios in ES-1 and ES-2 at 2 months



## Tri-plot of $^{235}\text{U}/^{238}\text{U}$ vs $^{234}\text{U}/^{238}\text{U}$

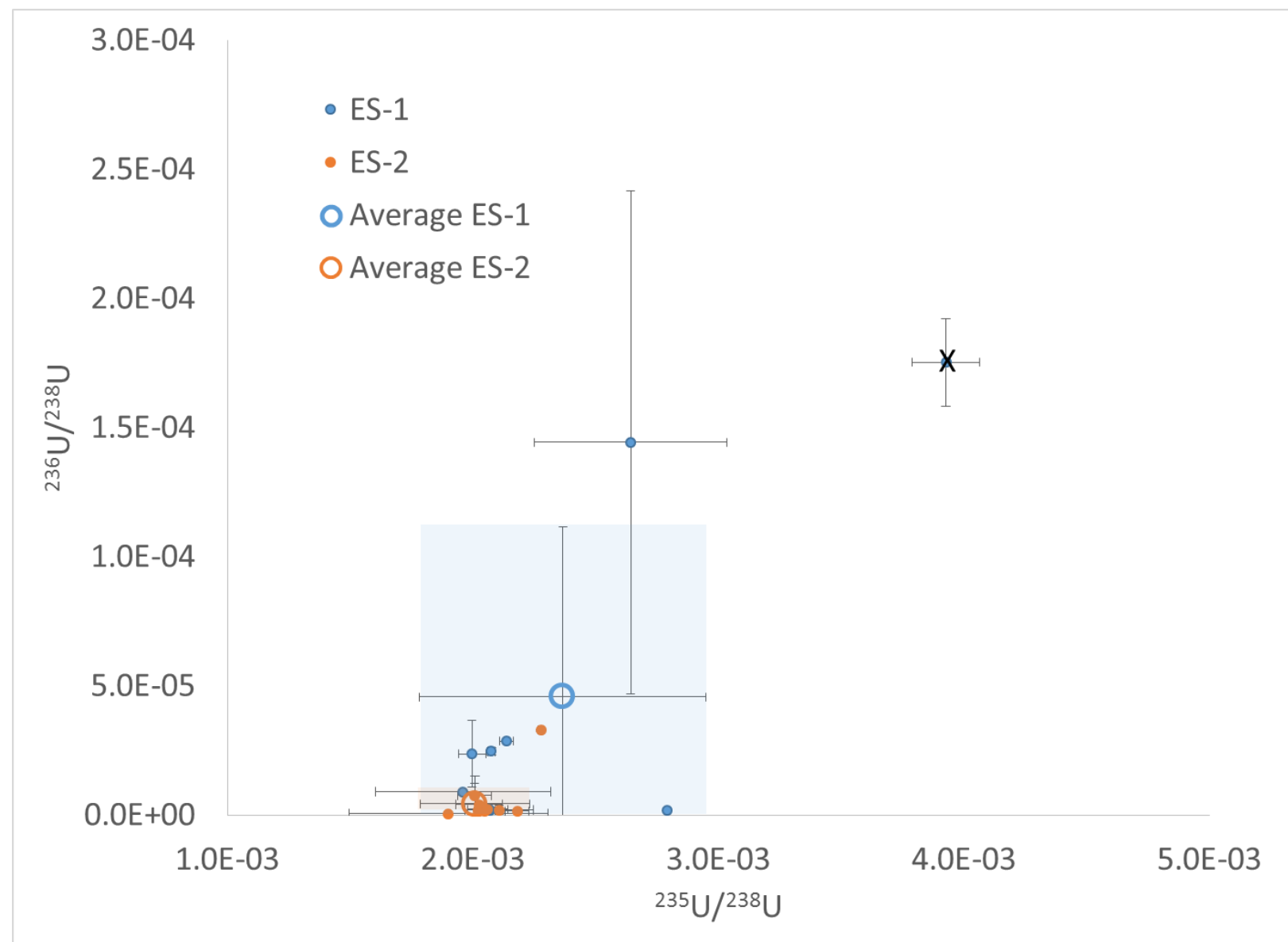
- 8 labs reported values for ES-1
- 15 labs reported values for ES-2
- Most results agree well with no significant difference between ES-1 and ES-2



$^{234}\text{U}/^{238}\text{U}$  vs  $^{235}\text{U}/^{238}\text{U}$  tri-plot for ES-1 and ES-2 at 1 week.

## Tri-plot of $^{235}\text{U}/^{238}\text{U}$ vs $^{236}\text{U}/^{238}\text{U}$

- 7 labs reported values for ES-1
- 10 labs reported values for ES-2
- Most results agree well with no significant difference between ES-1 and ES-2

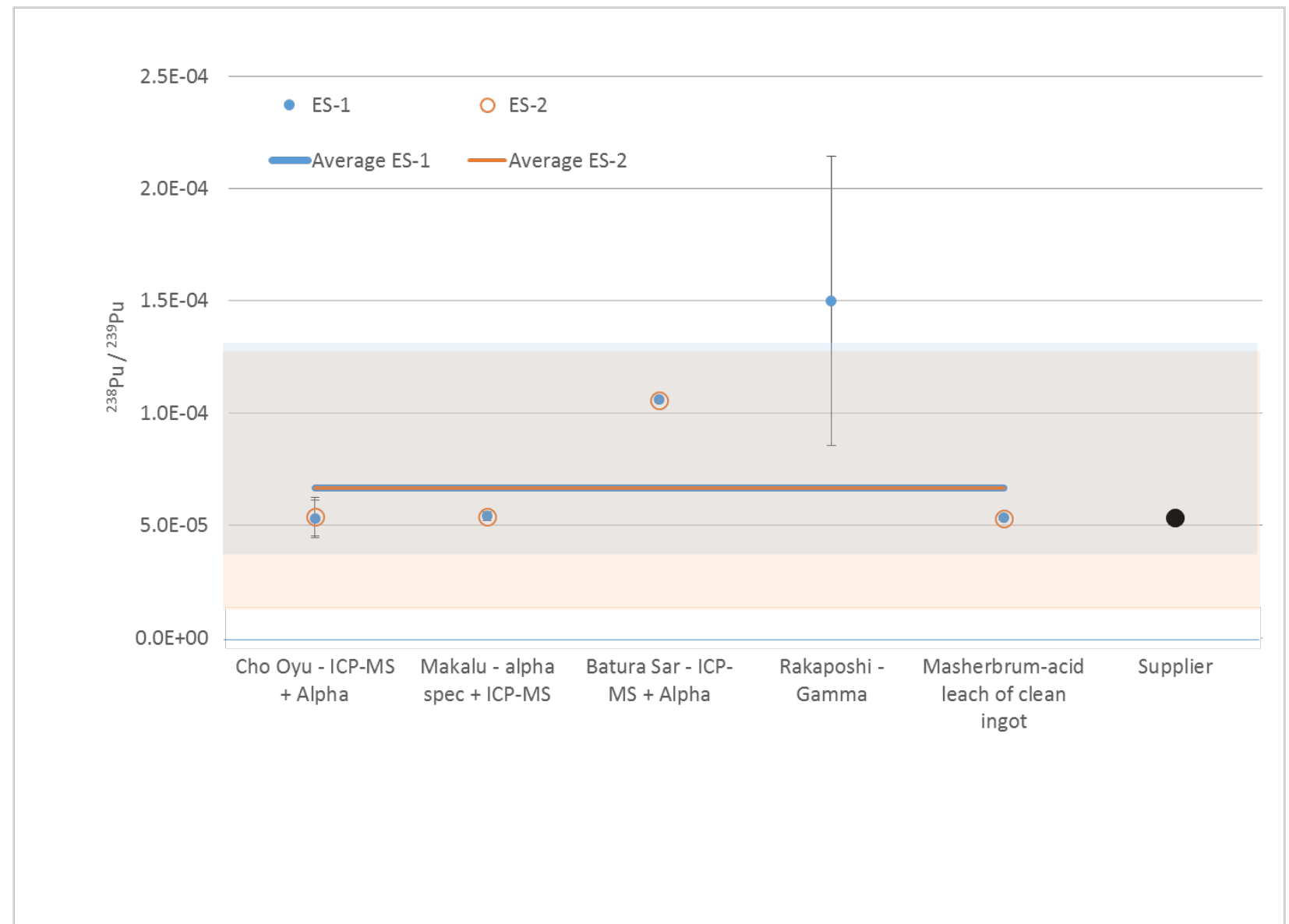


Tri-plot of  $^{235}\text{U}/^{238}\text{U}$  vs  $^{236}\text{U}/^{238}\text{U}$  for ES-1 and ES-2 at 2 months



