

# Multi-element Reference Materials for XRF (and Filter Orientation)

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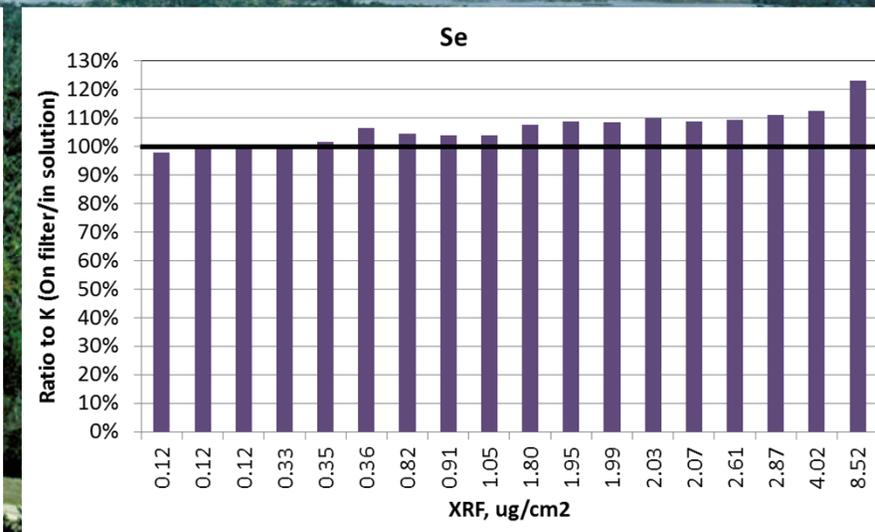
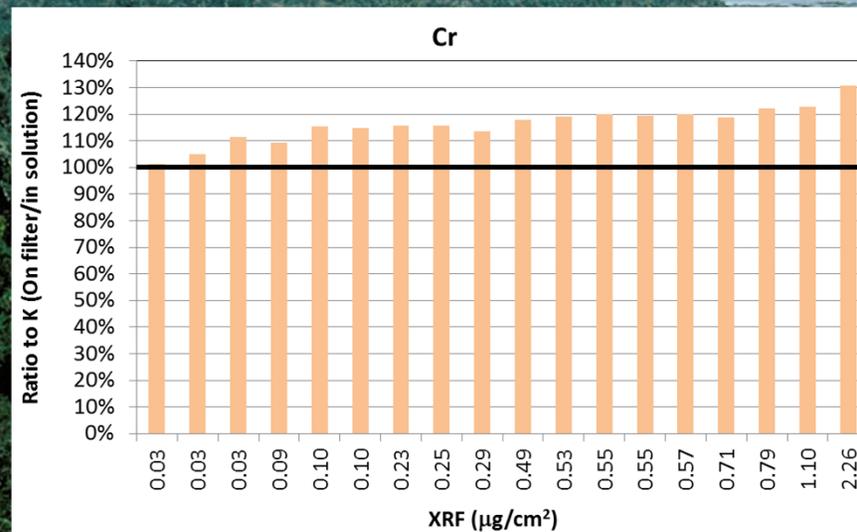
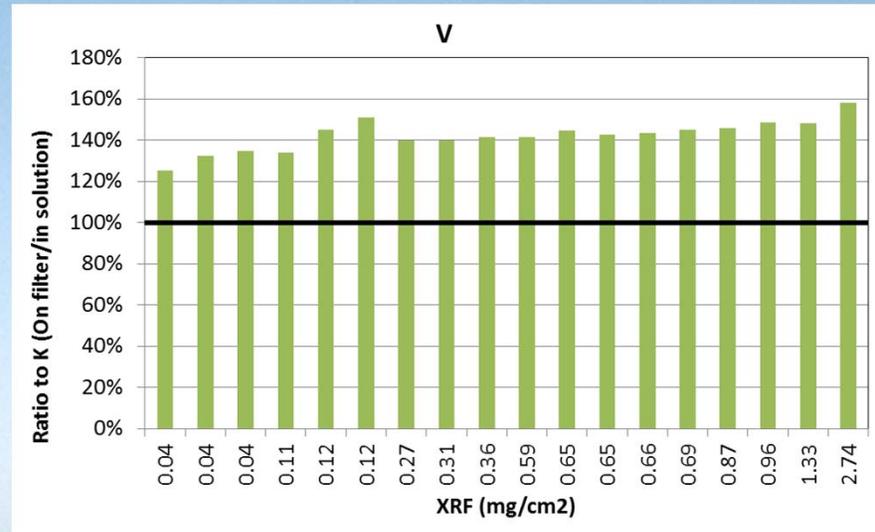
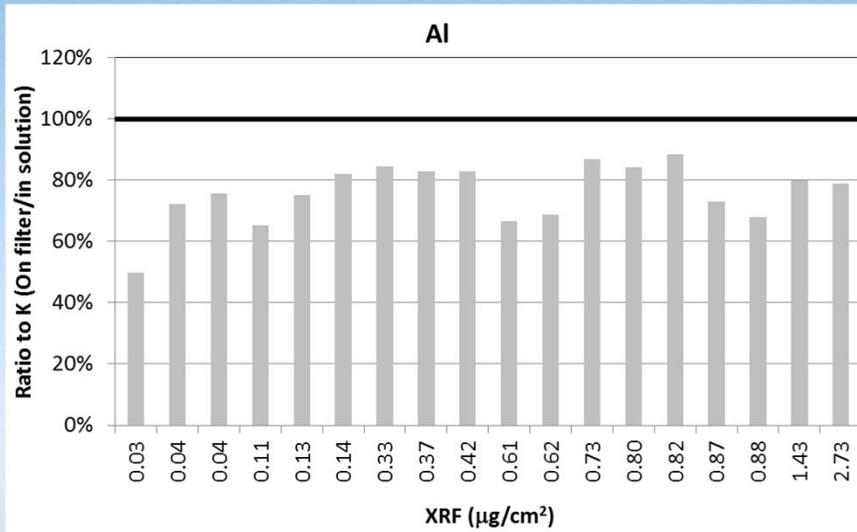


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# Multi-element (ME) XRF Reference Filters

- Quickly evaluate instrument performance for multiple elements
- Assess spectral interferences between elements
- Limited multi-element reference filters for XRF
  - NIST Standard Reference Material (SRM) created with dust collected from HVAC system
    - Does not contain all the elements of interest
    - Only some elements are certified, others are qualitative only
- CPMS uses multi-element standard solutions
  - Using these solutions in the aerosol chamber to create reference filters for the XRF instruments

# UCD Evaluation of ME Standards



# Inter-laboratory Comparison of Multi-element (ME) samples

- Labs

1. CNL, XRF (Epsilon 5)
2. DRI, XRF (Epsilon 5) and ICPMS
3. South Coast Air Quality Management District, XRF (Epsilon 5) and ICPMS
4. New Zealand (NZ), XRF (Epsilon 5) and PIXE
5. United Kingdom (UK), XRF (Epsilon 5) and ICPMS
6. PANalytical, XRF (Epsilon 5)
7. RTI, XRF (Quant'X with linear calibration)
8. JRC, XRF (Quant'X with standardless)

