



2016 Study: Field Validation of XRF Method for Measuring Soil Hg

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Preliminary Work in 2015



	Task	Finding
✓	Site-specific Hg calibration developed	Great calibration results ($R^2 = 0.996$) over range from 1 to 500 ppm Hg. Improved performance over factory calibration
✓	Method Detection Limit Study	Detection limit of 7.4 ppm Hg achieved with 60 sec analysis time
✓	Method Comparison Study	XRF results agreed with Method 7471A ($R^2 = 0.995$). Precision was equivalent (Mean RSD = 23%)
✓	Moisture Interference Study	Moisture not a significant interference

2016 Field Validation Study

- Task 0 - Literature review
- Task 1 - Field setup and protocol development
- Task 2 - Field validation
 - Coordinated field campaign with AECOM
 - XRF field measurement on collected sample
 - Method 7471A lab measurement on collected sample
- Task 3 – Additional laboratory validation



2016 Field Study Approach



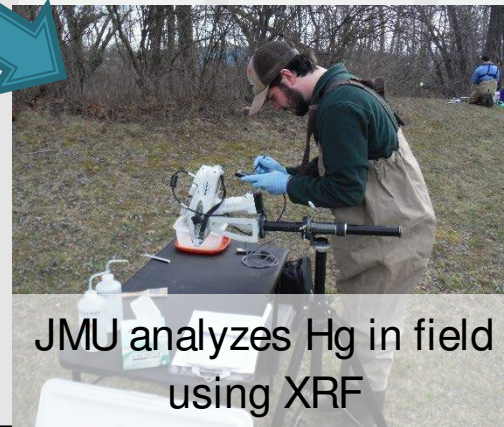
AECOM collected
cores or surficial soil



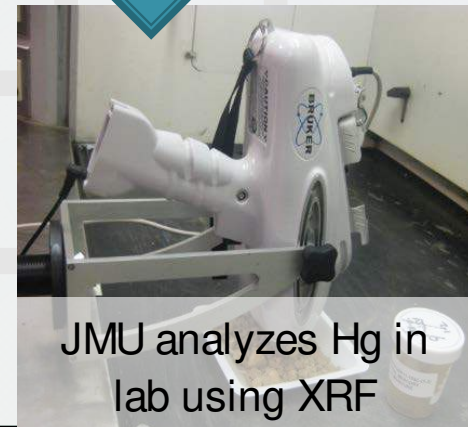
Samples shipped to Lancaster Labs
for Hg analysis using Method 7471A



AECOM homogenized
and characterized
composites



JMU analyzes Hg in field
using XRF



JMU analyzes Hg in
lab using XRF

Field Setup (surficial soil)



AECOM hand collected soils within 2-ft interval with a trowel and homogenized in stainless steel bowl

JMU analyzed homogenized sample directly in stainless steel bowl



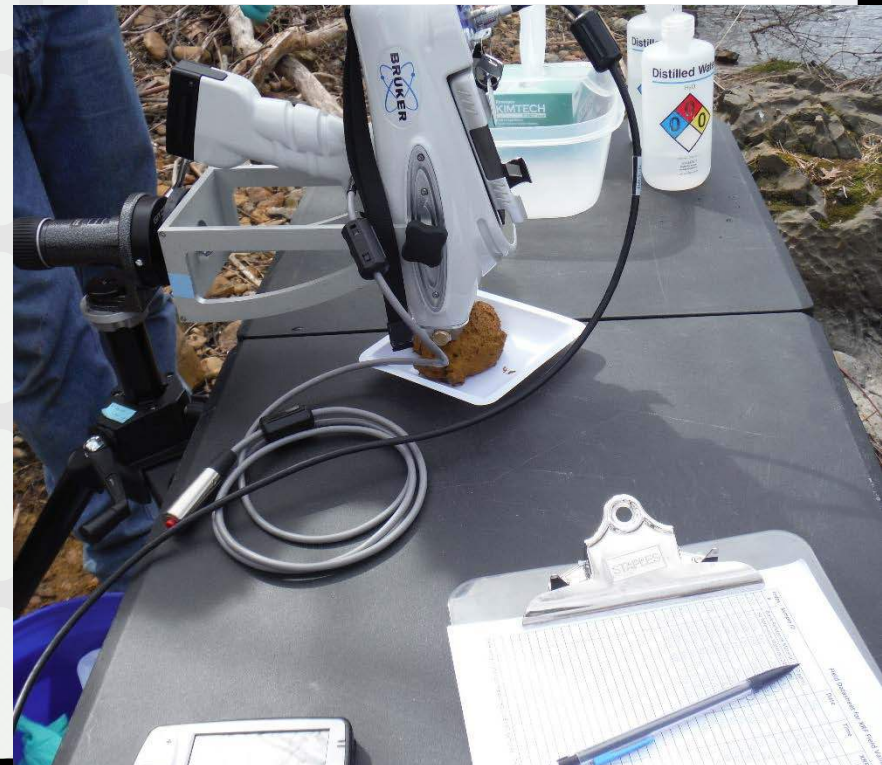
Field Setup (cores)



JMU placed homogenized sample in plastic weigh boats and analyzed in field



AECOM collected cores with a hand auger and homogenized 1-ft intervals on plastic sheeting



Field Setup (cores)



JMU placed homogenized sample in plastic weigh boats and analyzed in field

Video



AECOM collected cores with a hand auger and homogenized 1-ft intervals on plastic sheeting