## Isotopic Results – $^{238}\text{Pu}/^{239}\text{Pu}$

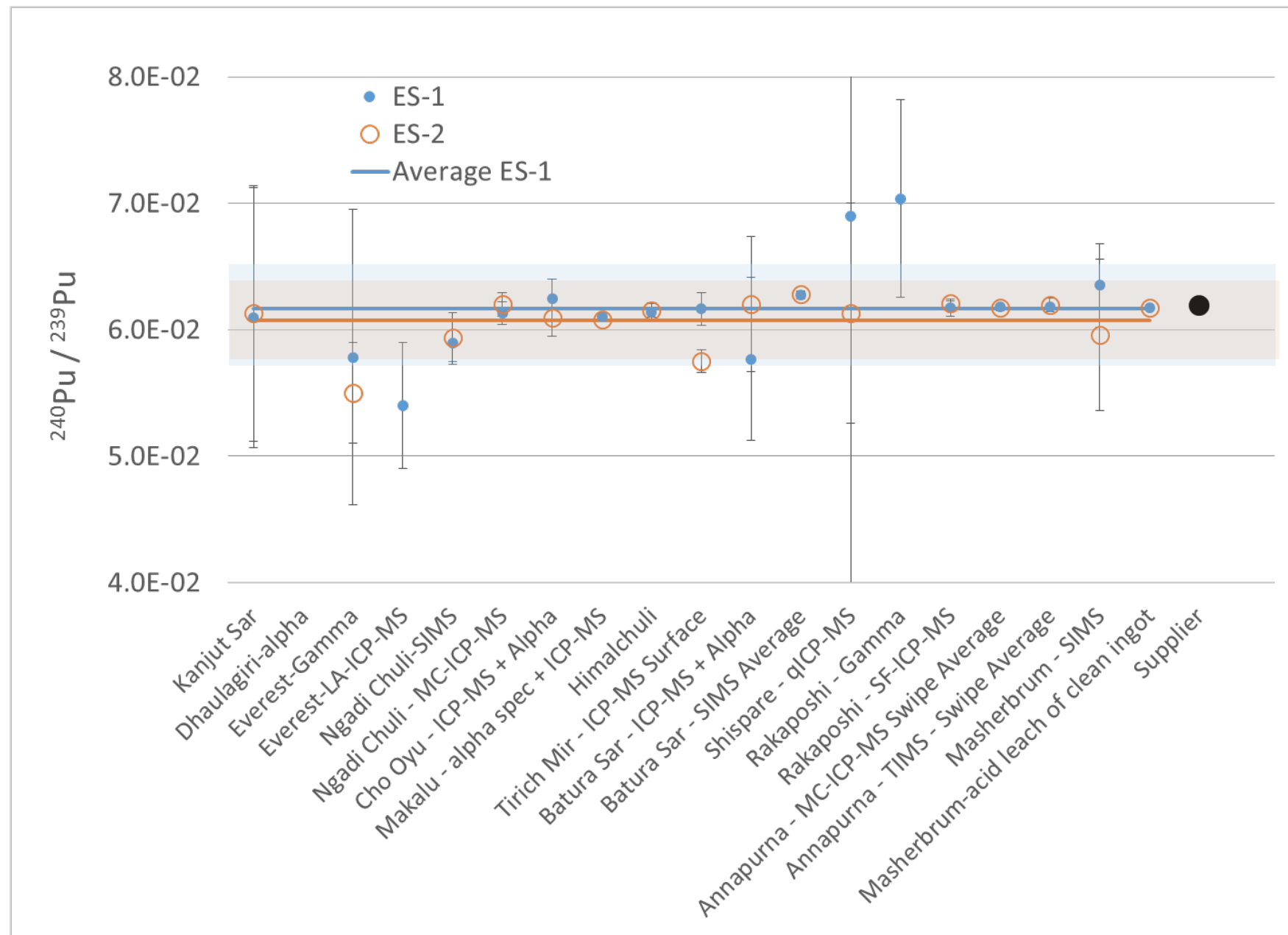
- 5 labs reported values for ES-1
- 4 labs reported values for ES-2
- Of the 4 labs reporting for both ES-1 & ES-2, all labs found both results with one another
- All measurements are consistent with supplier declarations



$^{238}\text{Pu}/^{239}\text{Pu}$  isotopic ratios in ES-1 and ES-2 at 2 months

## Isotopic Results – $^{240}\text{Pu}/^{239}\text{Pu}$

- 18 values reported for ES-1
- 16 values reported for ES-2
- Of the 16 labs reporting values for both ES-1 & ES-2, 15 labs found both to be consistent
- 90% of the analyses consistent with supplier declarations

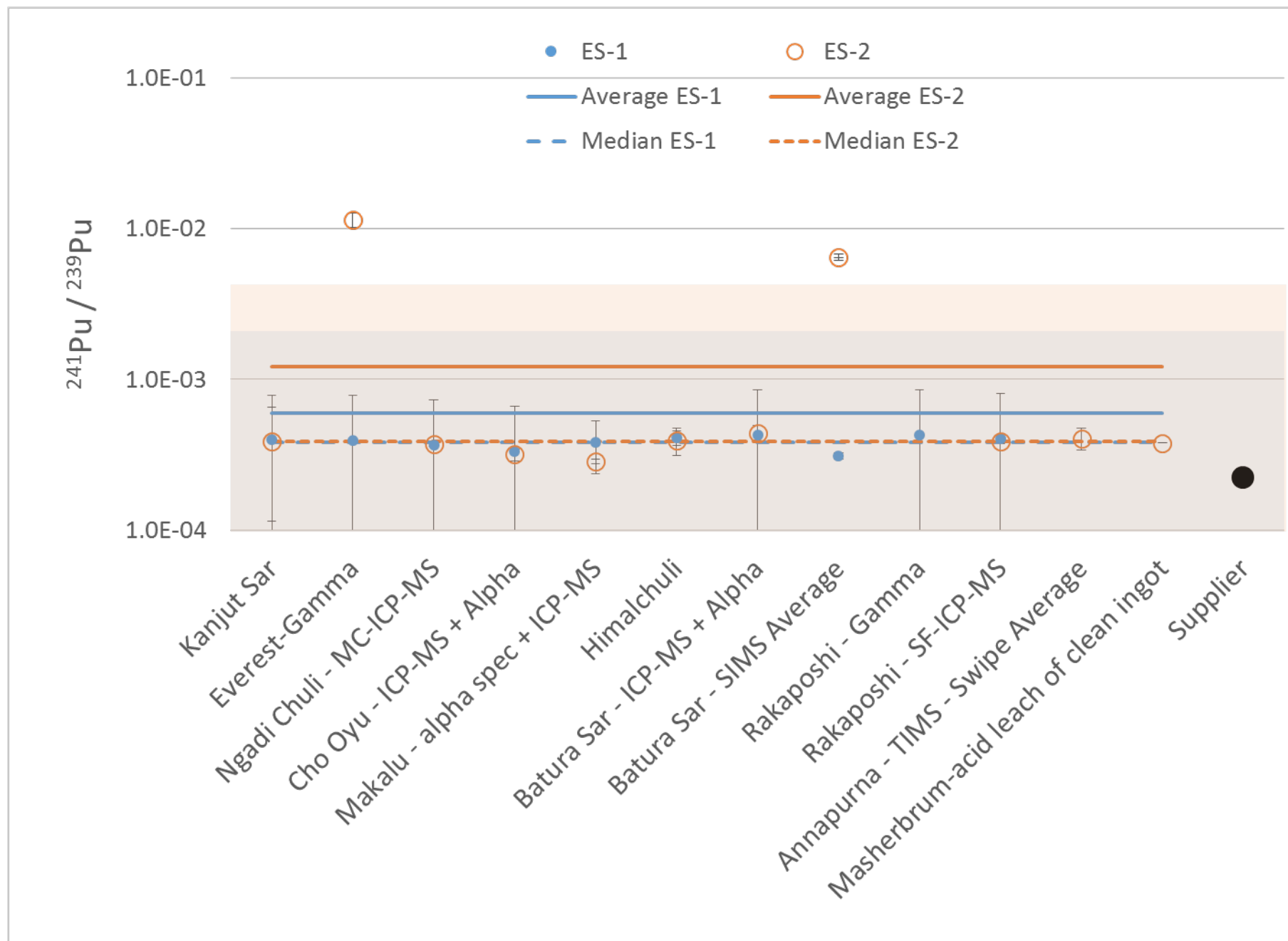


$^{240}\text{Pu}/^{239}\text{Pu}$  mass ratios for ES-1 and ES-2 at 2 months.



## Isotopic Results – $^{241}\text{Pu}/^{239}\text{Pu}$

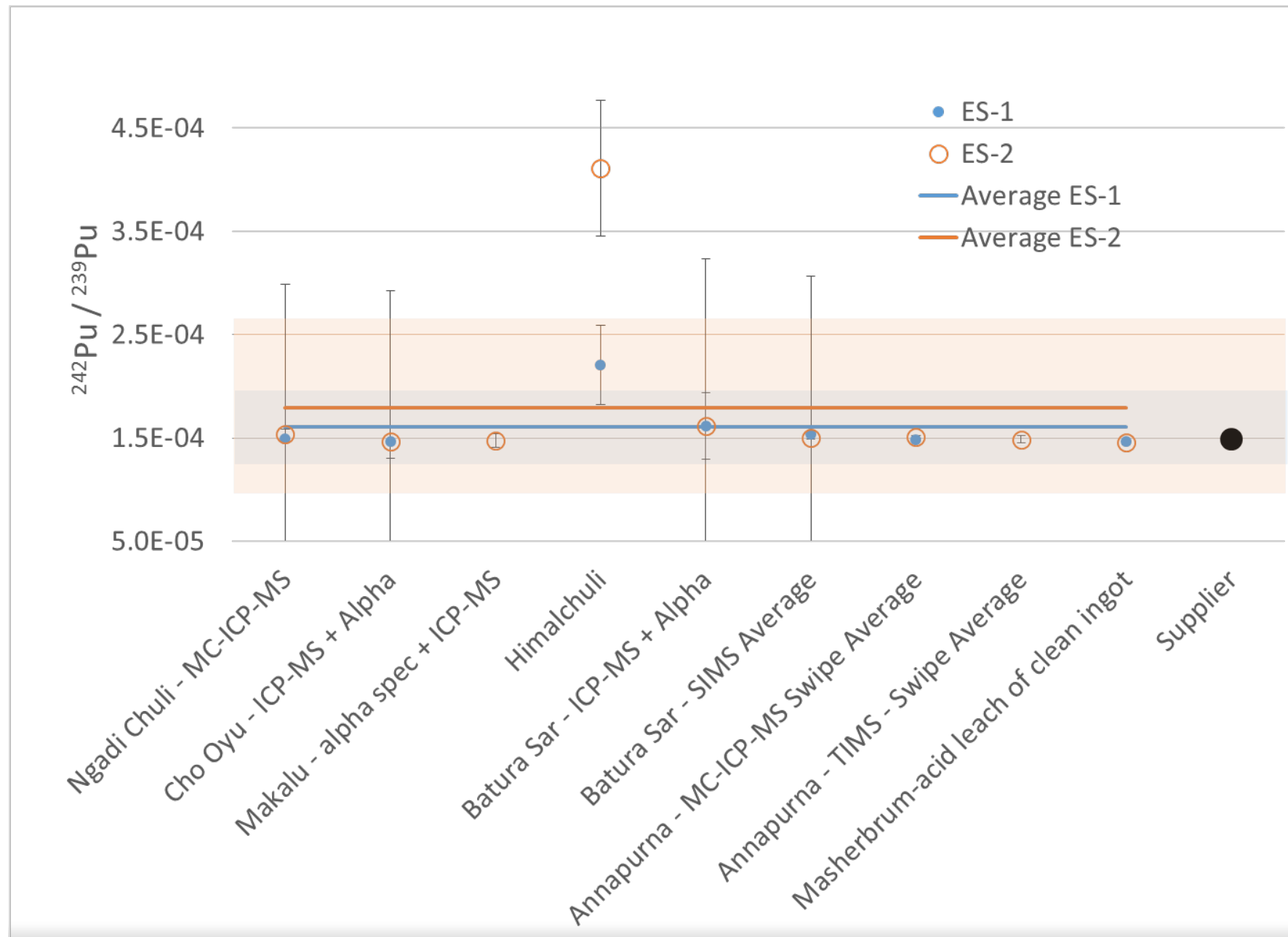
- 15 values reported for ES-1
- 13 values reported for ES-2
- All of the 13 labs reporting values for both ES-1 & ES-2, found the two samples to be consistent



$^{241}\text{Pu}/^{239}\text{Pu}$  mass ratios for ES-1 and ES-2 at 2 months. Shaded areas show standard deviation of participant values.

