XRF Analysis of Soil Hg –

What about Moisture?

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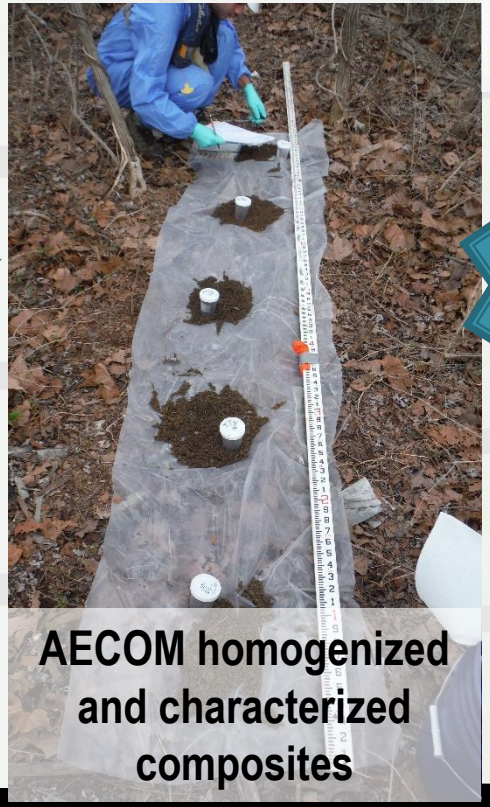
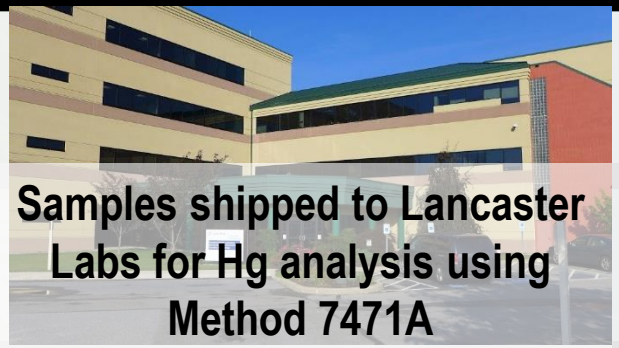
Hunter Wines, Joseph Luther

Review: 2016 XRF Study Approach

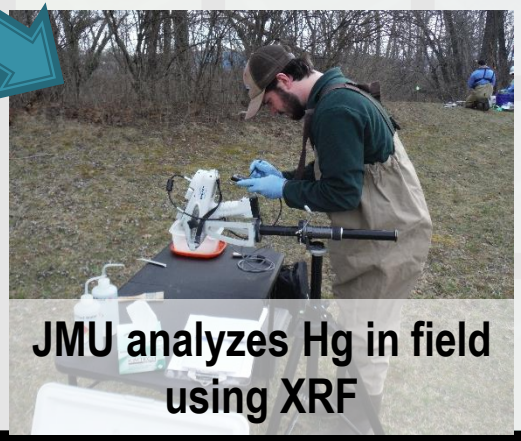
AECOM collected cores or surficial soil



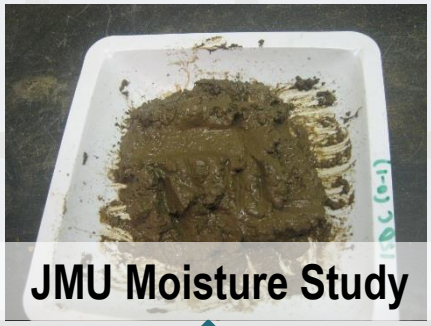
AECOM homogenized and characterized composites



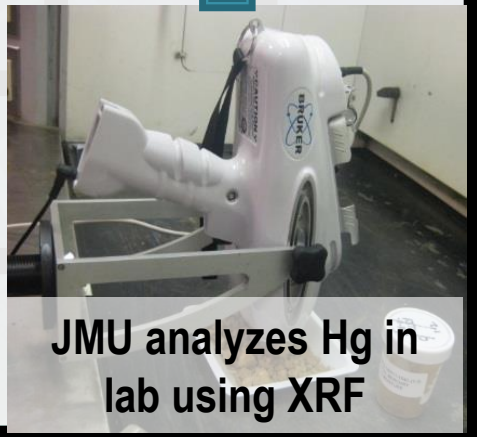
Samples shipped to Lancaster Labs for Hg analysis using Method 7471A