- 6 MEs samples from each

- Low : Mimicking the medians of IMPROVE
- High : Trace elements over than the QLS, major elements the same as Low
- 23 elements, IMPROVE : Co, Cd, Mn and Ba

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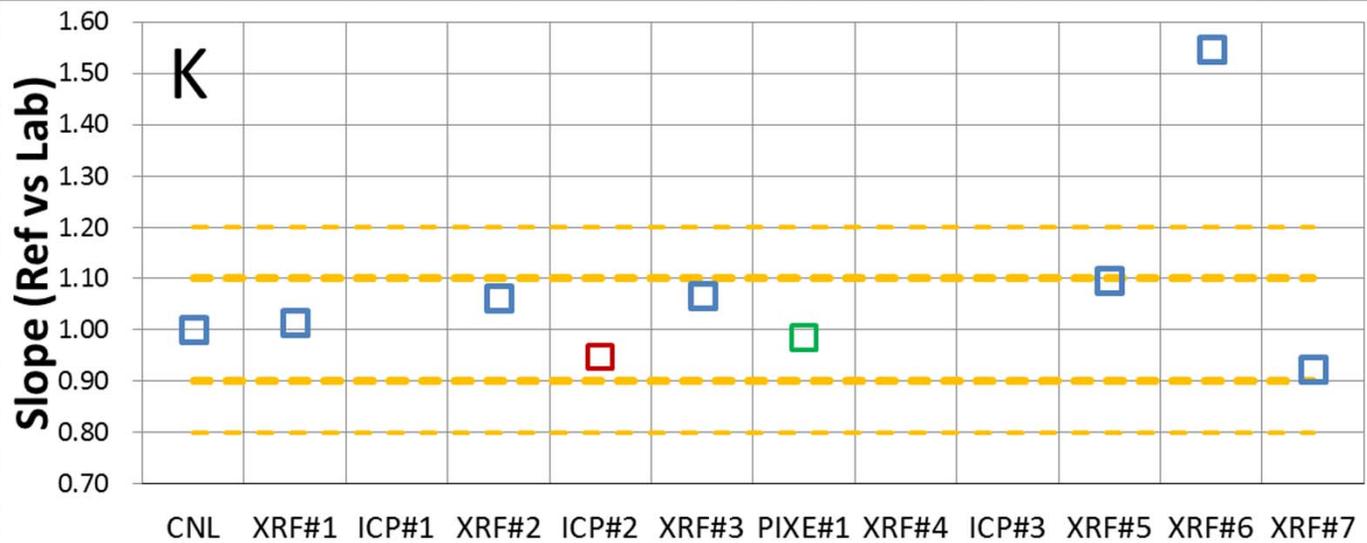
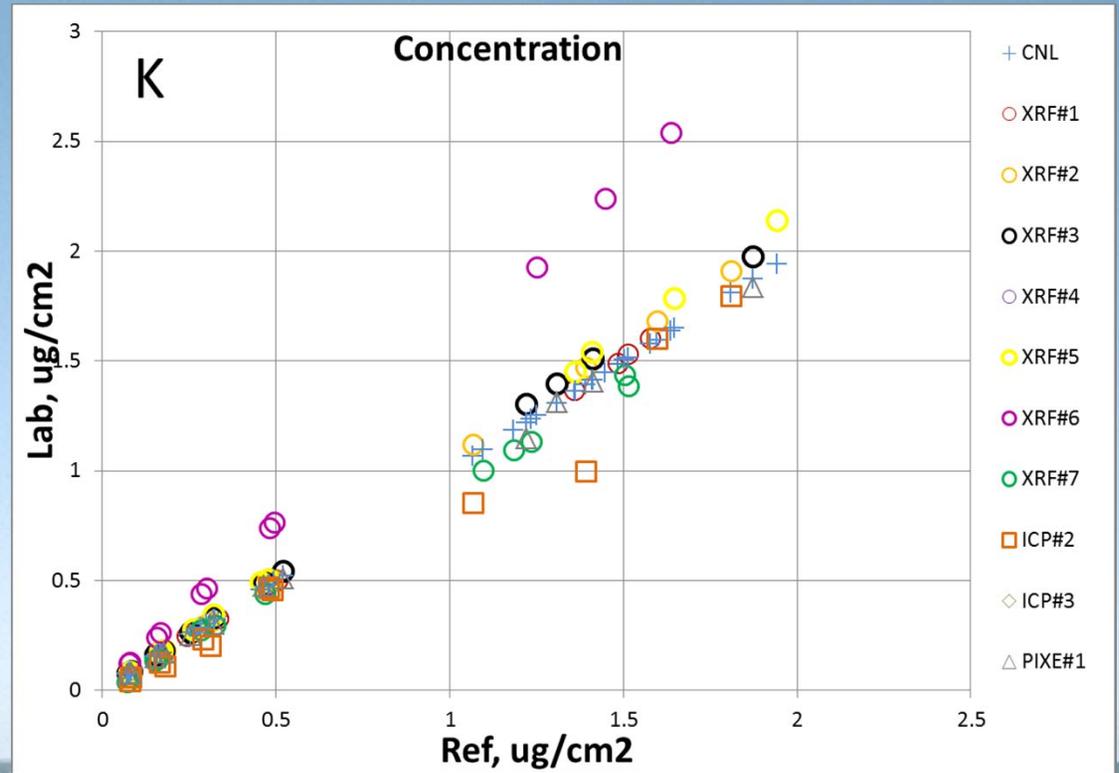
# Methodology for ME Study

- Cannot use mass for reference value
- Assigning reference values for MEs
  - UCD analyzed all samples on Odin

- $$\text{Ref}_{i,filter} \left( \frac{\mu g}{cm^2} \right) = L_{K,filter} \left( \frac{\mu g}{cm^2} \right) \times \left( \frac{C_{i,solution}}{C_{K,solution}} \right)$$