# Isotopic Results – $^{242}\text{Pu}/^{239}\text{Pu}$

- 8 values reported for ES-1
- 9 values reported for ES-2
- 6 of the 7 labs reporting values for both ES-1 & ES-2, found the two samples to be consistent

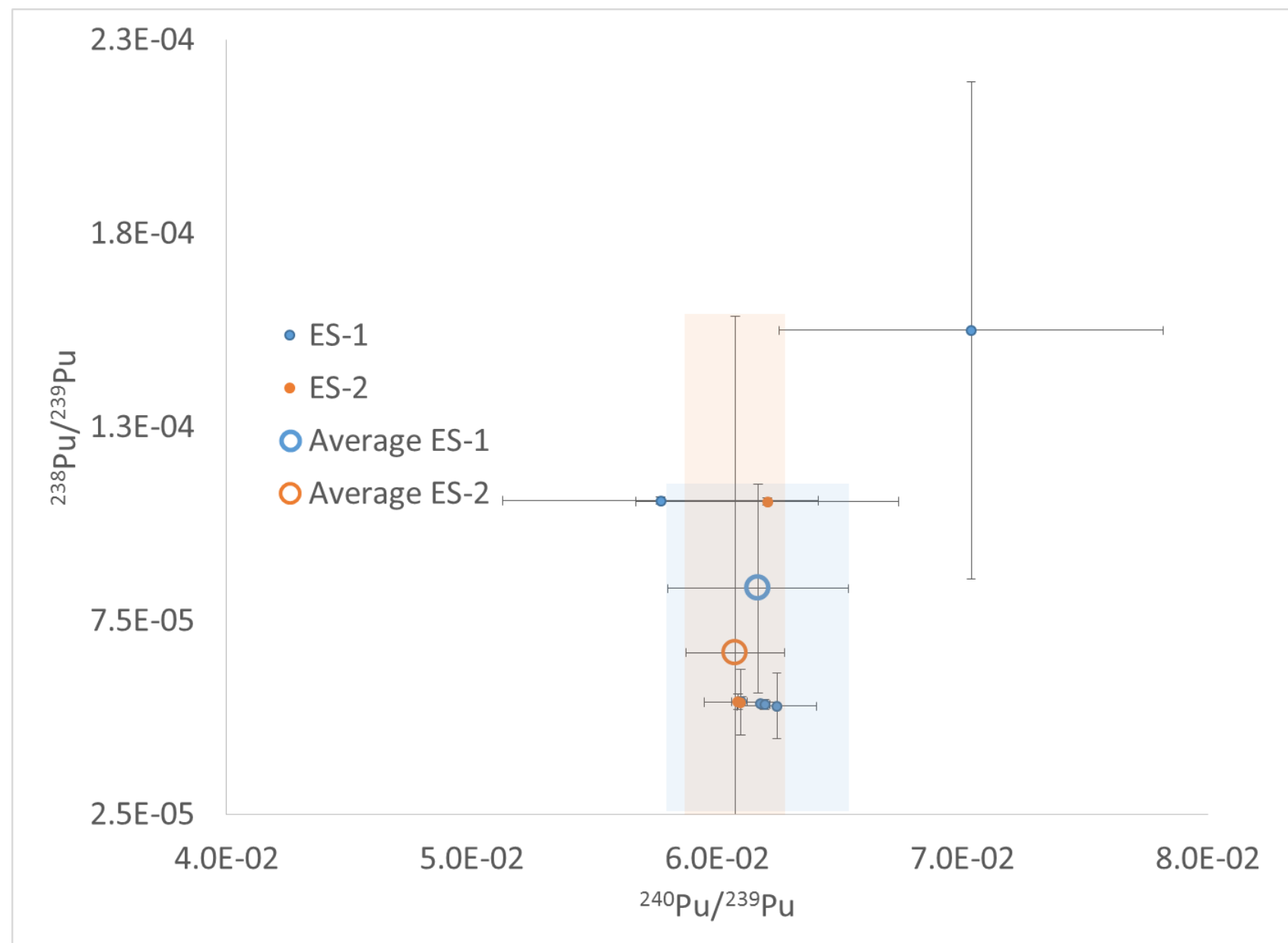


$^{242}\text{Pu}/^{239}\text{Pu}$  mass ratios for ES-1 and ES-2 at 2 months. Shaded areas show standard deviation of participant values.



## Isotopic Results – Tri-plot of $^{238}\text{Pu}/^{239}\text{Pu}$ vs $^{240}\text{Pu}/^{239}\text{Pu}$

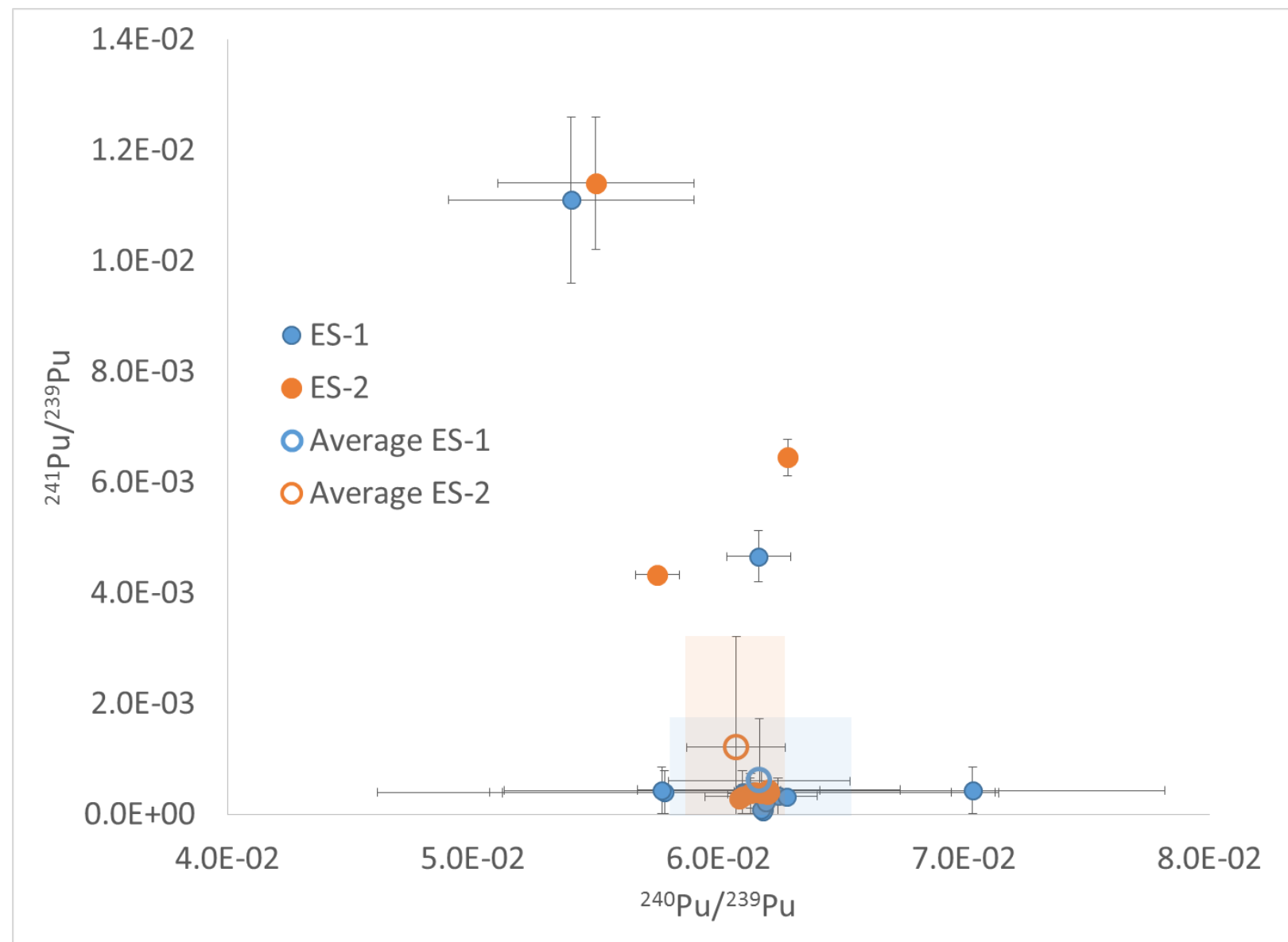
- 5 labs reported values for ES-1
- 4 labs reported values for ES-2
- Results suggest ES-1 and ES-2 are similar



$^{238}\text{Pu}/^{239}\text{Pu}$  vs  $^{240}\text{Pu}/^{239}\text{Pu}$  mass ratios for ES-1 and ES-2 at 2 months. Shaded areas show standard deviation of participant values.