2016 Field Study Details

- Dates: March 7-16, 2016
- Collected: 236 samples from 9 BMAs
- Hg Results:
 - 37% of samples were < MDL of 7.4 ppm
 - Hg levels ranged from < MDL – 1411 ppm
- QA Elements:



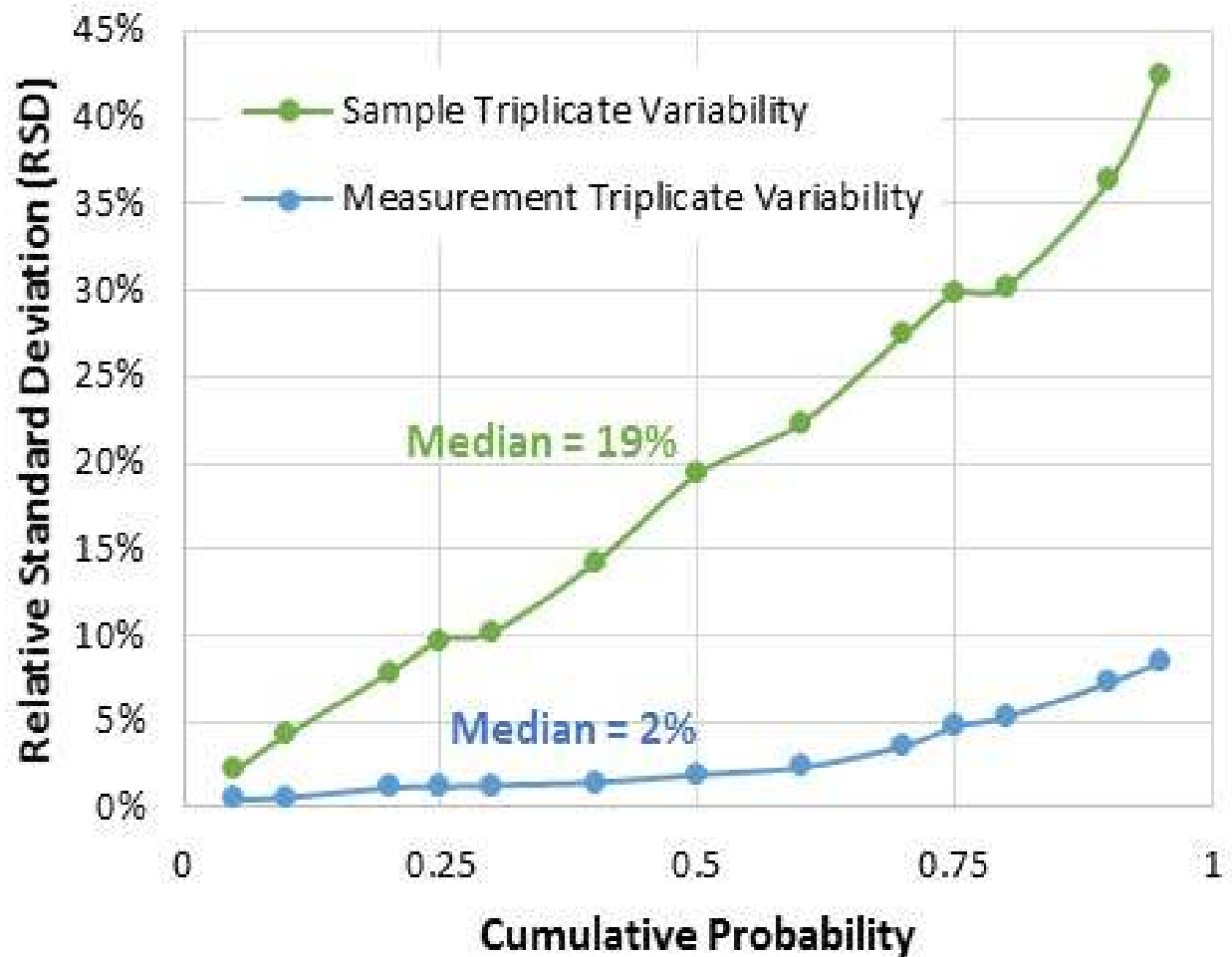
QA Sample	Frequency	Description
Blank	5% (5.5%)	Soil sample with <1 ppm Hg
Hg Reference Material	5% (5.5%)	Soil sample with 245 ppm Hg
Sample Triplicates	15% (16%)	Analysis of 3 subsamples
Measurement Triplicates	5% (6.8%)	3 analyses of same subsample

General Field Observations

- XRF was a robust field instrument
 - 7 full days of continuous use
 - Over 380 measurements
 - No issues
- Results were practically instantaneous
 - Allowed real-time decision making
 - Extended BMA delineations
 - Avoided additional field deployments