JMU analyzes Hg in field using XRF



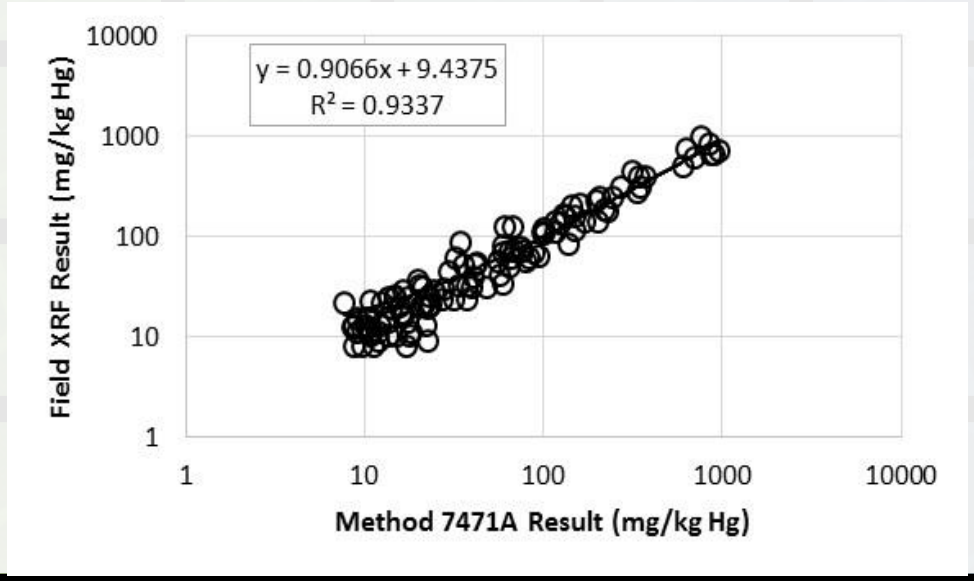
JMU Moisture Study



JMU analyzes Hg in lab using XRF

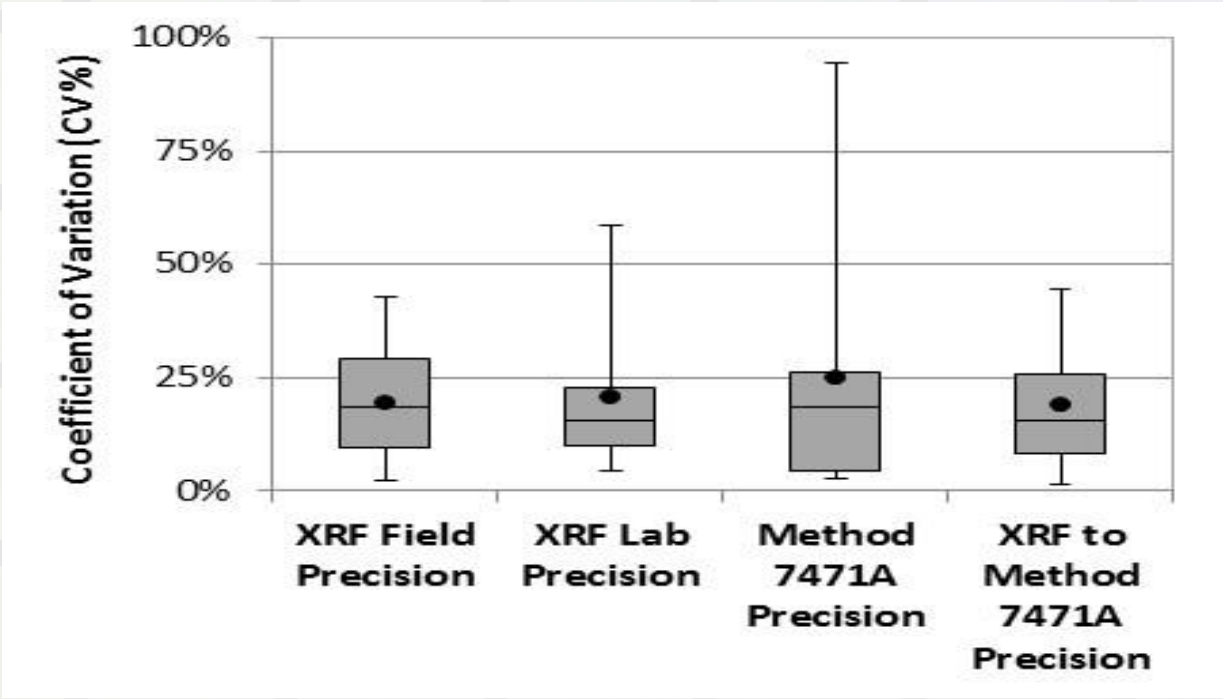
Previously Summarized Findings

- Accuracy at the detection levels was very good – XRF results agreed with 93% of samples that were <7.4 ppm according to Method 7471A
- Accuracy throughout the range of 7.4 to 1000 ppm was very good



Previously Summarized Findings

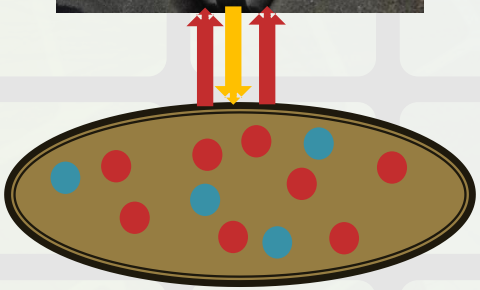
- Precision was comparable to Method 7471A and constrained by sample heterogeneity
  - Median XRF precision = 18% CV
  - Median Method 7471A precision = 19% CV

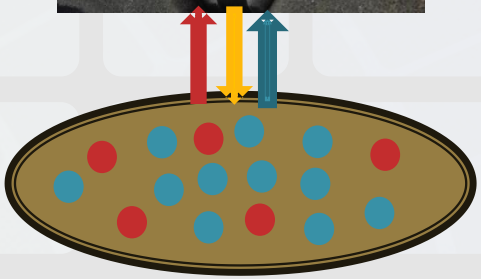


Potential Impact of Moisture

- Water molecules can be an interference with the XRF reading

- X-rays can strike Hg and/or water molecules and dilute the Hg signal, when moisture is high





Low Moisture

Hg molecules

Water molecules

High Moisture

Moisture Study Design

- Spiked Hg samples were prepared at 12 different concentrations (0, 2, 5, 10, 15, 25, 50, 75, 125, 250, 500, and 1000 ppm)