## Isotopic Results – Tri-plot of $^{241}\text{Pu}/^{239}\text{Pu}$ vs $^{240}\text{Pu}/^{239}\text{Pu}$

- 6 labs reported values for ES-1
- 4 labs reported values for ES-2
- Results suggest ES-1 and ES-2 are similar

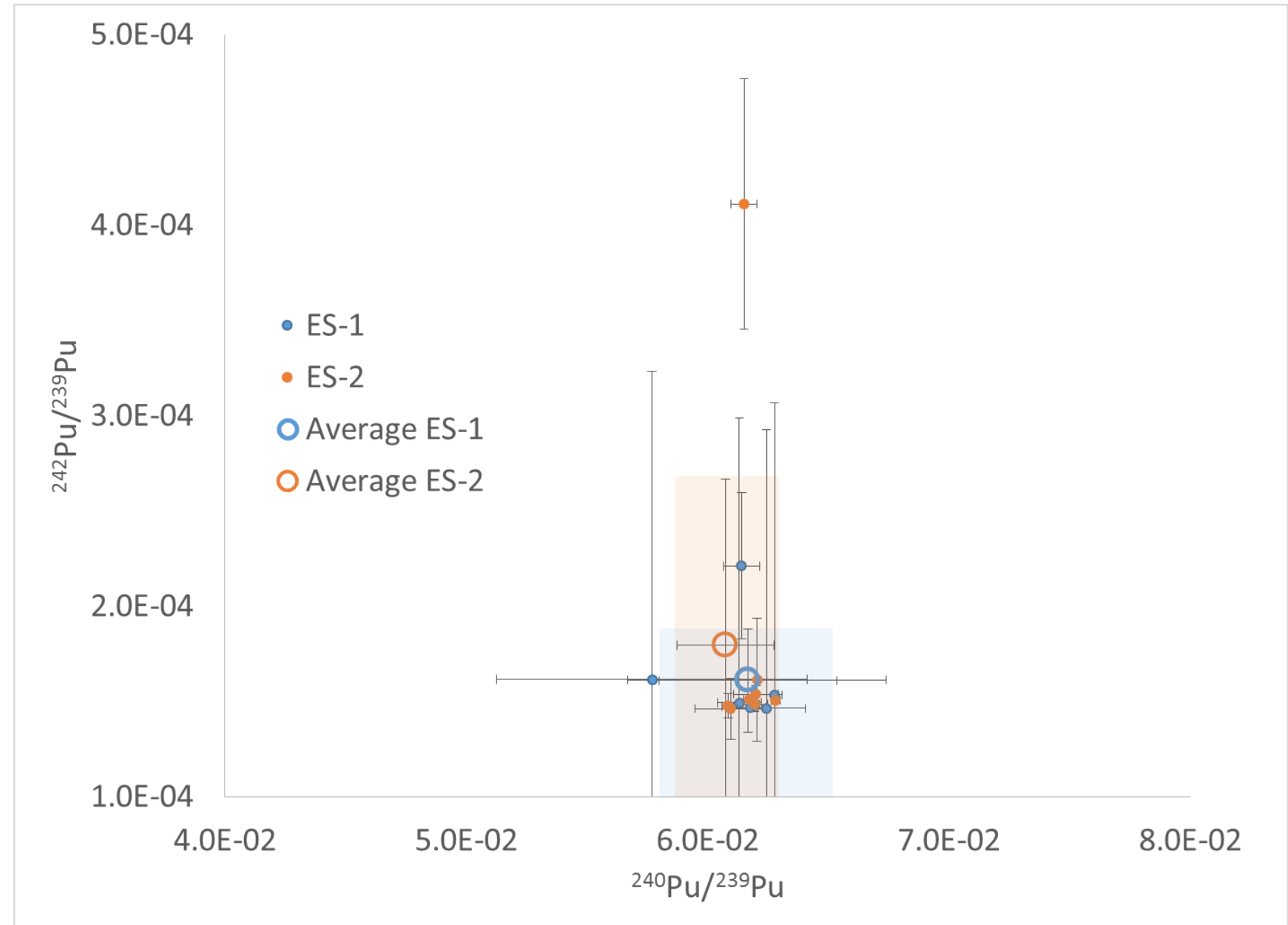


$^{241}\text{Pu}/^{239}\text{Pu}$  vs  $^{240}\text{Pu}/^{239}\text{Pu}$  mass ratios for ES-1 and ES-2 at 2 months. Shaded areas show standard deviation of participant values.



## Isotopic Results – Tri-plot of $^{242}\text{Pu}/^{239}\text{Pu}$ vs $^{240}\text{Pu}/^{239}\text{Pu}$

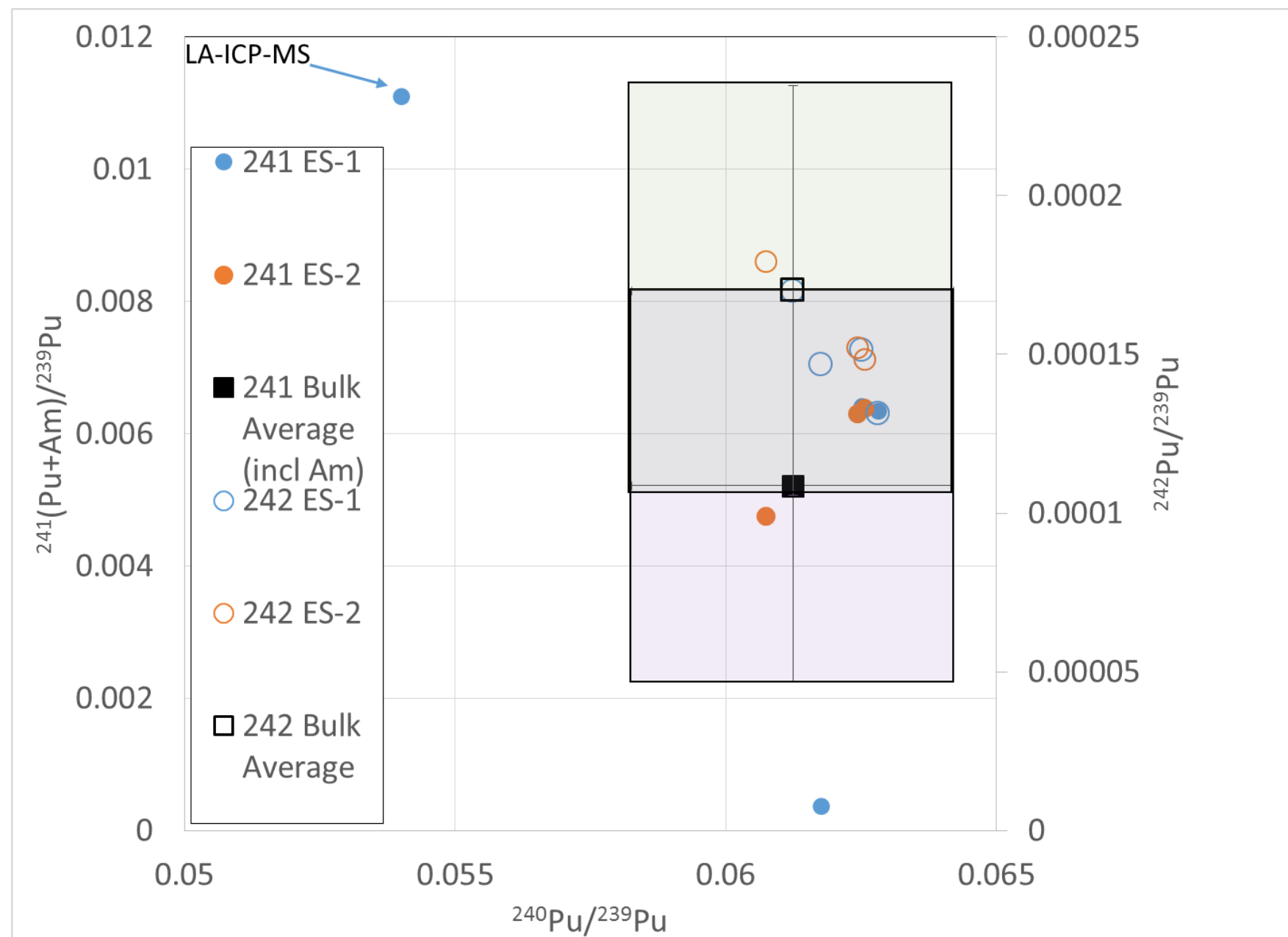
- 6 labs reported values for ES-1
- 7 labs reported values for ES-2
- Results suggest ES-1 and ES-2 are similar



$^{242}\text{Pu}/^{239}\text{Pu}$  vs  $^{240}\text{Pu}/^{239}\text{Pu}$  mass ratios for ES-1 and ES-2 at 2 months. Shaded areas show standard deviation of participant values.

## Particle Analysis – $^{241}\text{Pu}/^{239}\text{Pu}$ & $^{242}\text{Pu}/^{239}\text{Pu}$ vs $^{240}\text{Pu}/^{239}\text{Pu}$

- Gnadi Chuli, Masherbrum, and Batura Sar reported SIMS data for ES-1 and ES-2
- Everest reported LA-ICP-MS results
- Majority of data suggest ES-1 and ES-2 are consistent
- Most particle analysis consistent with bulk analysis

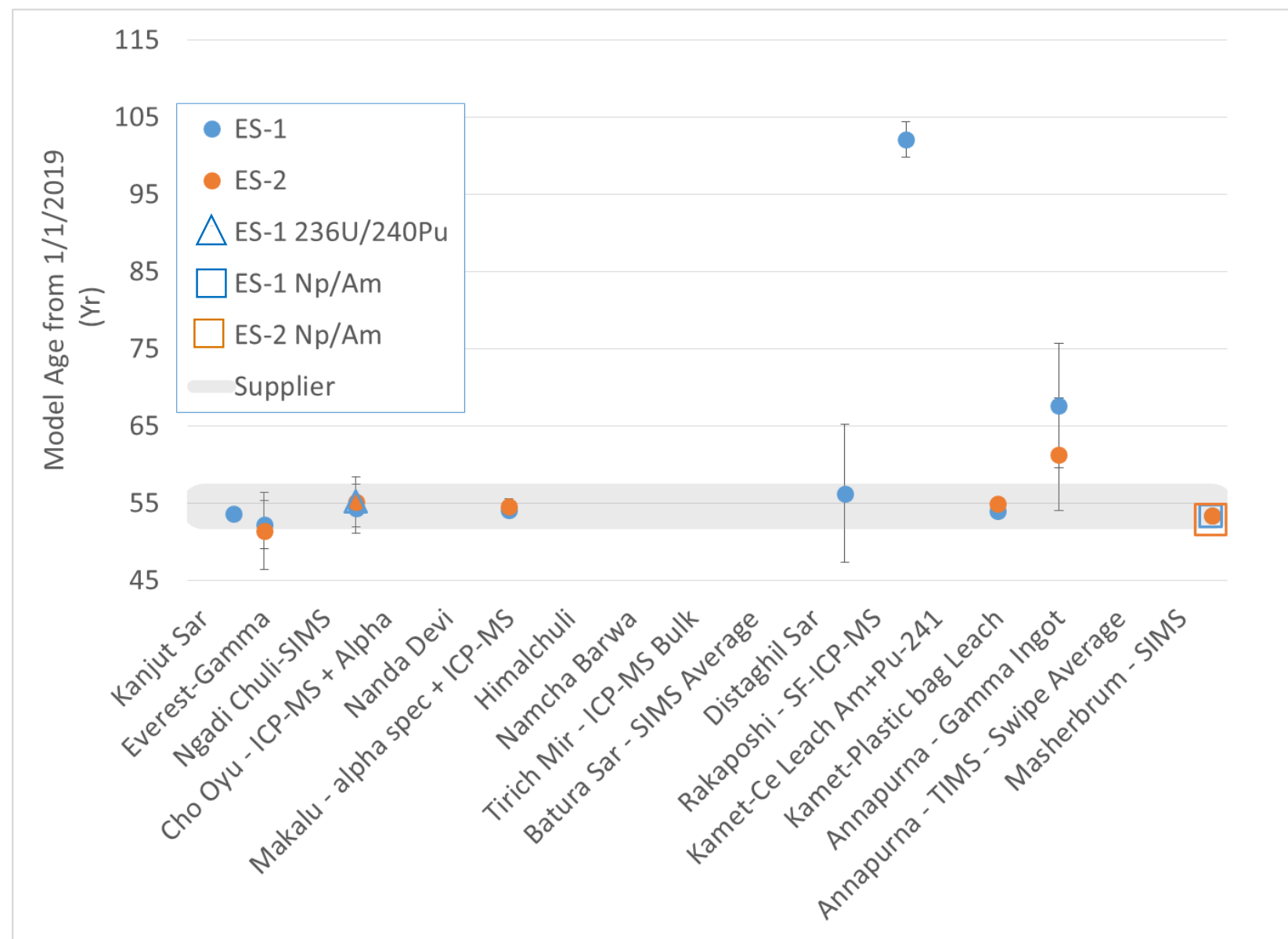


$^{242}\text{Pu}/^{239}\text{Pu}$  vs  $^{240}\text{Pu}/^{239}\text{Pu}$  mass ratios for ES-1 and ES-2 at 2 months. Shaded areas show standard deviation of participant values.