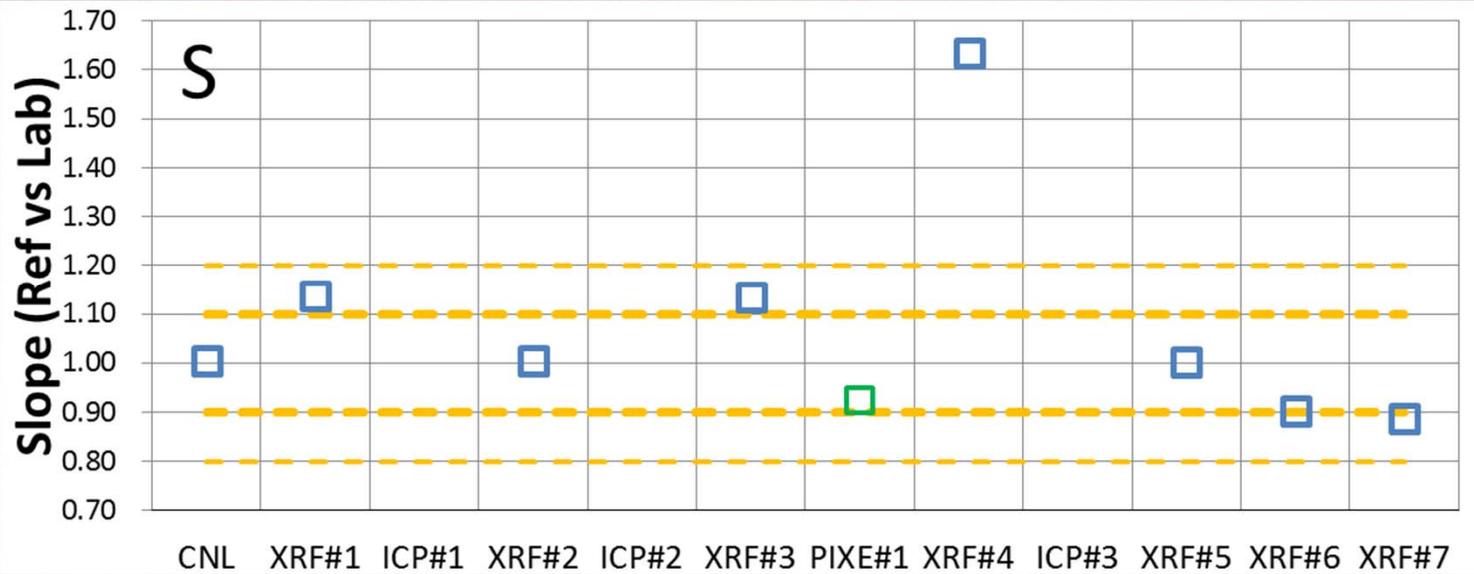
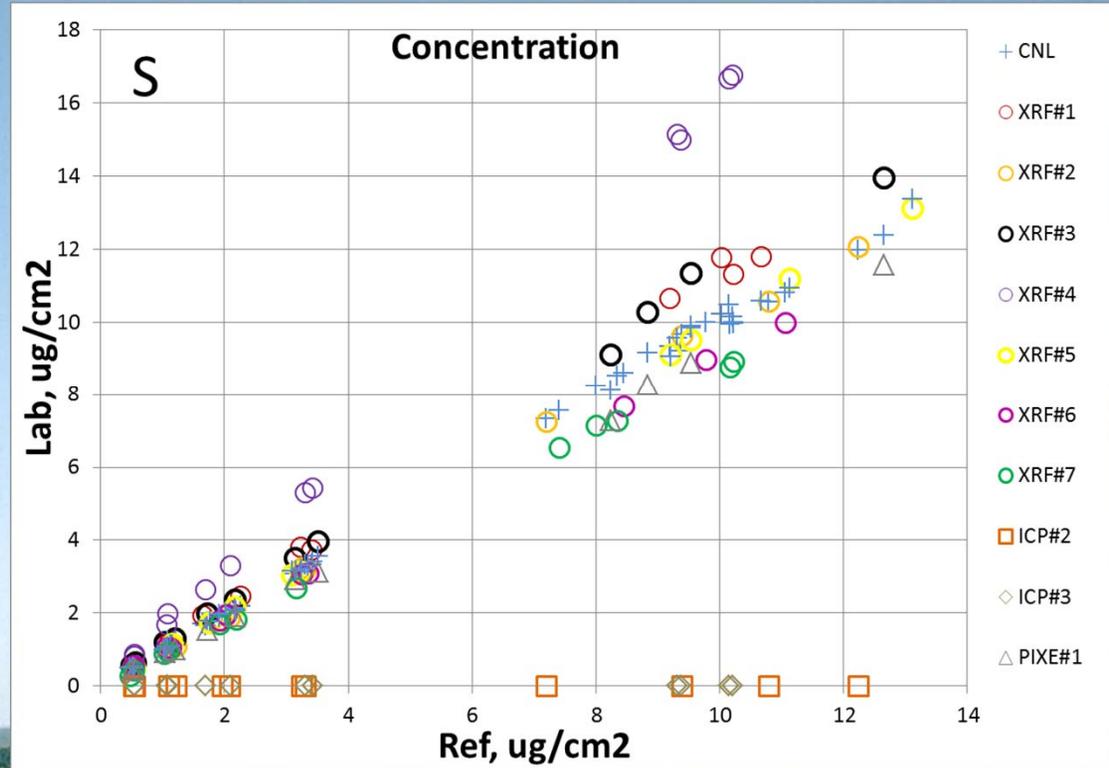
- Where  $\text{Ref}_{i,filter}$  = reference value for element i,
- $L_{K,filter}$  = Potassium loading measured by UCD XRF
- $C_{i,solution}$  = concentration of element i in solution
- $C_{K,solution}$  = concentration of potassium in solution