North River soil

dried and sieved

spiked with Hg

shaken 24 hr

air dried

ground and sieved

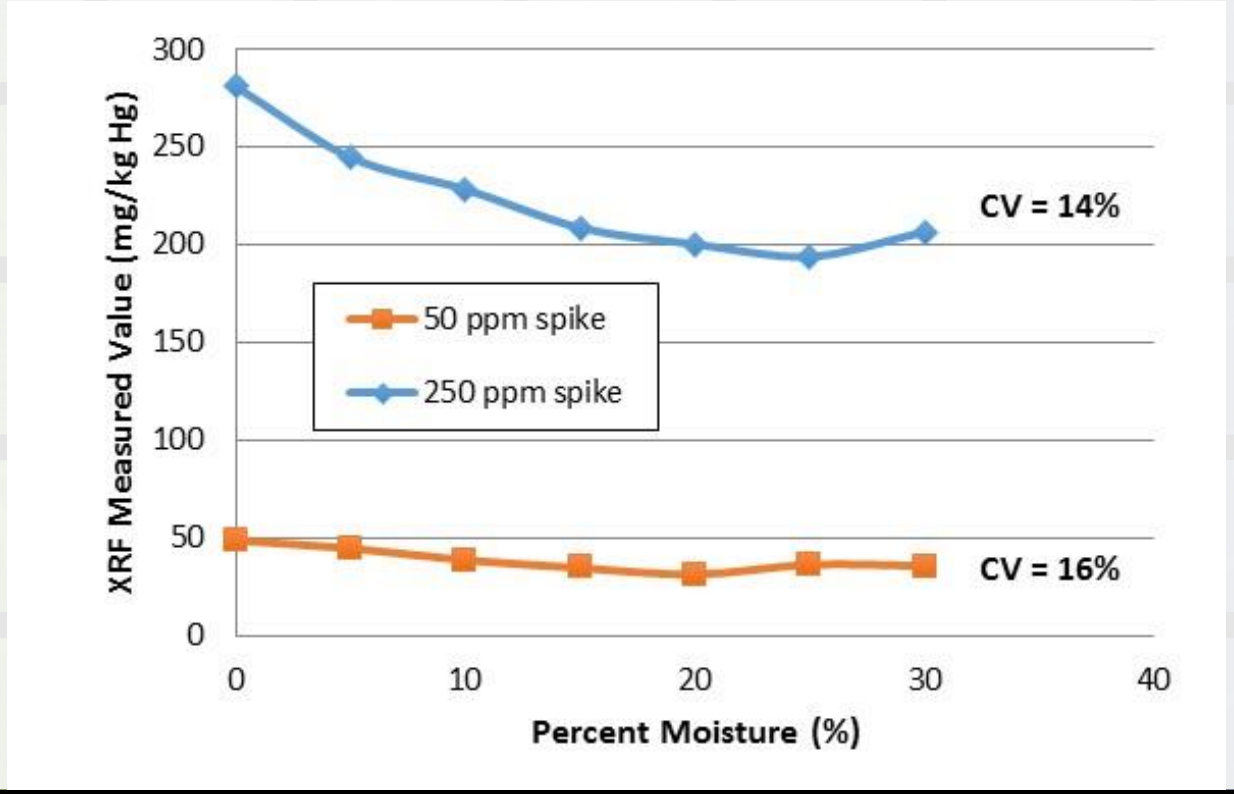
Hg-spiked samples

Moisture Study Design

- Water was gravimetrically added to spiked soil samples to achieve moisture of 0, 5, 10, 15, 20, 25, and 30%
- Samples analyzed for Hg using XRF