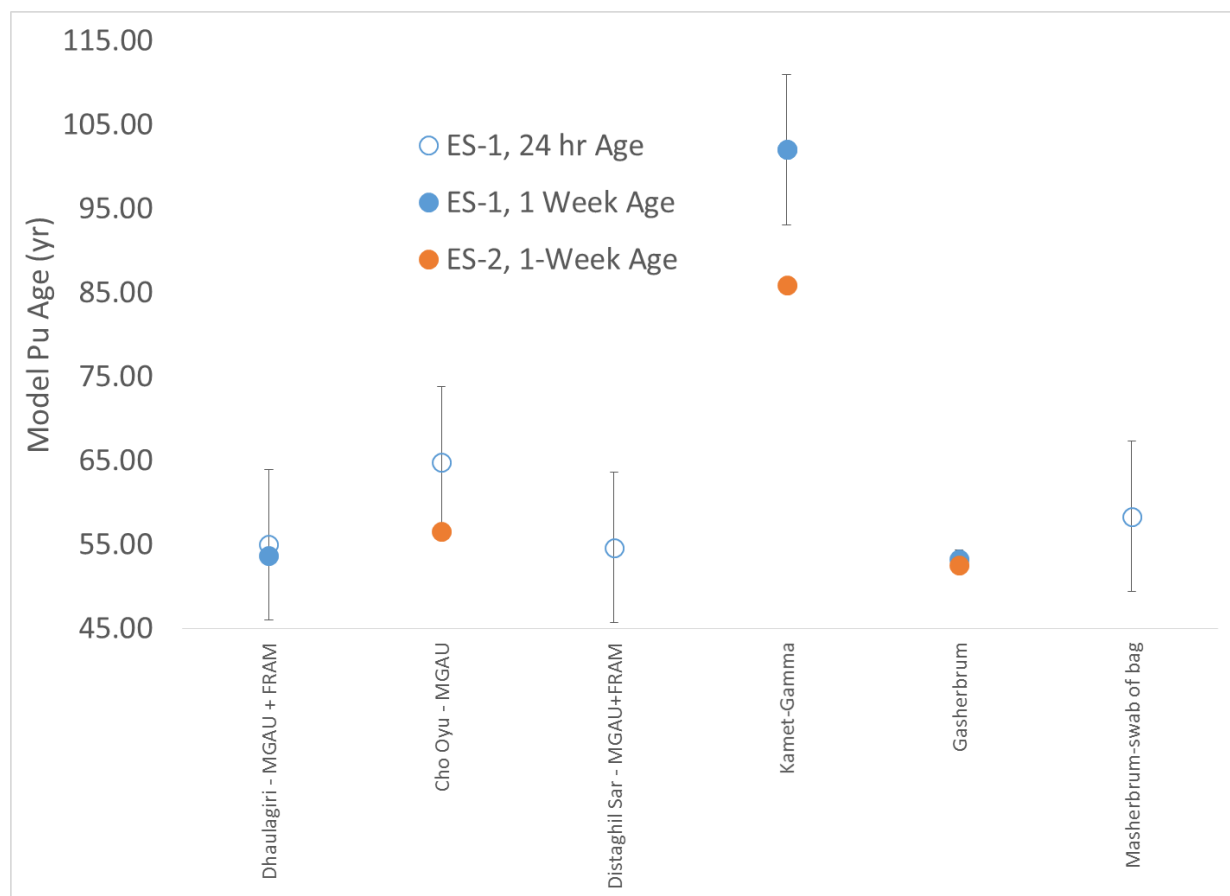
## Evaluation Results – Radiochronometry

- 9 labs reported values for ES-1
- 6 labs reported values for ES-2
- 6 out of 6 labs reporting for both ES-1 & ES-2 found ages to be consistent
- Cho Oyu and Masherbrum employed multiple chronometers effectively
- 15 out of 17 measurements consistent with supplier model age