# Laboratory Inter-Comparison Results: K

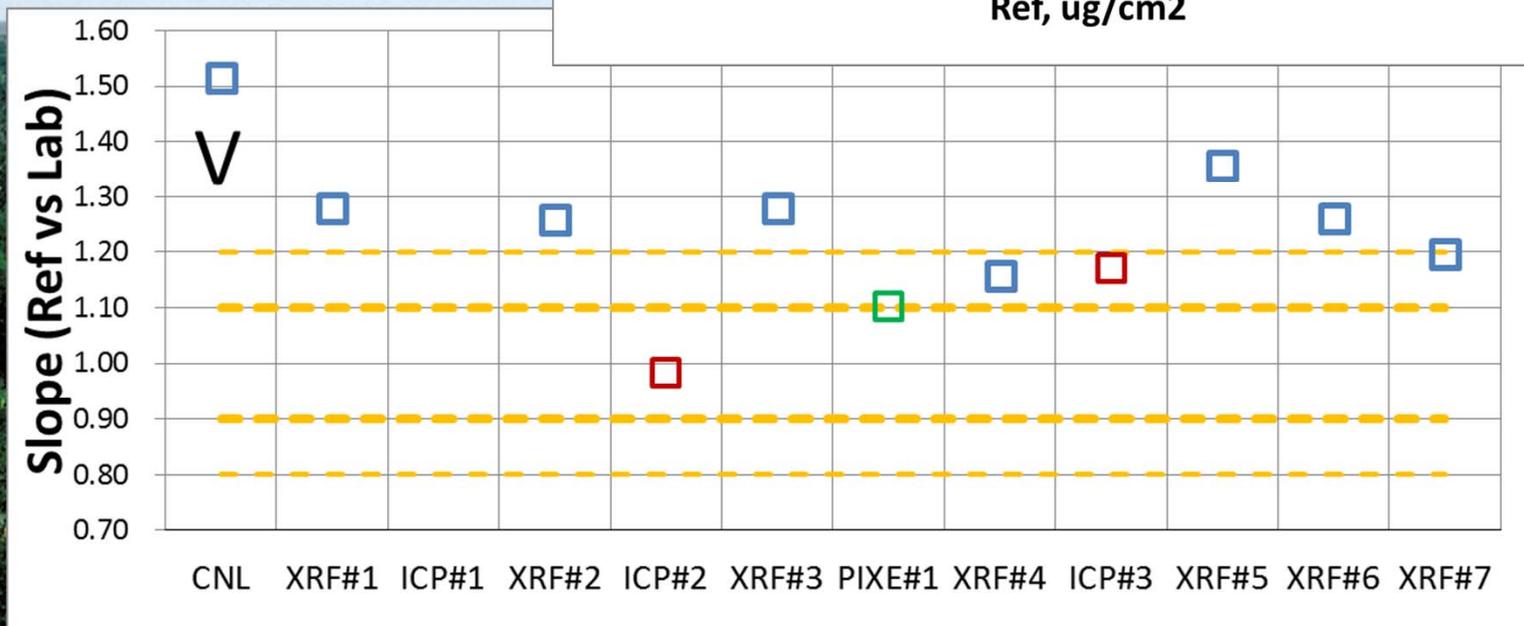
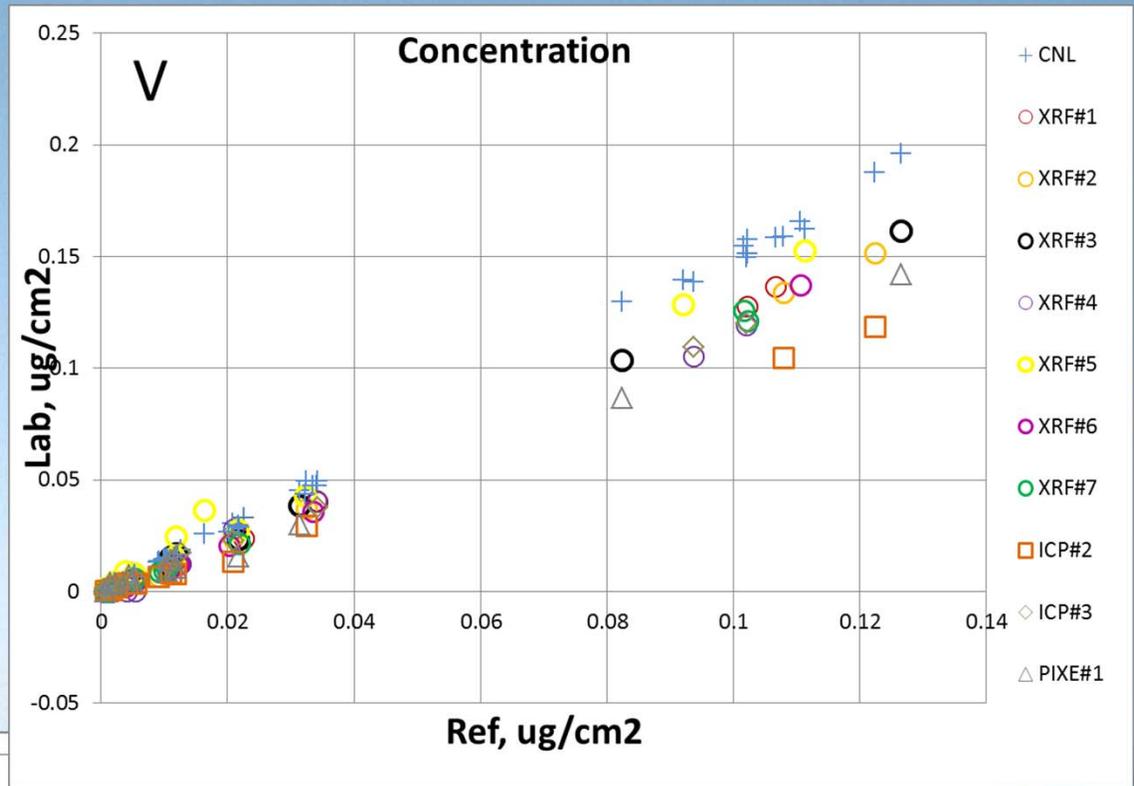


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# Laboratory Inter-Comparison Results: S