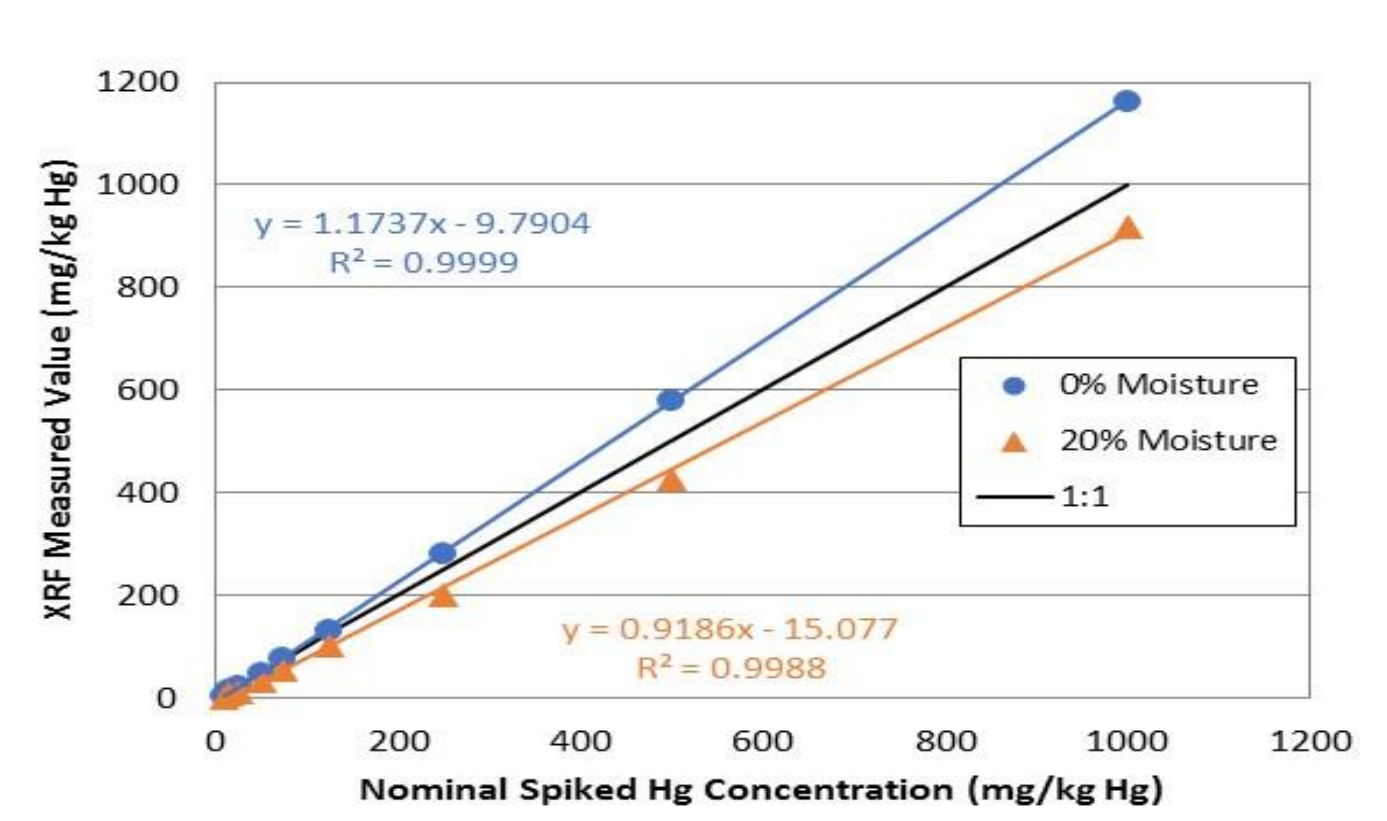
Moisture Addition to Spiked Samples

- Hg results decreased with increasing moisture, but variability was