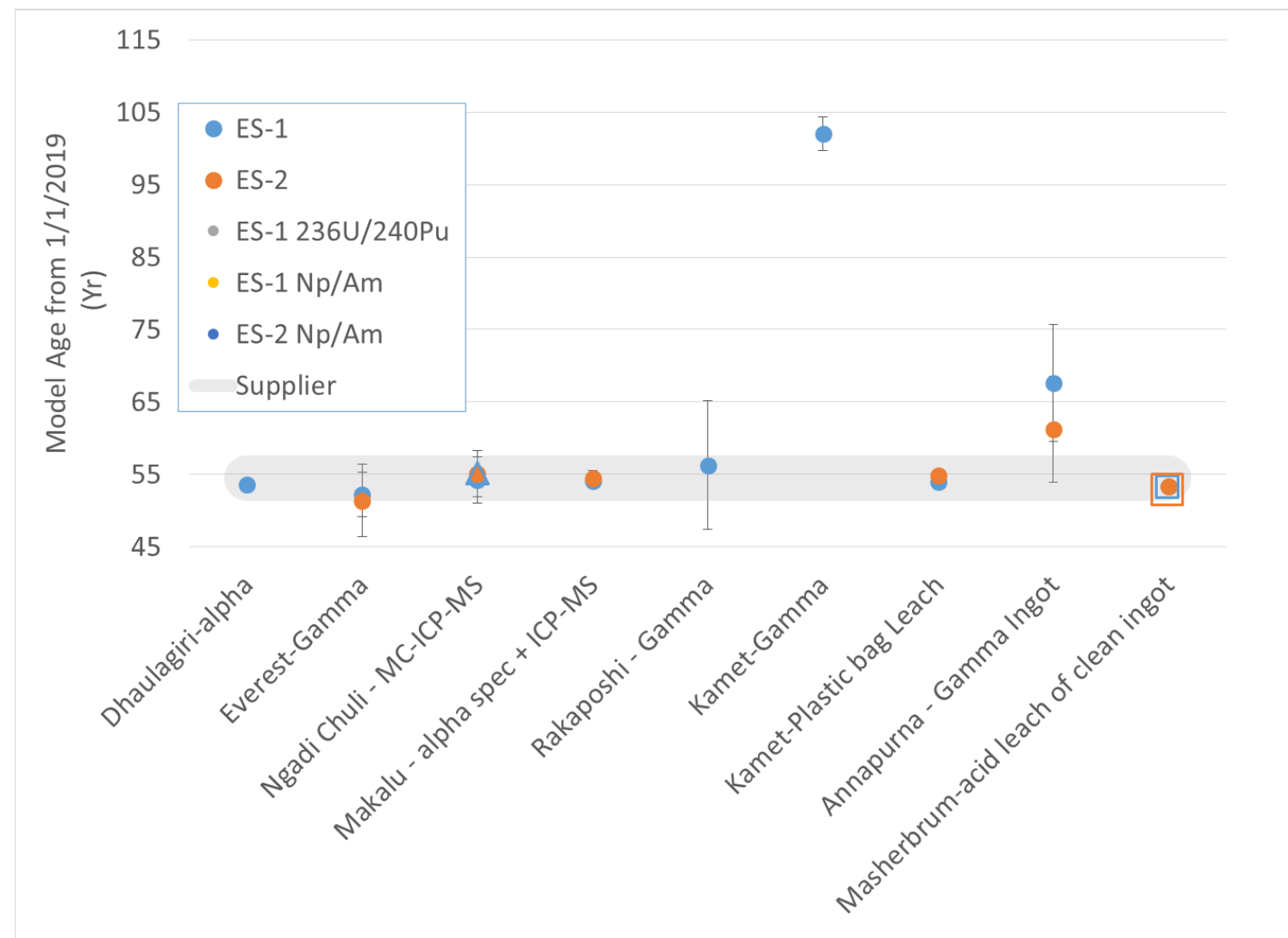


Model age of Pu derived from multiple chronometers

# Evolution of Radiochronometry from 24 hr report to 2 month report



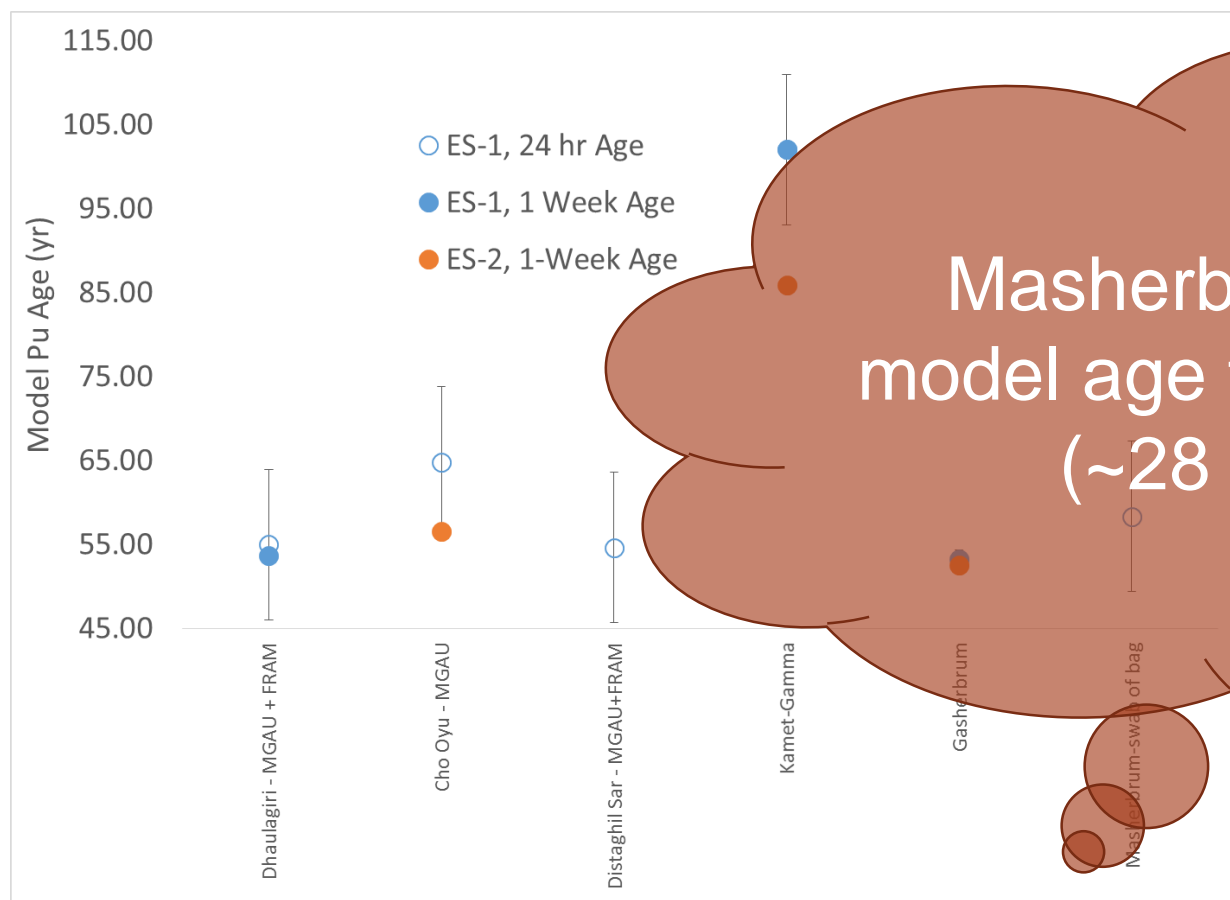
Model age of Pu at 24 hours and 1 week



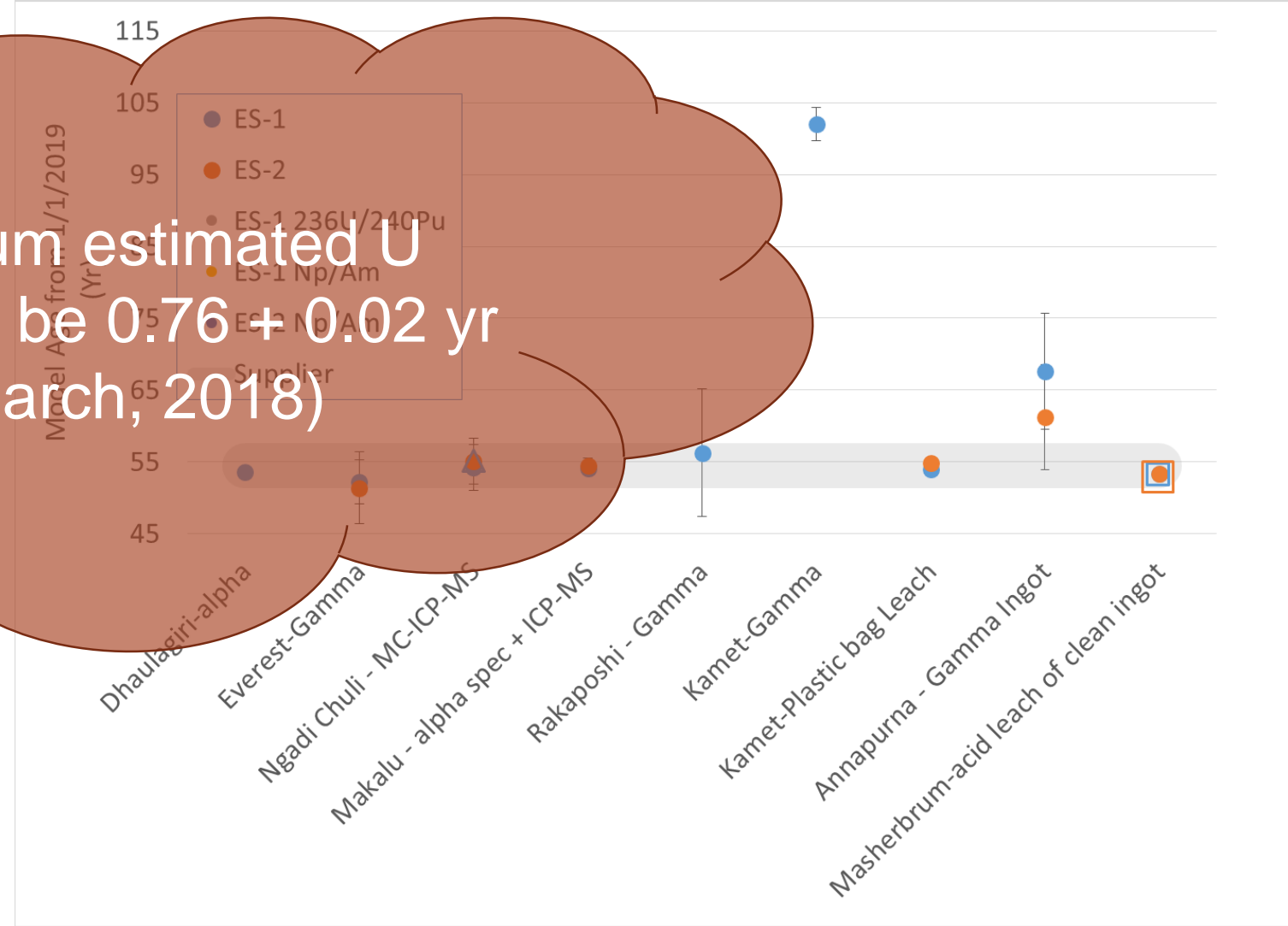
Model age of Pu at 2 months



# Evolution of Radiochronometry from 24 hr report to 2 month report



Masherbrum estimated U model age to be  $0.76 \pm 0.02$  yr (~28 March, 2018)



Model age of Pu at 24 hours and 1 week

Model age of Pu at 2 months

# Discussion – 2 Month

## Small Group Discussion

Group 1: Romania, Russia, Ukraine, Azerbaijan

Group 2: Japan, Korea, Singapore, Australia, USA

Group 3: Switzerland, Poland, Sweden, Germany, UK

Group 4: Hungary, Israel, JRC, Canada, France

Questions to discuss (spend 10 minutes per question):

- 1) What were the three most useful measurement techniques used during CMX-6?
- 2) Are there any analytical results that you believe provide insight into the process history of the materials or the intent by any person of interest to authorities?
- 3) List three aspects of the exercise that you liked.
- 4) List three aspects of the exercise that you would change.
- 5) Please provide any additional comments/suggestions you have about the Data Review Meeting.
- 6) Regarding future exercises:
  - What would you like the scope to include (e.g. linking with the Libraries Task Group and Galaxy Serpent, linking with the Evidence Task Group?, etc.)
  - What materials would you like to see be used in future exercises?



# CMX-6 Full Design, Development, Scenario & Backstory

Changes to RNL retirement plan go into effect

Plot includes pilfering lab supplies and materials, including  $CeF_4$ ,  $UF_4$ , and  $PuF_4$ , over a long period of time to avoid detection.  $PuF_4$  from sealed source generated in July, 1964.

Cutting contaminated U and Ce metal rings into pieces for disposal at Corben Foundry

Discovery of contaminated Ce (ES-1) and U (ES-2) at Greene's Recycling and Corben Foundry

Disgruntled employee, Dr. Evanovich, plots a plan to generate Pu metal from bomb reduction of  $PuF_4$  and sell on black market

1st test run of metal reduction process using  $CeF_4$  successful

2nd test run of metal reduction process using  $UF_4$  successful

2016

2017

2018

2019



Dr. Evanovich (pictured here before becoming disgruntled!)

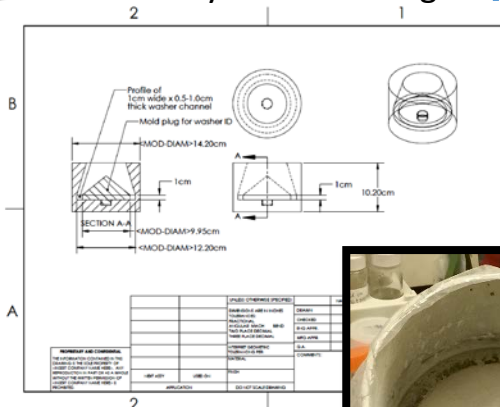


RNL  $PuF_4$  neutron source

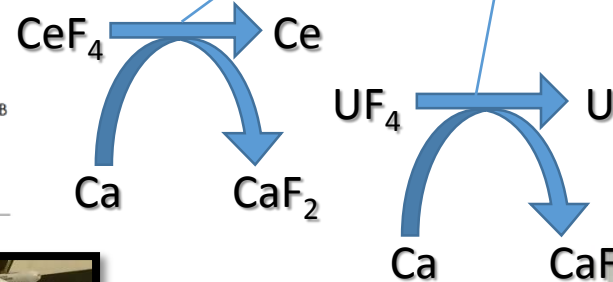


Dr. Evanovich's makeshift glovebag for metal casting operations

Preliminary crucible design



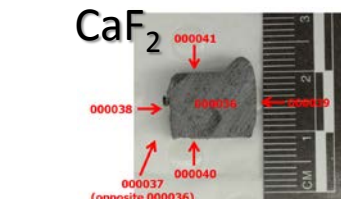
Crucible used to cast Ce and U



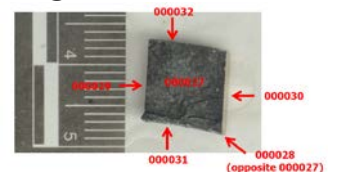
U metal ring



ES-1



ES-2





# Exercise Design

## Parameters:

- New material (Pu)
- Exempted quantity for ease of shipping ( $<200 \mu\text{g}$ )
- A scenario that makes sense!