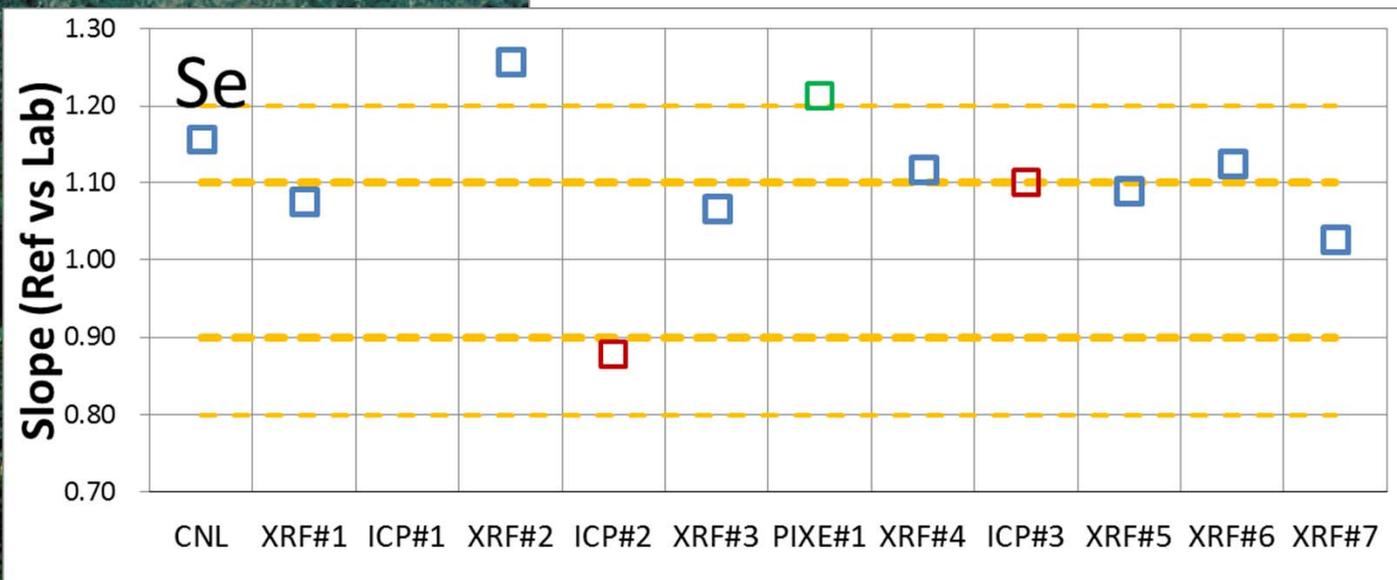
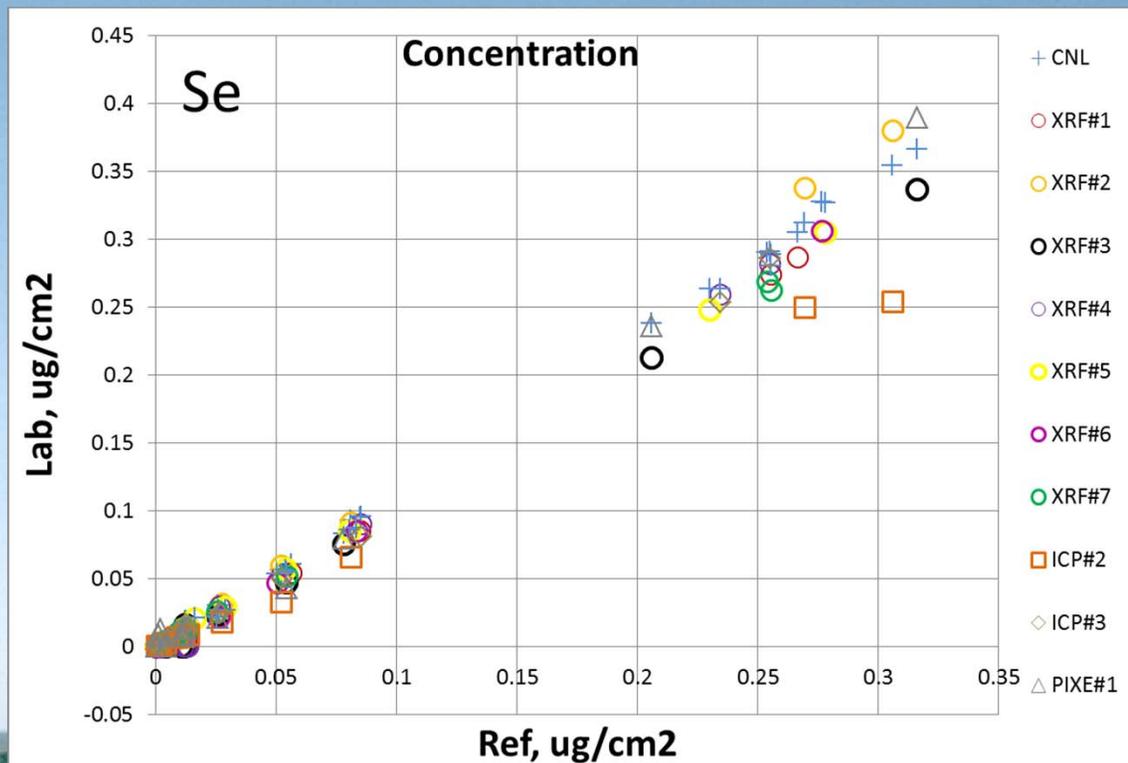


# Laboratory Inter- Comparison Results: V



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# Laboratory Inter- Comparison Results: Se

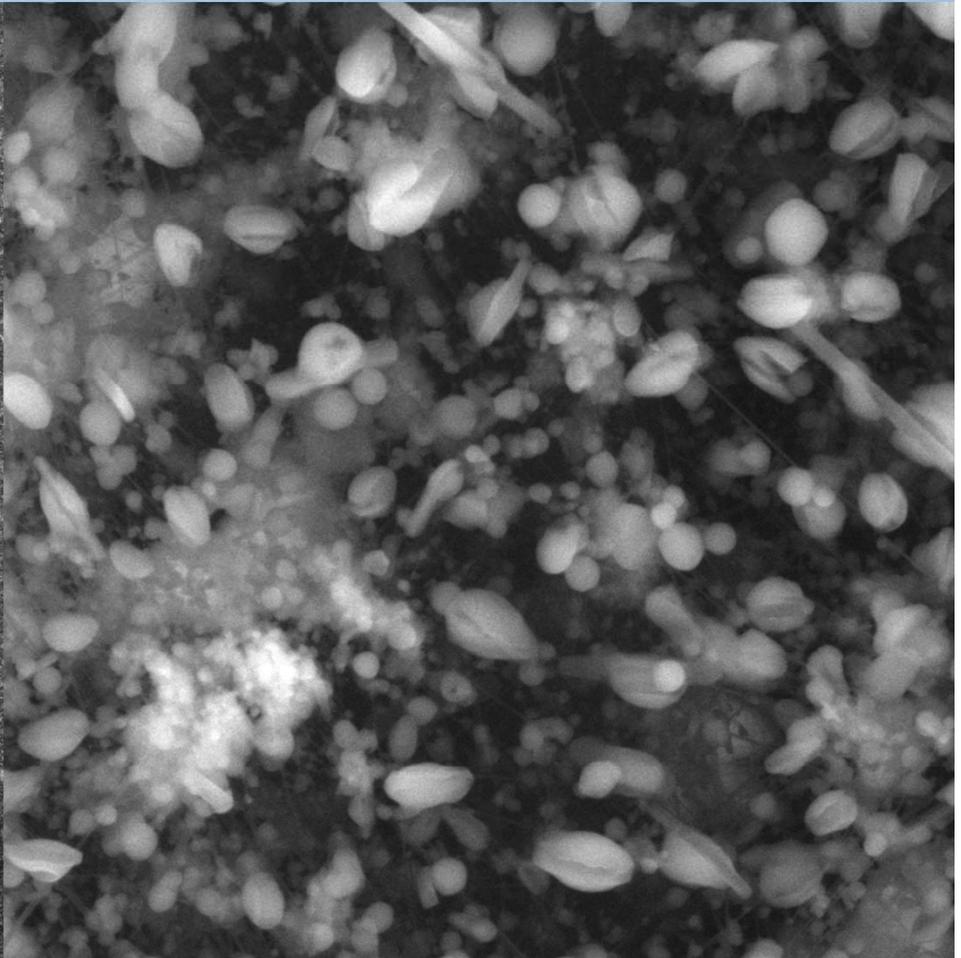
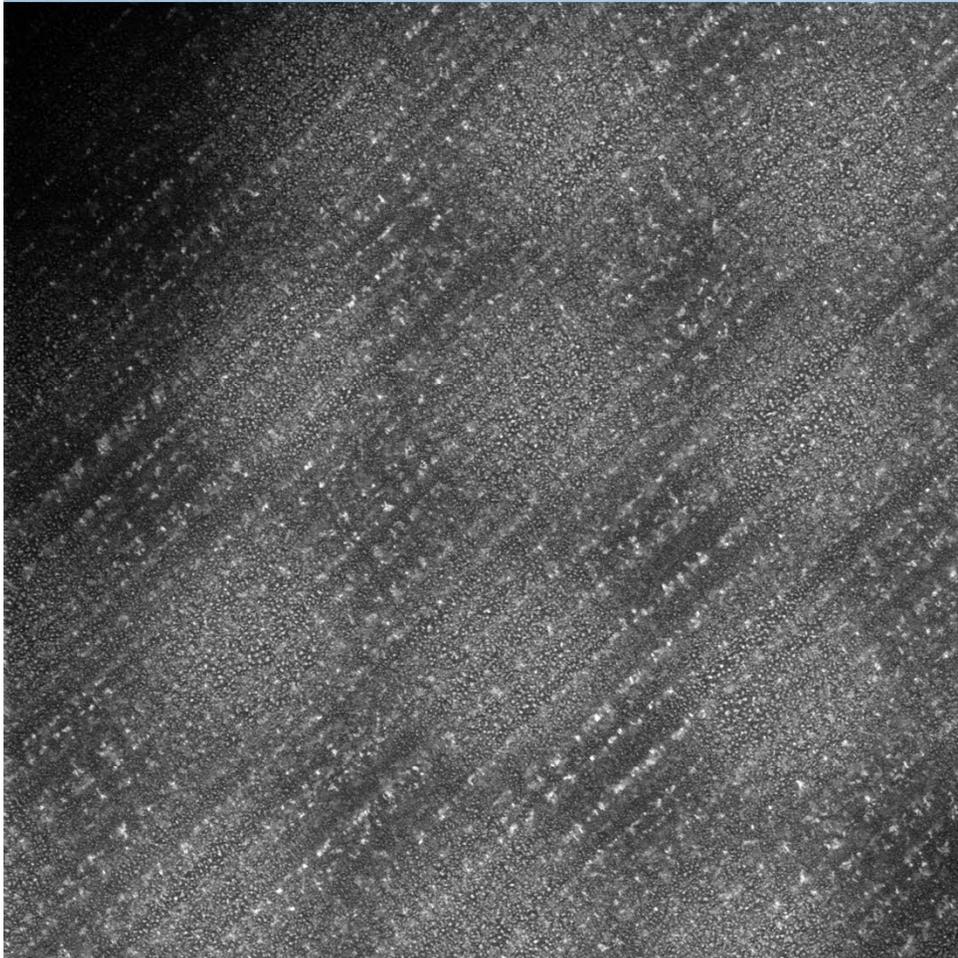