less than field triplicate variability (median = 18%)



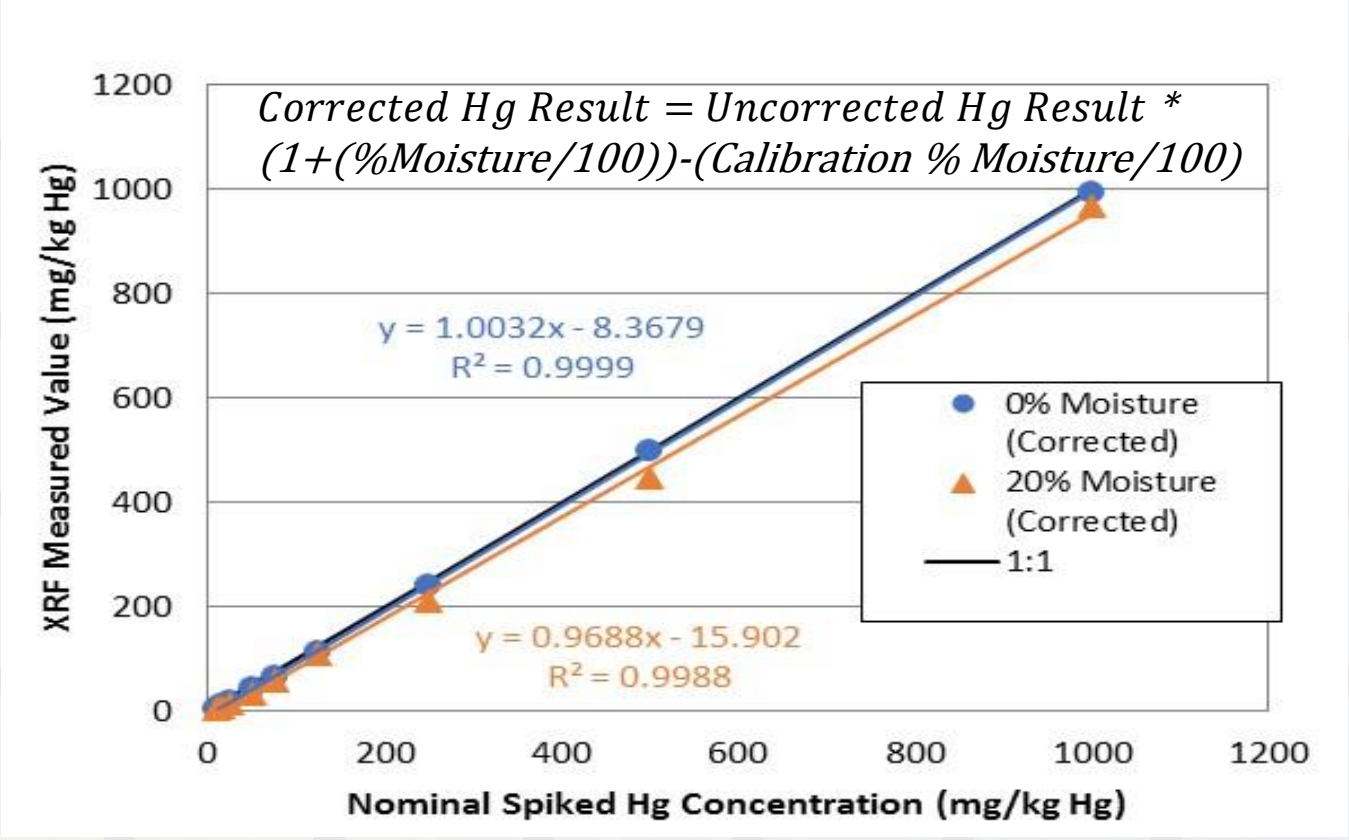
Moisture Addition Across Spiked Concentrations