## Features:


- Difficult to ID with HPGe (U masking)
- Incorporate TE that is separable from radioactivity
  - Patent print
  - Latent print
  - Tool marks
  - Trace elements
  - Contaminants from cutting, casting
- Isotopics defining feature
- Age defining feature
- Chemical form defining feature
- Shape defining feature
- Possible detection of  $^{22}\text{Na}$

Envisioned Scenario

- 1) Disassembled PNNL Employee
  - Stole PuF<sub>4</sub>
  - UF<sub>4</sub>
  - CeF<sub>3</sub>
- 2) Set up Bomb red in Garage
  - Makeshift Glovebox
  - Storing material in there
- 3) CeF<sub>3</sub> Bomb Reduction
- 4) UF<sub>4</sub> Bomb Reduction
- 5) Cast Ce
- 6) Cast U
- 7) Cut Metals for Disposal as Metal & Waste
  - Placed small amounts in waste pipe + disposed of

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8) Pu




Expected Material

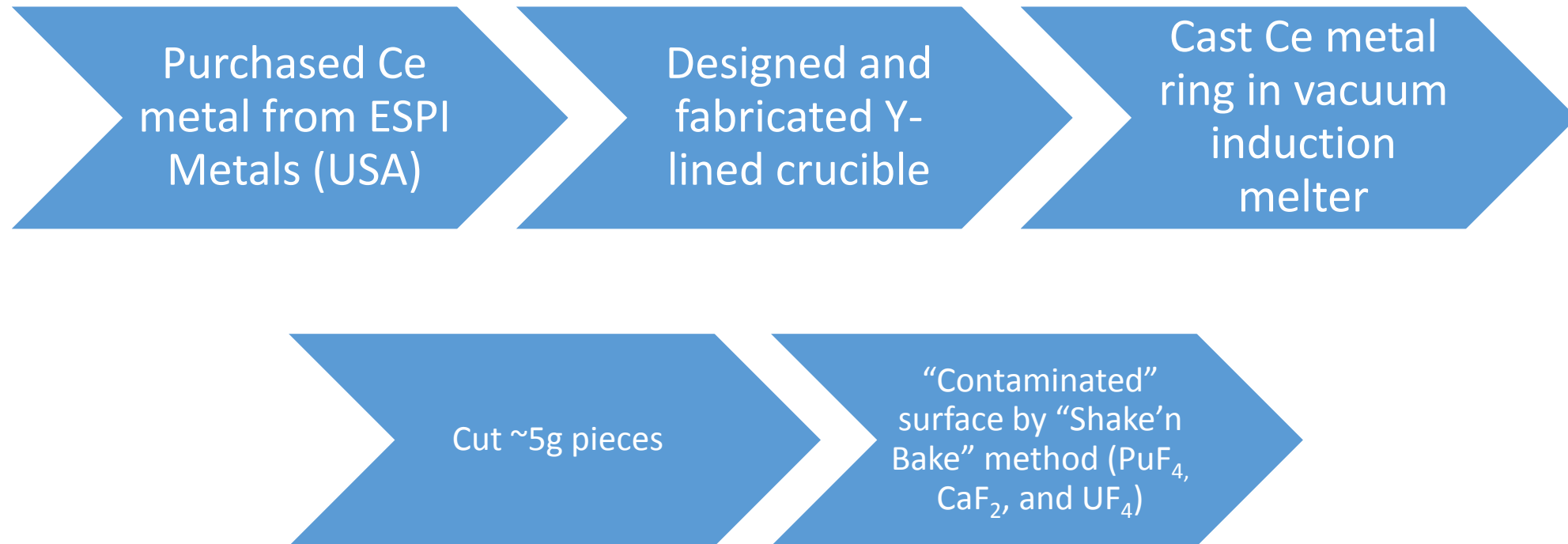
- 1) Pipe
  - same pipe
  - same cut surfaces
- 2) FP 2X
  - contain, separable
  - Trace contain from
- 3) Ce
  - Ce-Mold
  - U-Mold
  - Ce-Cutting
  - U-Cutting
- 4) U
- 5) PuF<sub>4</sub>
  - contain
- 6) CeF<sub>3</sub>
- 7) UF<sub>4</sub>

u!

- 1) Detect at local Recycle
  - List of 6 major industries as source
- 2) Search turns up Sample #2 Ce



# CMX-6 Exercise Sample 1 Production





# First attempt at casting Ce ring



Graphite mold with ytttria wash post cast



Casting surface showing oxidation and cavities



Casting retrieval

# Final version of Ce cast



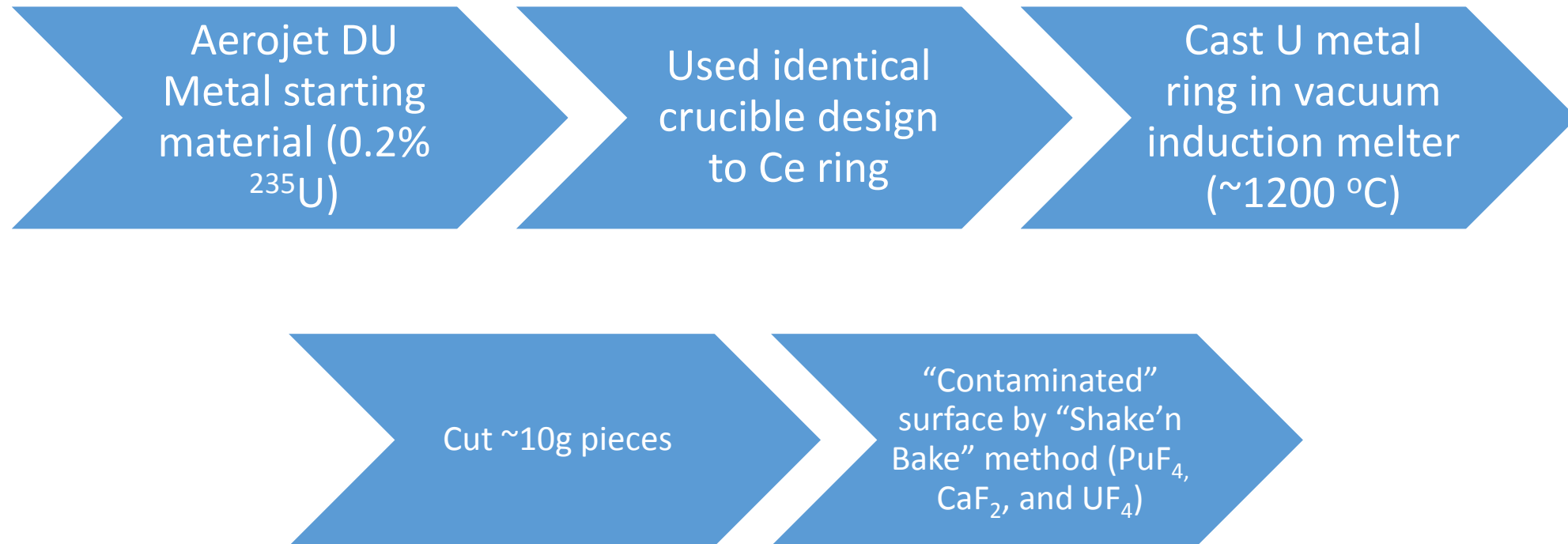
Degassed Ce metal



Optimized casting



# CMX-6 Exercise Sample 2 Production



# Uranium Metal Casting: Casting Retrieval in Fume Hood



Top down view of casting in mold post cast



Base of broken graphite plug showing base of fused U ring



Top of casting



Base of casting



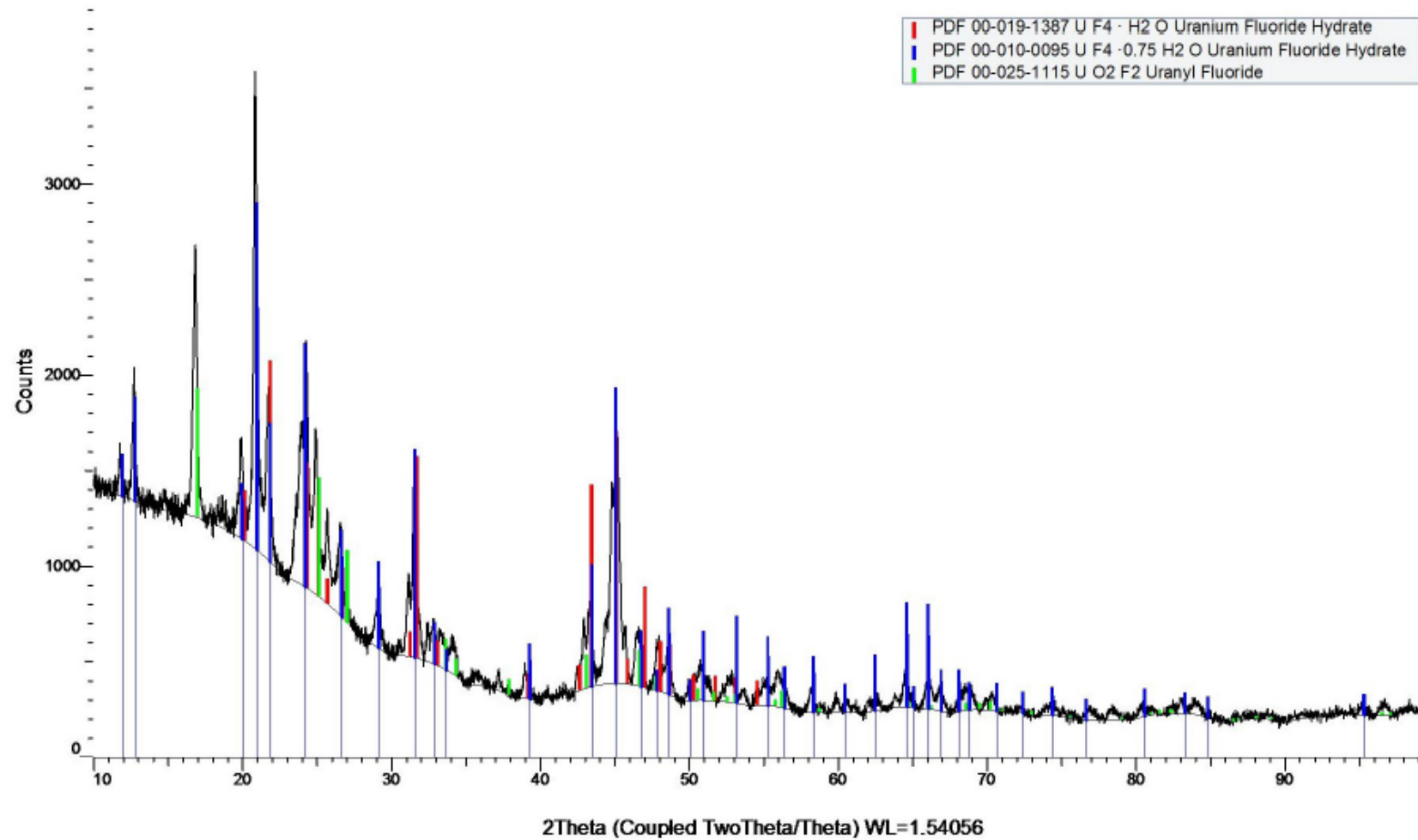
# Uranium Metal Sectioning



Glovebox saw used to section U

# UF<sub>4</sub> Starting Material for Bomb Reduction to U Metal

(Coupled TwoTheta/Theta)