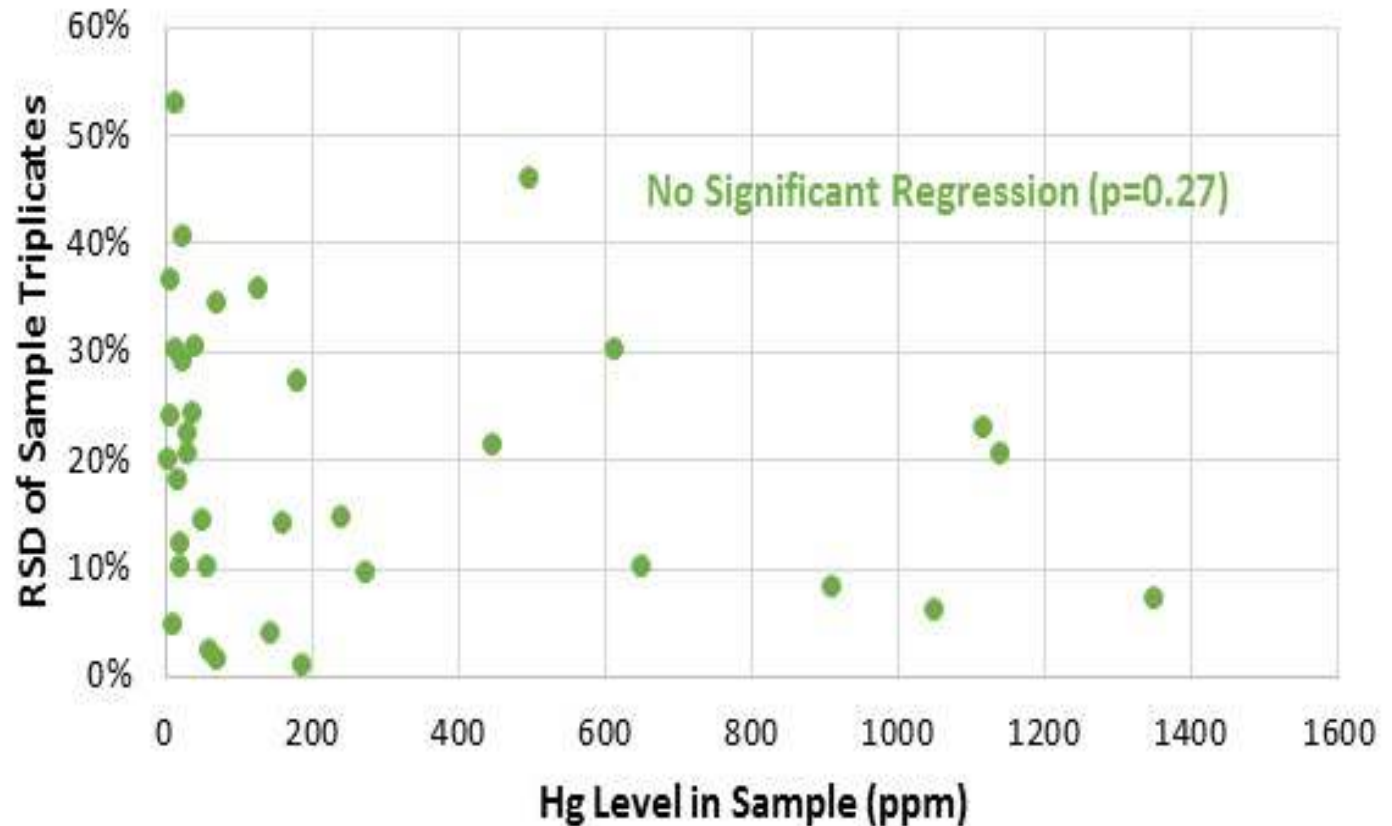


Preliminary Data: Triplicate Results



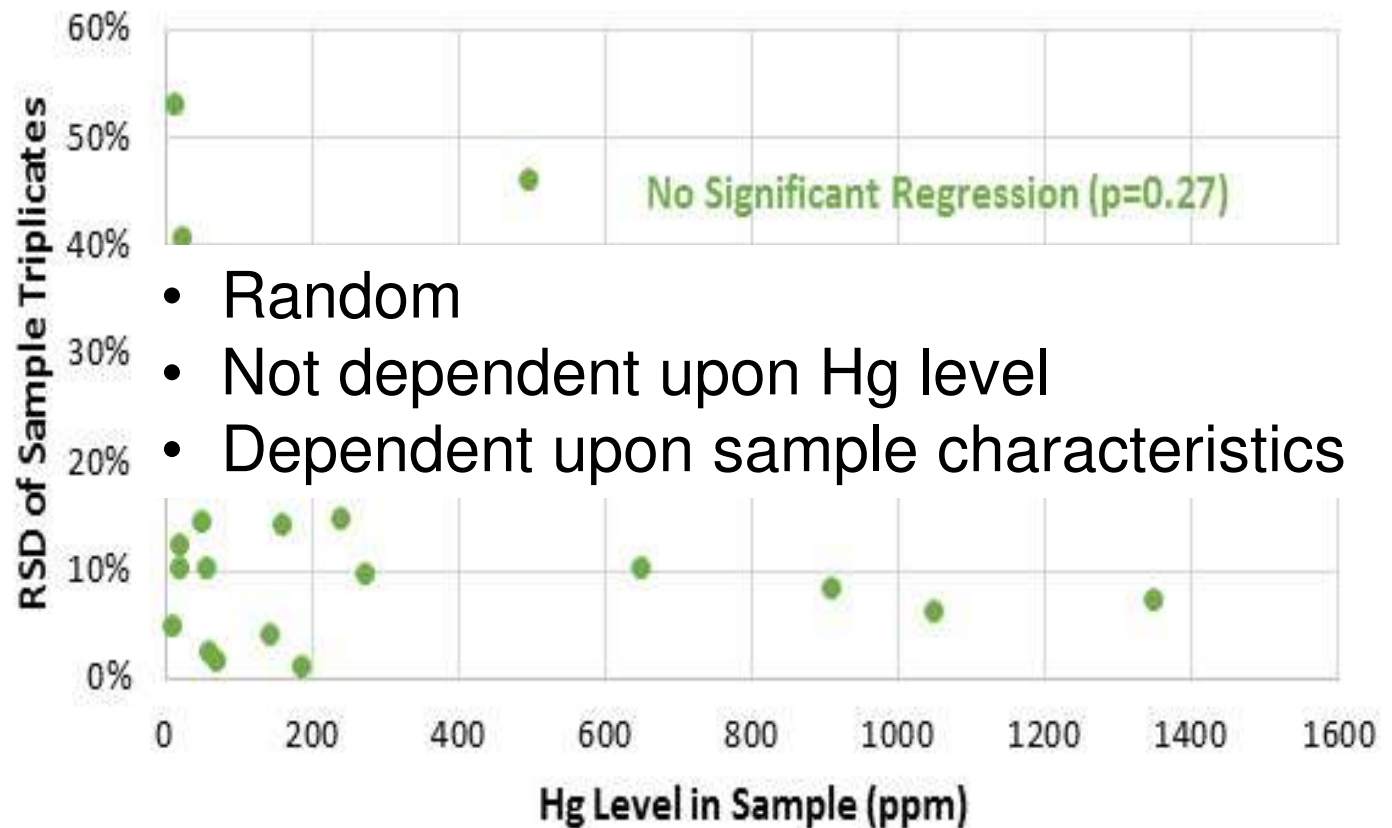
Preliminary Data: Triplicate Results

Sample Variability



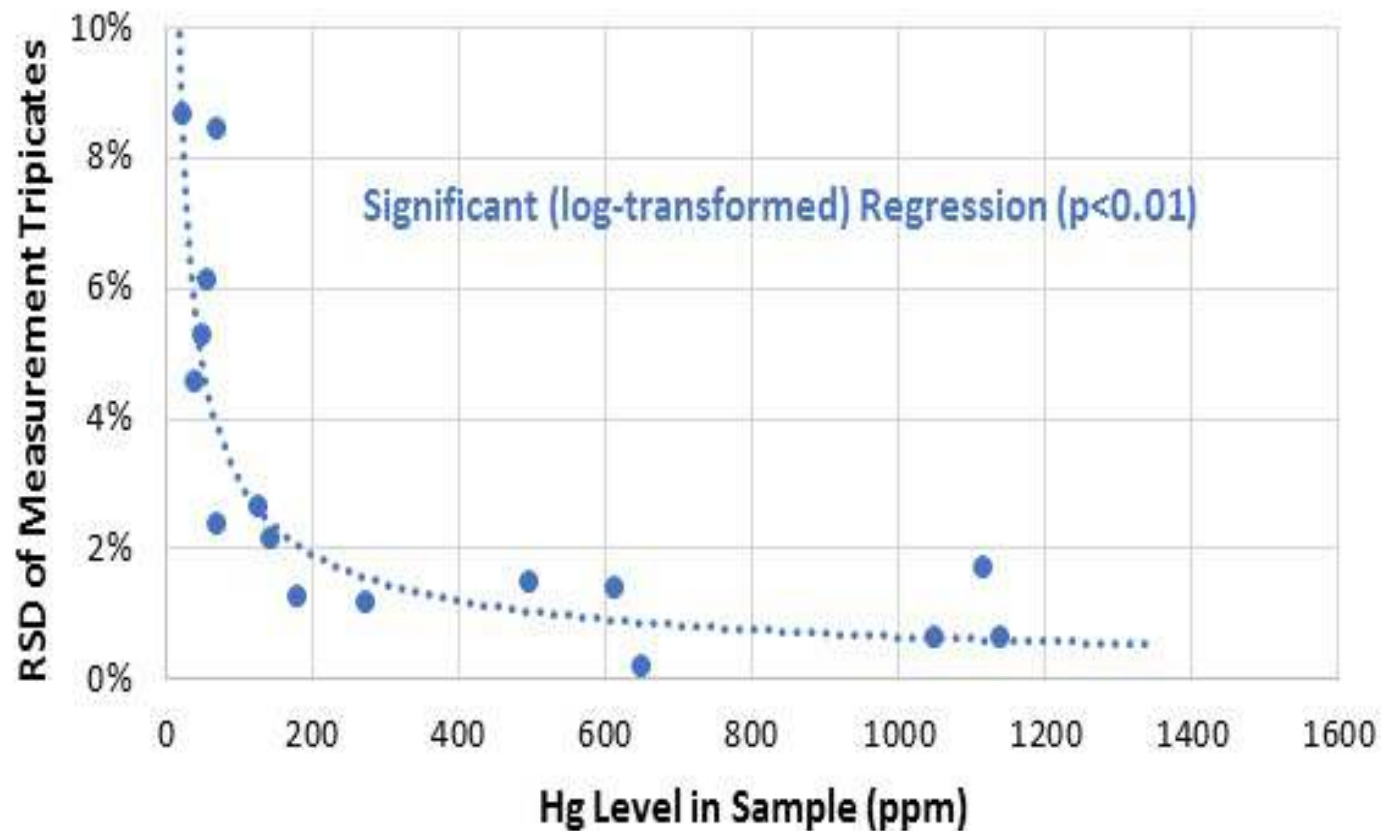