- Results slightly biased high in dry samples, and biased low in wet samples



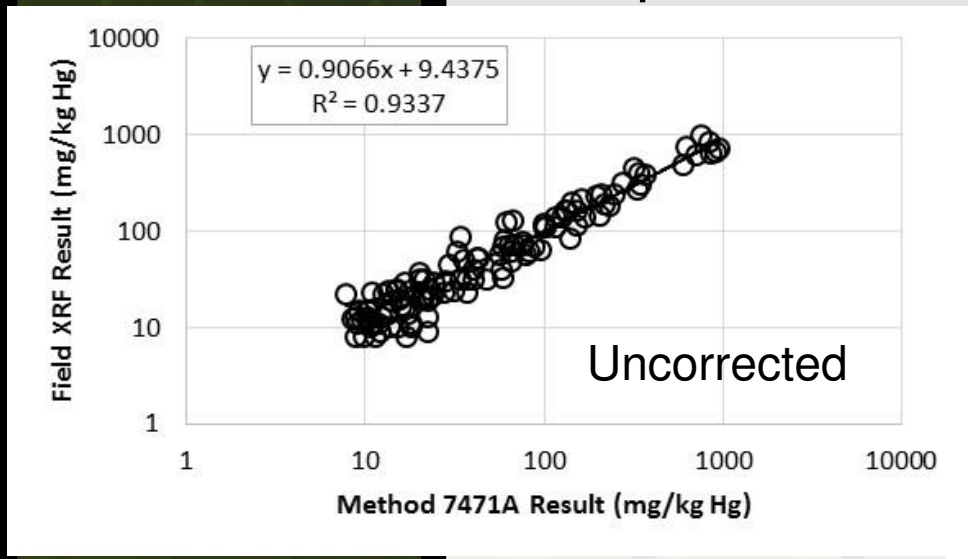
Moisture Correction of Spiked Samples

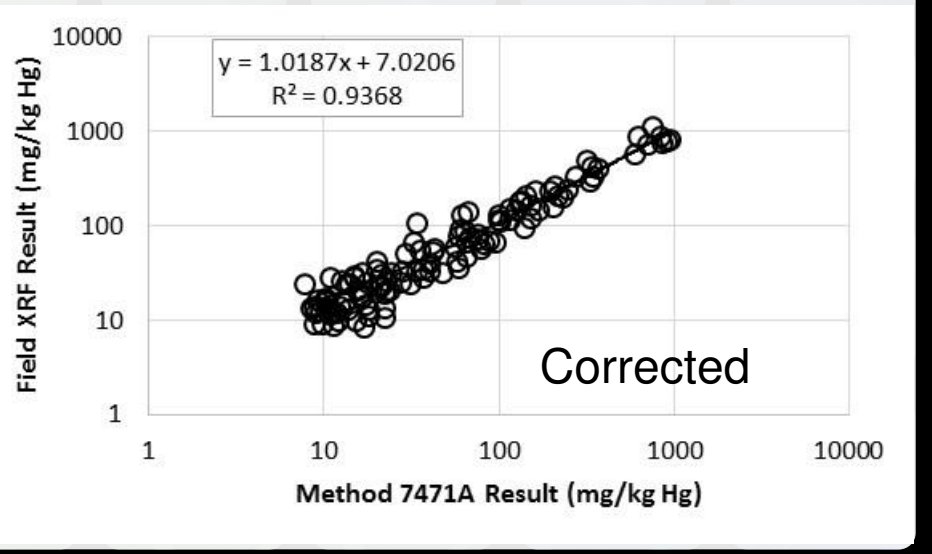