X-ray diffraction pattern of uranium fluoride and related species



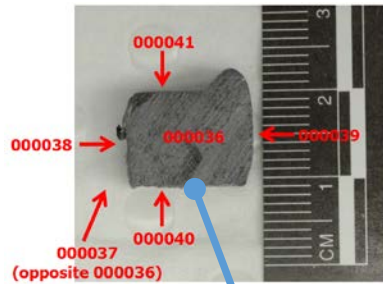
# Traditional Forensic Evidence



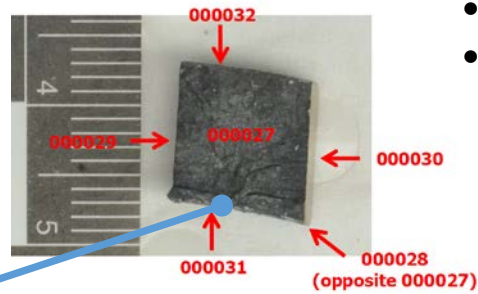
Conclusive Similar

- Composition
- Dimensions / Manufacturing characteristics
- Tool marks

ES-1



ES-2



Conclusive Dissimilar

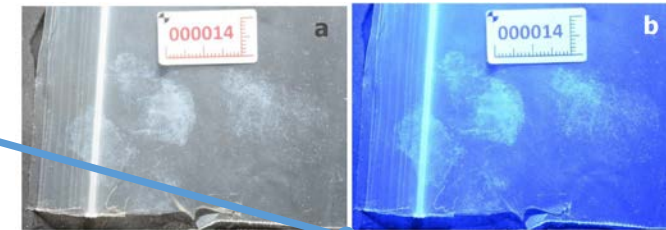
- Composition
- Conclusive Similar
- Tool marks
- Trace evidence
- Dimensions



Conclusive Similar

- Fingerprints

ES2 Latent Prints



ES1 Patent Prints

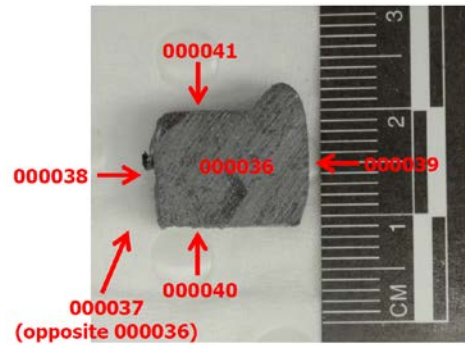


# Nuclear Forensic Evidence

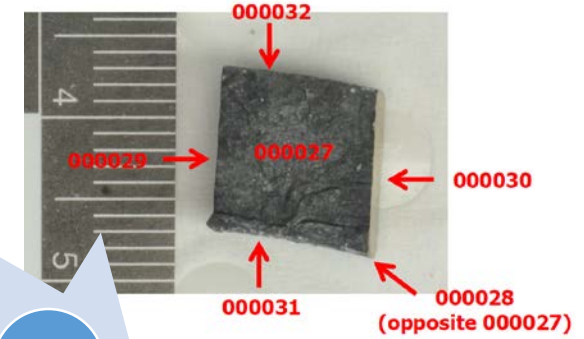


ES-1

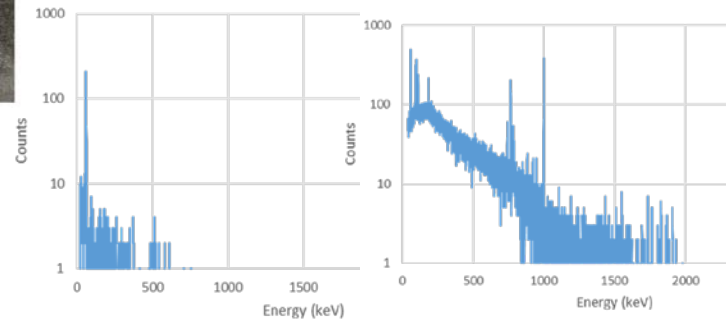
ES-2



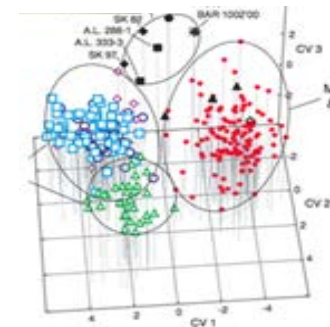
ES-3



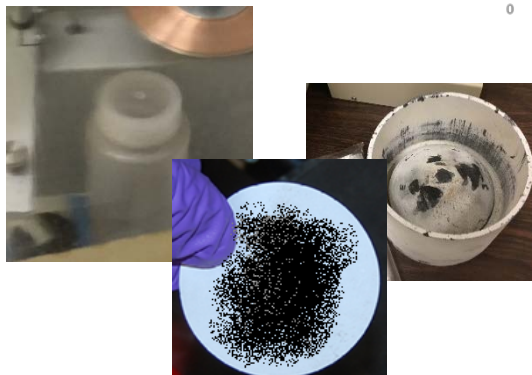
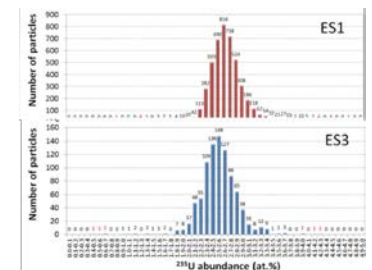
- 24 Hour Report
- Gamma Spectrometry
  - Inconclusive
  - $^{22}\text{Na}$  detection



- 1 Week Report
- Bulk Isotopics
  - Suggestive Similar
  - Morphology of Pu particles



- 2-Month Report
- Radiochronometry
  - Composition
  - Particle Analysis
  - Conclusive Similar





# Uranium Pedigree

Sample ID	Atomic Percent						Amount (g)	Date Measured	Chemical Form		
	<sup>234</sup> U	Uncertainty (2-σ)	<sup>235</sup> U	Uncertainty (2-σ)	<sup>236</sup> U	Uncertainty (2-σ)				<sup>238</sup> U	Uncertainty (2-σ)
RNL-543S	8.500E-01	1.500E-01	8.670E+01	1.500E+00	5.700E-01	8.000E-02	1.190E+01	9.000E-01	1.003E+03	18-Sep-13	Oxide
RNL-789N	1.050E+00	7.000E-02	8.940E+01	1.800E+00	6.900E-01	5.000E-02	8.900E+00	2.000E-01	2.560E+02	2-Jun-06	Oxide
RNL-560G	1.930E-02	2.702E-04	2.245E+00	1.700E-01	2.000E-03	5.000E-04	9.703E+01	8.000E-02	2.412E+03	14-Nov-09	Fluoride
RNL-495W	9.291E-01	6.000E-03	78.965	3.600E+00	3.878E-01	1.900E-03	1.972E+01	6.400E-02	1.452E+03	1-Jan-14	Oxide
RNL-118L	8.640E-01	6.600E-03	6.712E+01	3.800E+00	4.300E-01	3.200E-03	3.159E+01	6.900E-02	4.520E+02	4-Jun-08	Oxide
RNL-992K	bd	bd	2.400E-01	6.000E-02	bd	bd	9.975E+01	6.000E-02	1.438E+03	13-Jun-18	Fluoride
RNL-733Y	9.093E-01	4.000E-03	7.778E+01	3.100E-02	4.600E-02	1.200E-03	2.127E+01	6.400E-02	1.289E+03	4-Aug-11	Metal
RNL-629F	2.430E-02	3.200E-03	2.893E+00	9.300E-02	3.000E-04	5.000E-04	9.708E+01	5.000E-02	1.299E+03	14-Nov-14	Nitrate
RNL-373M	bd	bd	1.900E-01	6.000E-02	bd	bd	9.980E+01	6.000E-02	3.132E+03	8-Apr-11	Oxide

- <sup>235</sup>U and <sup>238</sup>U consistent with two possibilities in RNL holdings
- Form (UF<sub>4</sub>) and amount most consistent with RNL-992K

# Pu Pedigree

Sample ID	Atomic Percent						Amount (g)	Date Measured	Chemical Form	Last Processing Date
	<sup>239</sup> Pu	Uncertainty (2-σ)	<sup>240</sup> Pu	Uncertainty (2-σ)	<sup>241</sup> Pu	Uncertainty (2-σ)				
<del>RNL-132D</del>	<del>9.153E+01</del>	<del>4.595E+00</del>	<del>8.210E+00</del>	<del>9.832E-01</del>	<del>3.760E-02</del>	<del>4.920E-03</del>	<del>6.450E+02</del>	<del>3-Mar-11</del>	<del>Metal</del>	<del>Jan-82</del>
<del>RNL-235A</del>	<del>8.120E+01</del>	<del>4.060E+00</del>	<del>1.870E+01</del>	<del>9.348E-01</del>	<del>1.050E-01</del>	<del>5.250E-03</del>	<del>9.230E+02</del>	<del>18-Dec-08</del>	<del>Oxide</del>	<del>Apr-71</del>
RNL-194L	9.340E+01	3.736E+00	6.567E+00	8.340E-01	4.000E-02	5.200E-03	1.400E+02	26-Feb-15	Fluoride	Jul-64
<del>RNL-651H</del>	<del>9.341E+01</del>	<del>4.222E+00</del>	<del>6.698E+00</del>	<del>8.770E-01</del>	<del>4.700E-02</del>	<del>6.300E-03</del>	<del>3.540E+02</del>	<del>27-Nov-14</del>	<del>Oxide</del>	<del>&lt;1966</del>

- <sup>239</sup>Pu inconsistent with RNL-235A
- <sup>240</sup>Pu and age inconsistent with RNL-132D
- Form (PuF<sub>4</sub>) most consistent with RNL-194L



# Summary

- CMX-6 largest exercise in ITWG history  
...also the most complex!
- Essentially every major design feature introduced as evidence in CMX-6 samples were identified by at least one laboratory
- Tool marks
- Fingerprints
- Cutting tools
- Bag composition
- Pipe composition
- Contaminants – Ca, F, U, Pu
- Pu age
- U age
- F composition of Pu and U contaminants