Preliminary Data: Triplicate Results

Sample Variability



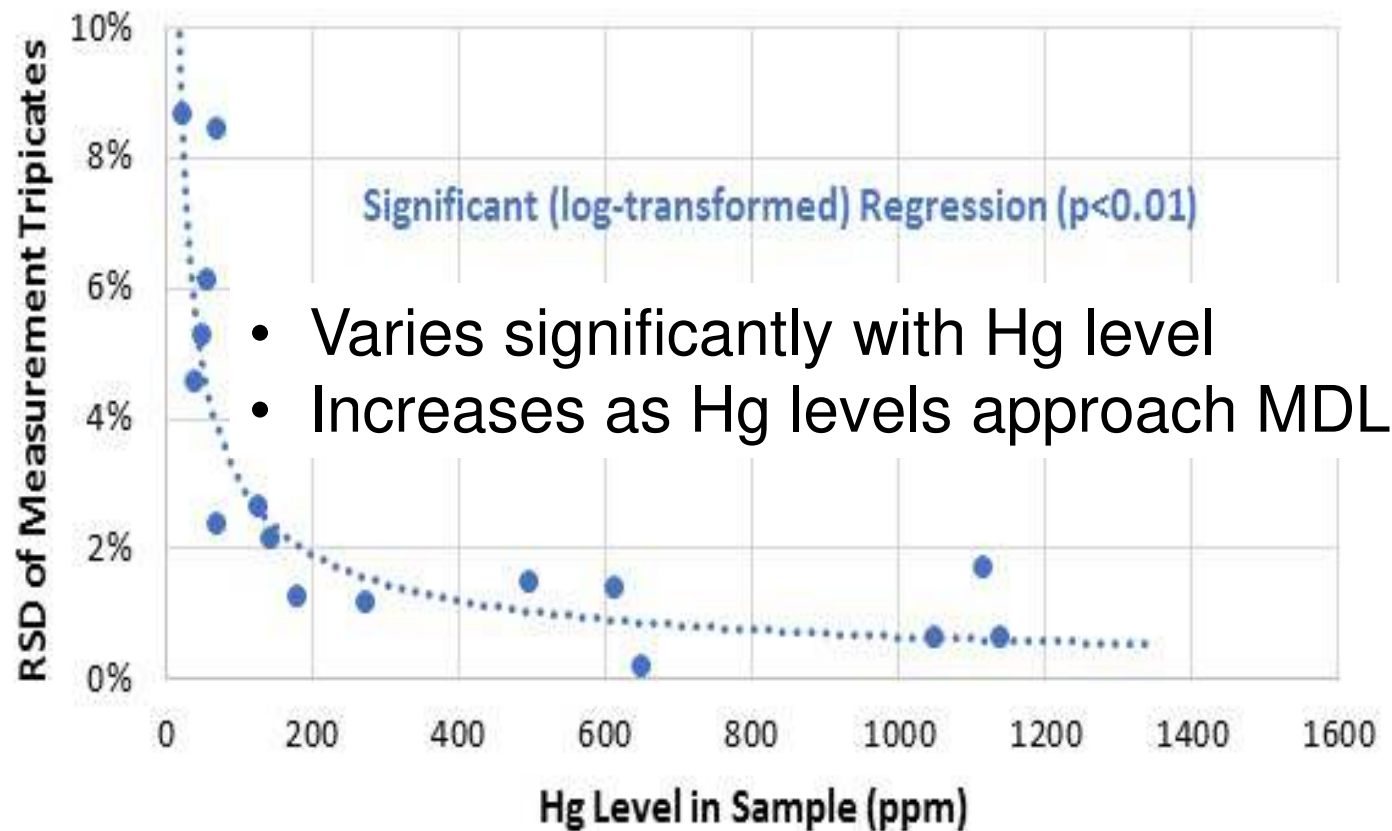
Preliminary Data: Triplicate Results

Measurement Variability



Preliminary Data: Triplicate Results

Measurement Variability