- If moisture is known, results can be corrected for moisture with a simple equation



Moisture Correction of Field Samples

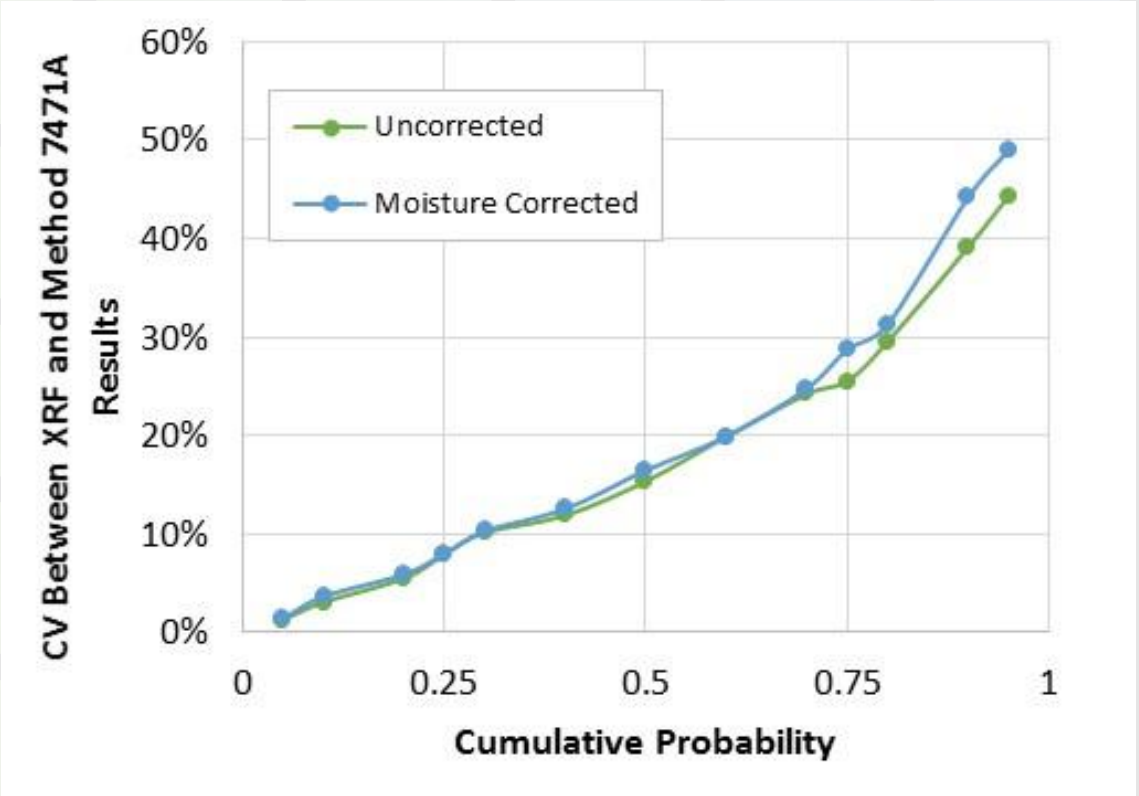
- Moisture correction can improve the overall slope of the fit across 236 samples.