# Deposit Area

- ICPMS analysis yields g/filter while XRF analysis yields g/cm<sup>2</sup>
  - Convert between units using filter area
  - Pall 47 mm PTFE filter internal diameter measures ~38 mm, thus 11.34 cm<sup>2</sup>
  - EPA uses 11.86 cm<sup>2</sup> for Partisol sampler for MTL filters

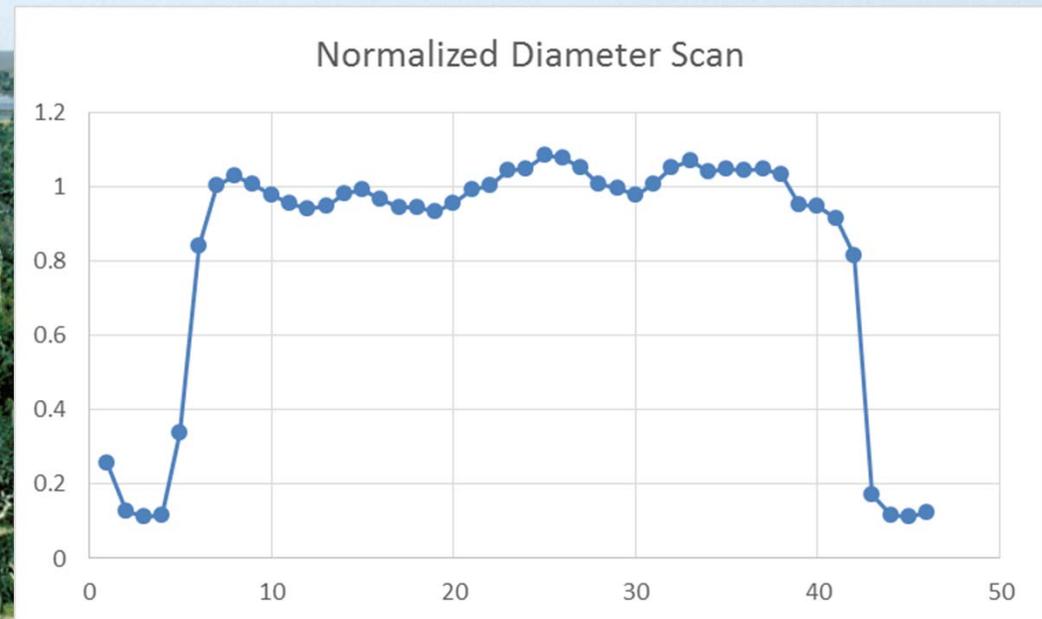
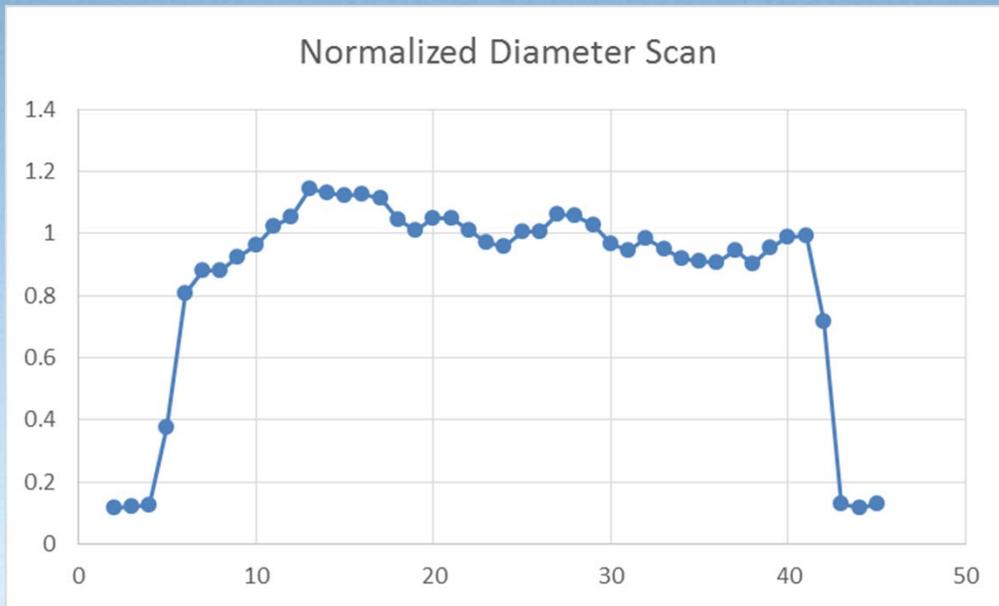
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# SEM Images of Multi-element Deposit