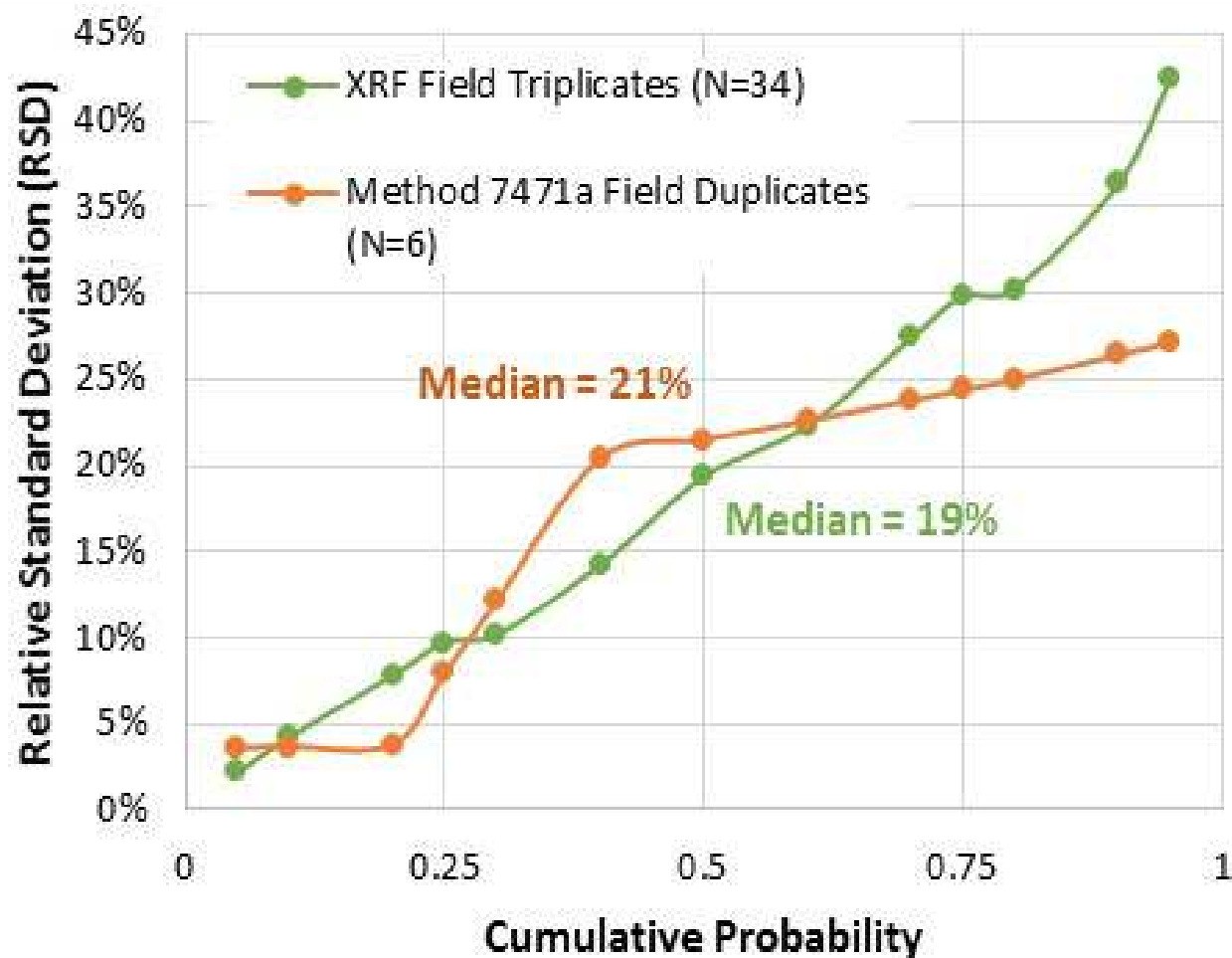


Implications

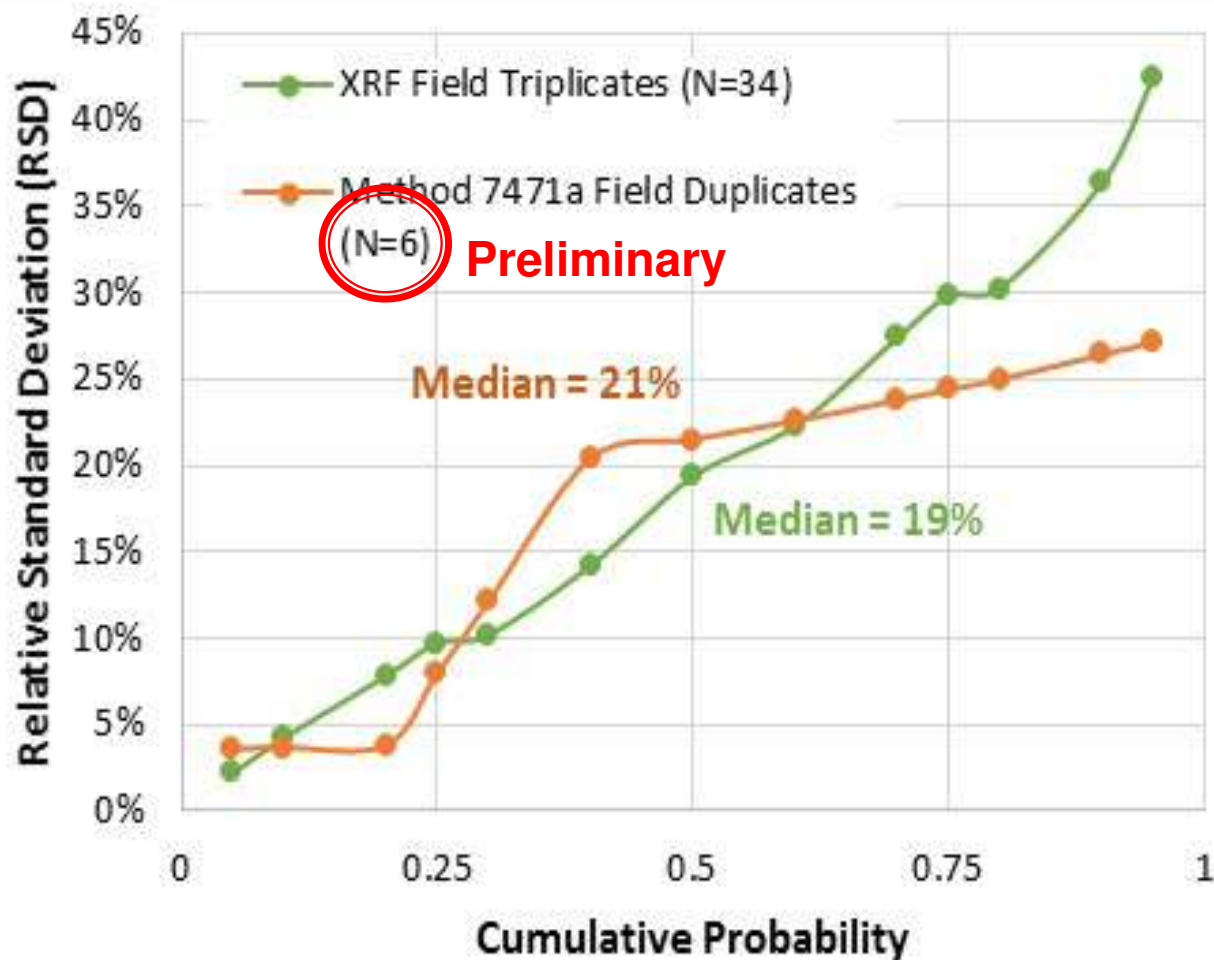
- XRF measurement variability is much lower than sample variability
- When comparing results between methods, 3 sources of variability exist
 - Sample variability (averaging 20%, but greater for some samples)
 - XRF measurement variability
 - Method 7471a measurement variability
- Regardless of the accuracy of a method (any method), precision will be constrained by sample variability