Moisture Correction of Field Samples

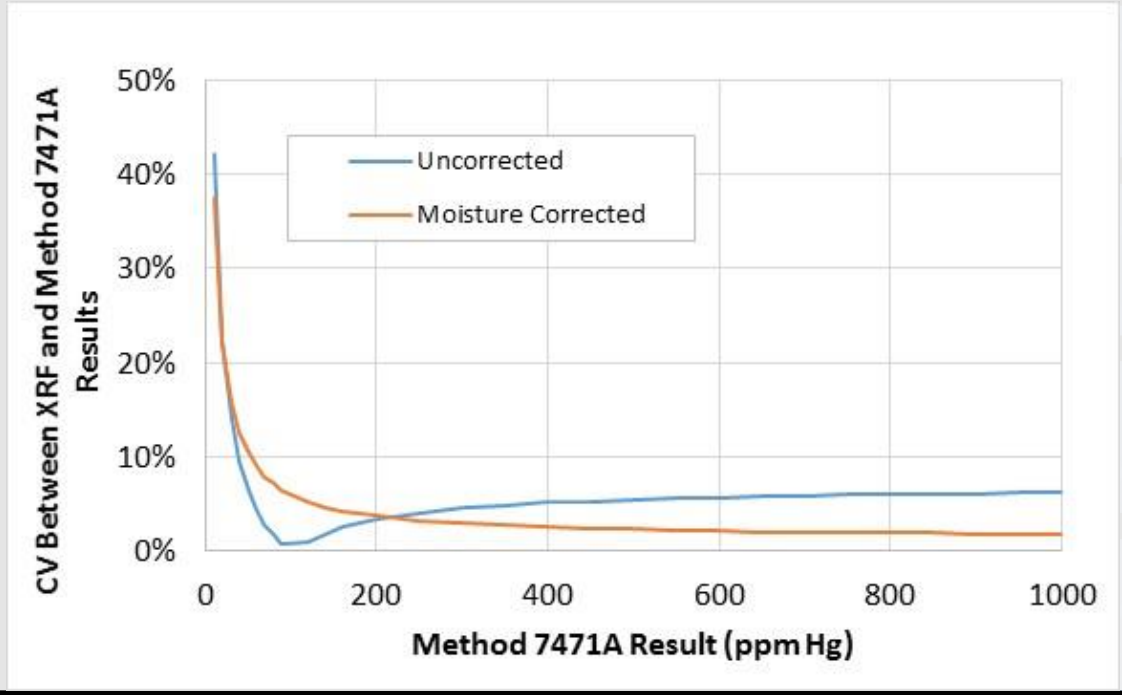
- But, moisture correction does not improve the fit of data on an individual sample basis



Why?

1. Variability associated with moisture is smaller than variability associated with sample heterogeneity

2. Benefits of moisture correction are mostly reserved for results at the upper end of the concentration range



How to Address Moisture

1. Calibrate with a moist soil that is similar in %moisture to the samples that you intend to measure (average of 14.53% in this study)

2. A moisture correction equation can be applied if moisture can be measured in the field

3. If calibration samples are representative of sample soil moisture, correction provides very minimal added value
   - Due to the relative magnitude of variability associated with soil sample heterogeneity
   - Due to decreased benefits at the lower end of the concentration range
   - Due to the questionable environmental relevance of dry-weight concentrations to start with

Discussion

1. What are pros and cons of utilizing XRF for routine floodplain and bank soil characterization (with some level of periodic laboratory validation ~10%)?

2. What applications would be more or less suited to the use of XRF?

3. Can we develop any consensus on moving forward with XRF field use?