SEM HV: 20.0 kV	WD: 15.74 mm		MIRA3 TESCAN	SEM HV: 30.0 kV	WD: 11.20 mm		MIRA3 TESCAN
View field: 809 μm	Det: LVSTD	200 μm		View field: 27.5 μm	Det: LVSTD	5 μm	
SEM MAG: 357 x	Date(m/d/y): 09/22/14			SEM MAG: 10.5 kx	Date(m/d/y): 09/22/14		

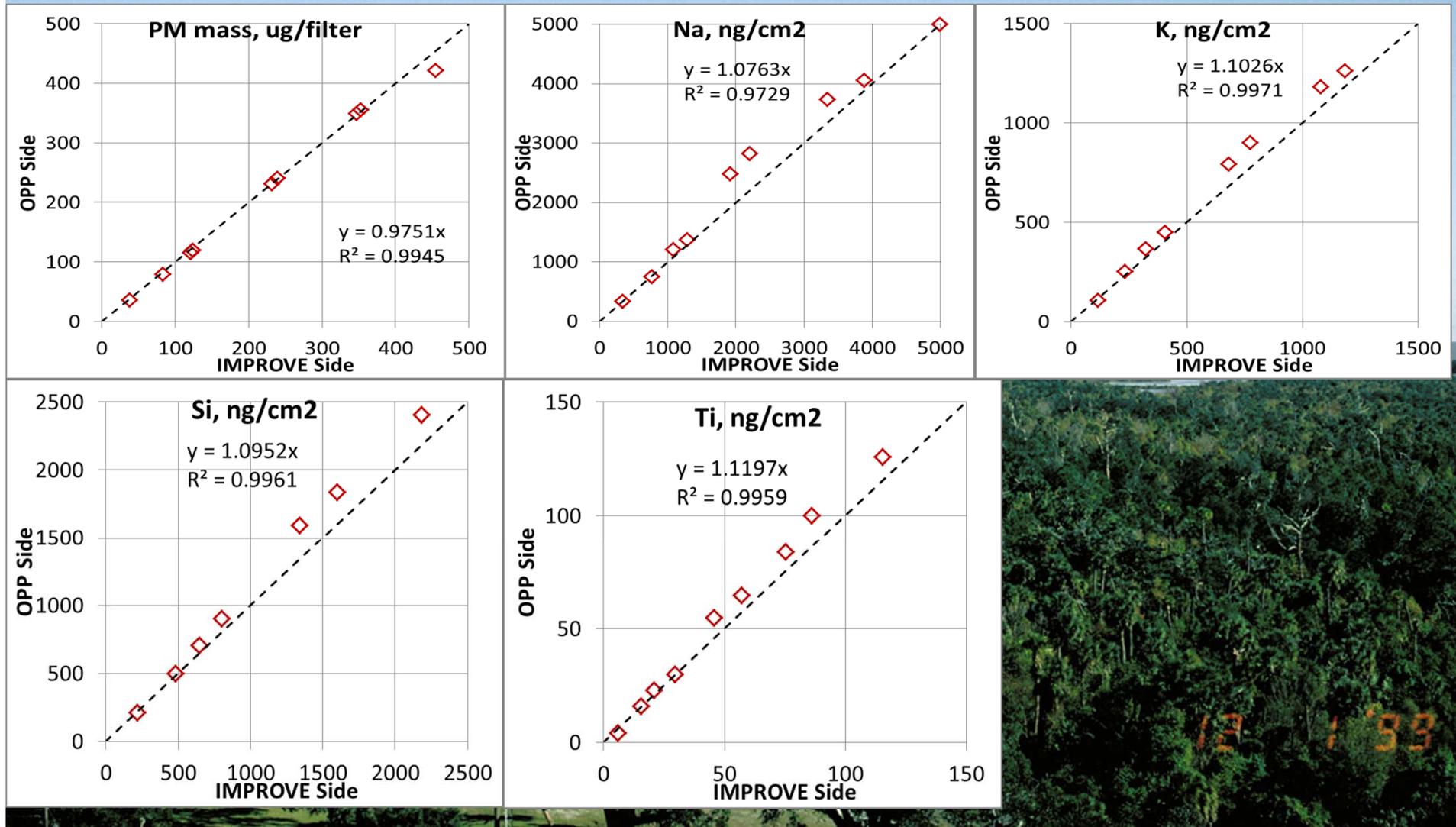
# SEM XRF Results of 47mm filter with deposit on PTFE-face