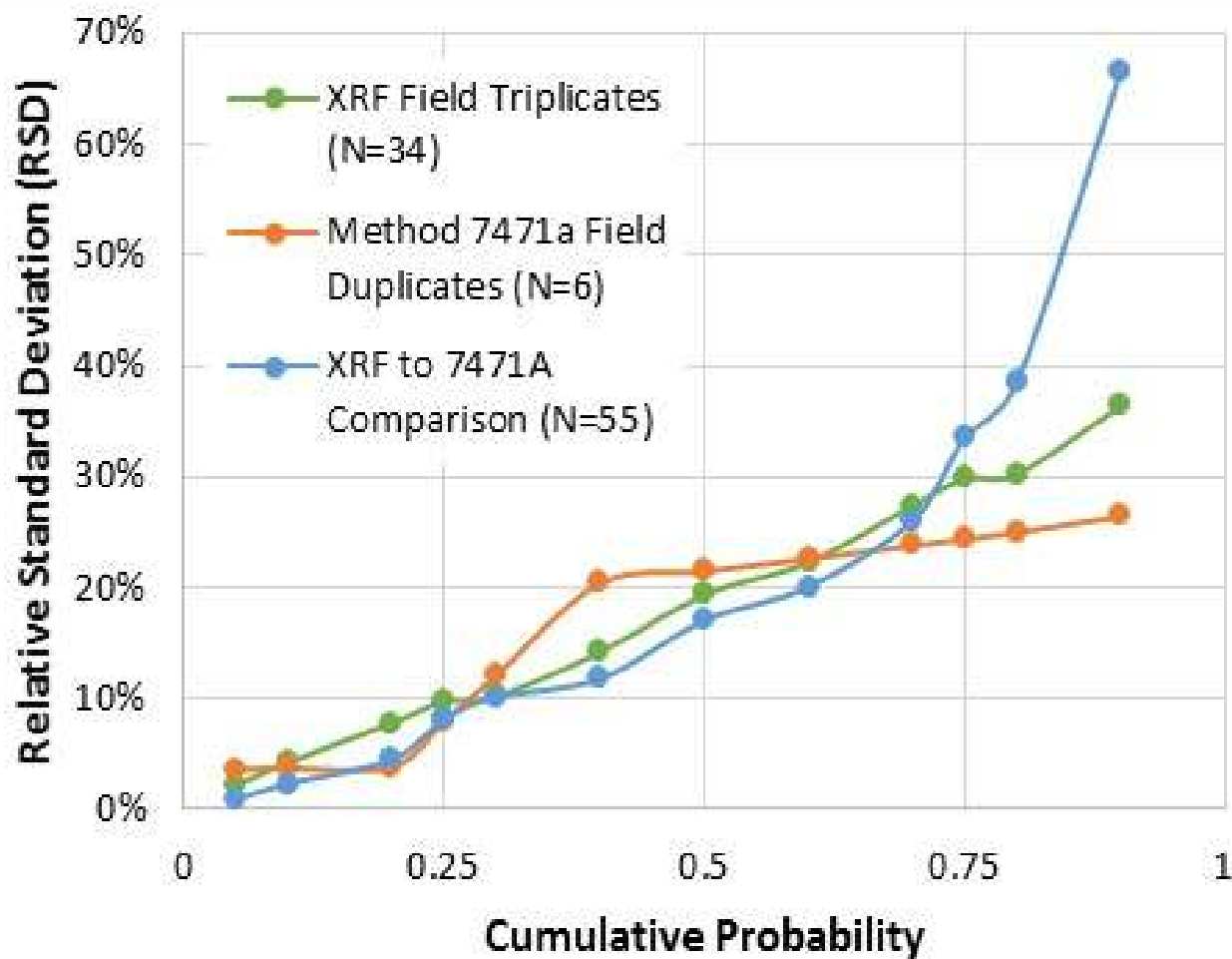
Preliminary Data: Method 7471A Comparison



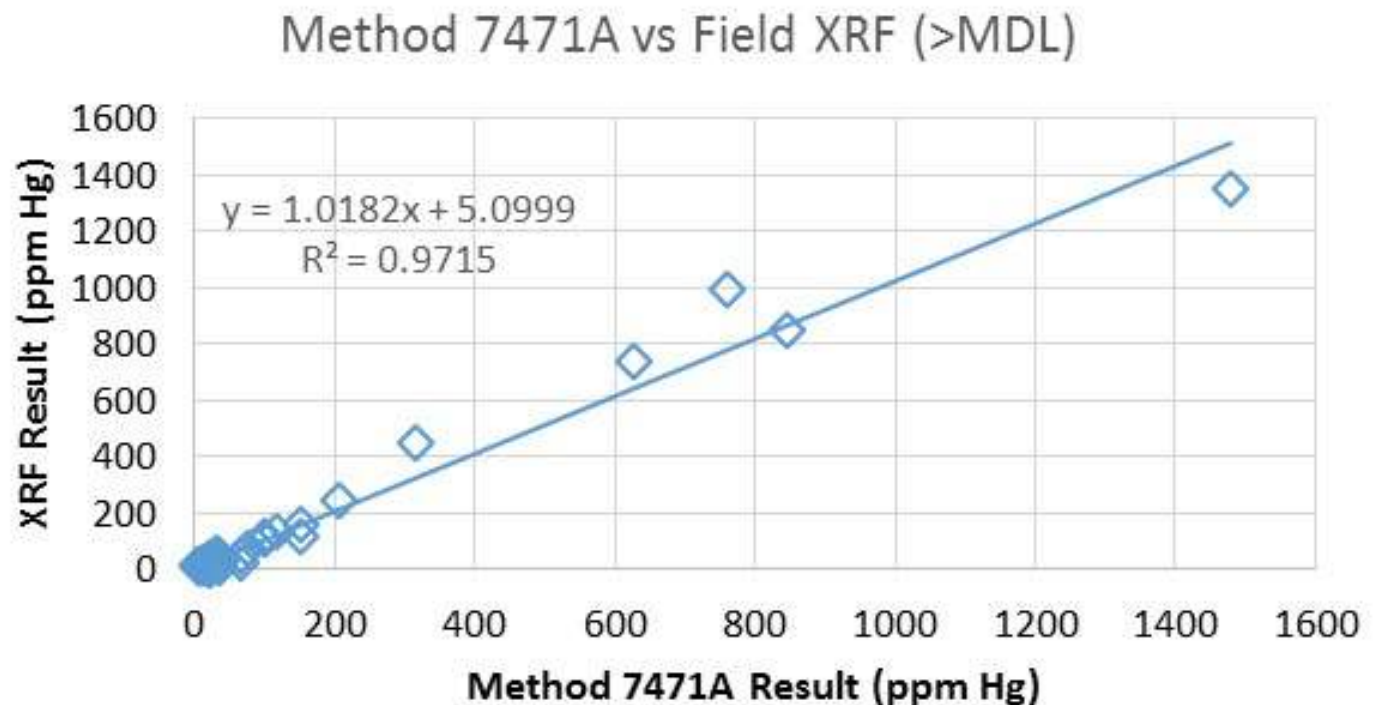
Preliminary Data: Method 7471A Comparison



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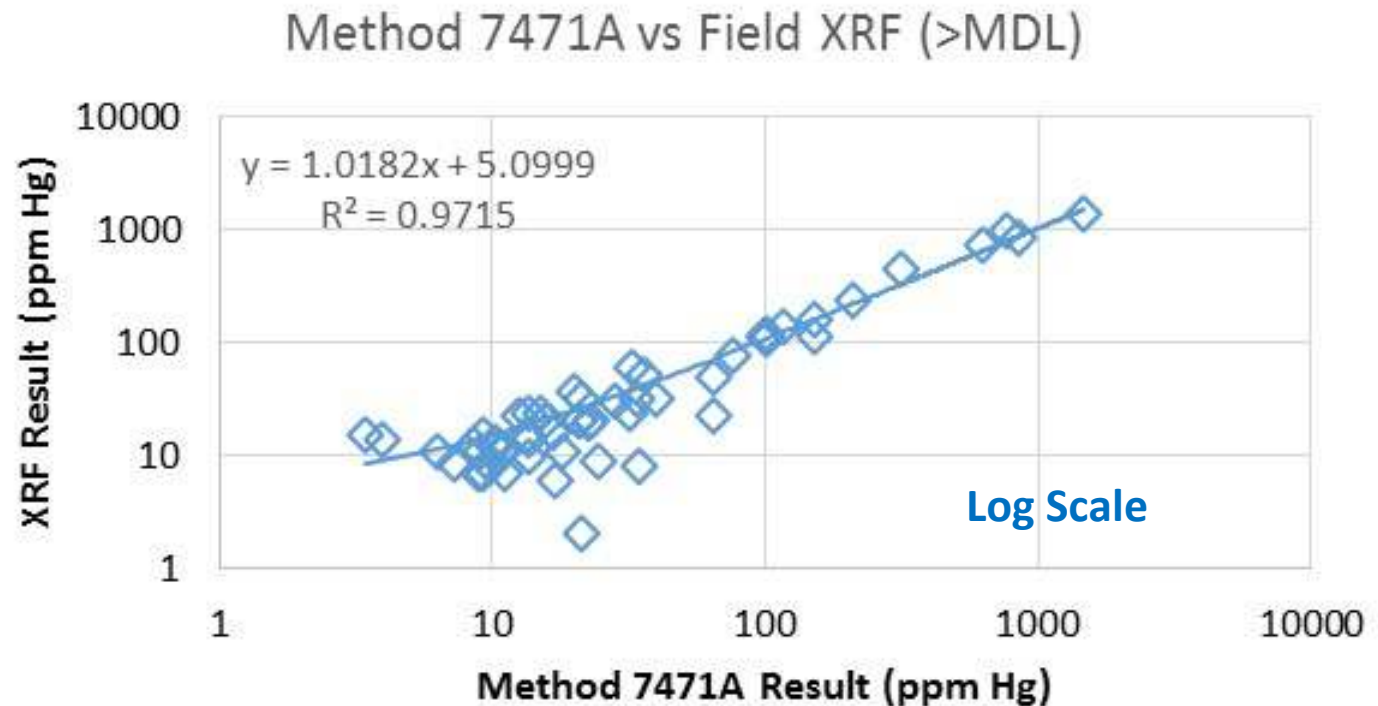


Preliminary Data: Field and Lab Comparison



Only about half of the data are back from the lab

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Preliminary Implications

- Differences between XRF and Method 7471a results are consistent with sample duplicate variability
- Regression between XRF and Method 7471a results is very good ($R^2=0.97$)
- Regression is nearly 1:1 (slope 1.018)
- Only about half of collected data are available and analyzed so far
 - More results and analysis to come