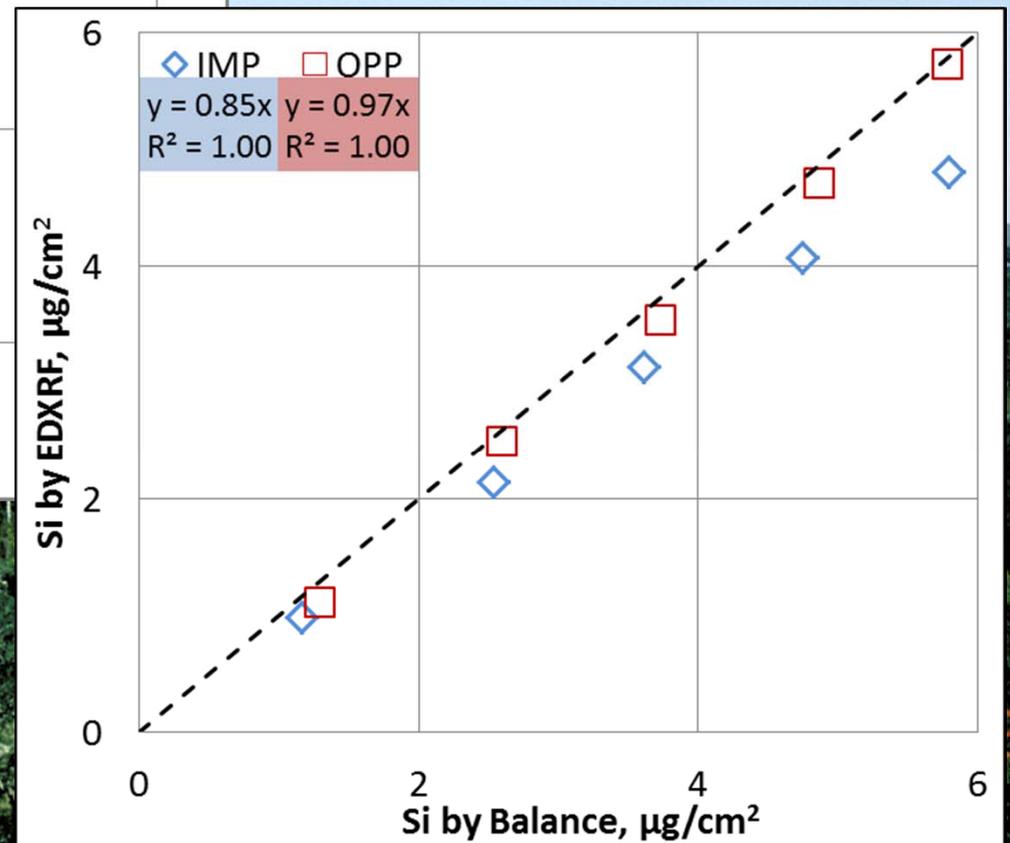
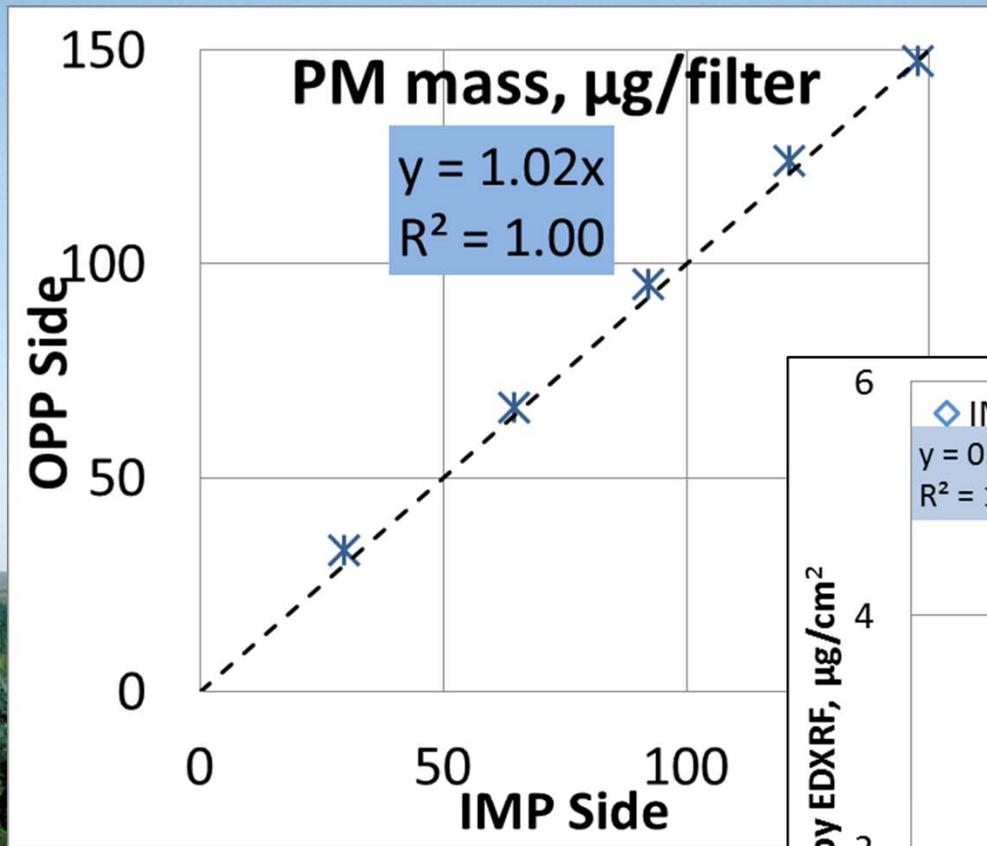
# Laser Ablation Inductively Coupled Plasma Mass Spectrometry (ICPMS)



# ME Solution Samples (Na, Si, K, Ti, Mo and Zr) on 47 mm

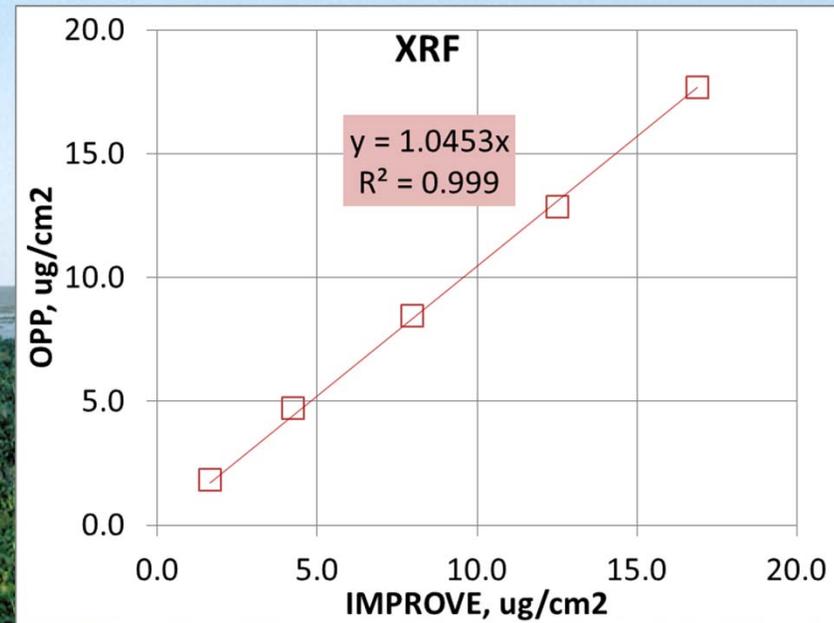
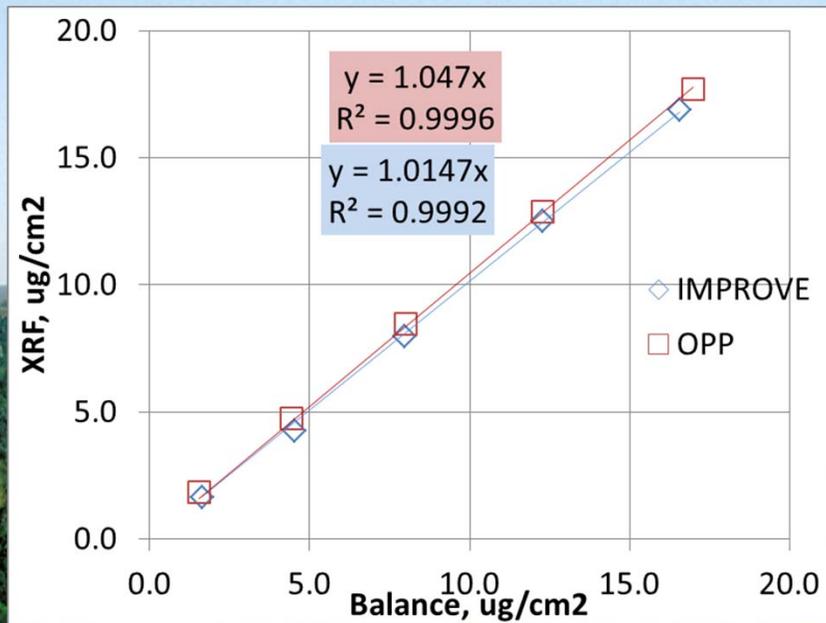


# SiO<sub>2</sub> Samples on 47mm Filters



# $(\text{NH}_4)_2\text{SO}_4$ Samples

25 mm



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# Filter Orientation

- Still assessing the impacts of filter orientation on our operations?



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