



Development and Use of Customized Quality Control Materials for Large- Scale Monitoring Projects

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Introduction

- **Quality control data can be obtain from Standard Reference Materials (SRMs)**
 - **Can be cost prohibitive for large projects**
 - **SRMs often are only certified for a limited number of elements**
 - **May not contain all elements of interest above detection levels**
 - **SRMs will not necessarily be a good matrix match for actual samples**
 - **Important if mineralogy of samples different from SRM**

Project Specific Quality Control Materials (QCMs)

- Designed to have same or highly similar matrix as project samples
- May reveal analytical problems missed by traditional SRMs due to limited element data available
- Mineralogy an important consideration for sediments, soils, and rocks
 - Most analytical procedures performed by USGS result in **total** elemental concentrations
 - Typical 4-acid total digestion (HCl , HNO_3 , HF , HClO_4) for soils/rocks will not digest all mineral types:
 - Ba in barite (BaSO_4), Sn in cassiterite (SnO_2), Cr in chromite (FeCr_2O_4), Ti in rutile (TiO_2), Al in corundum (Al_2O_3), REEs in monazite ($\text{Ce, La, Nd, PrPO}_4$)

QCM Production

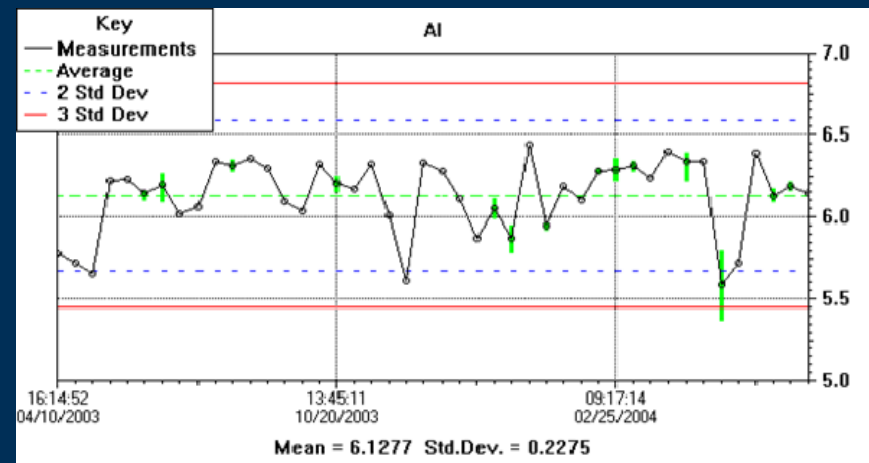
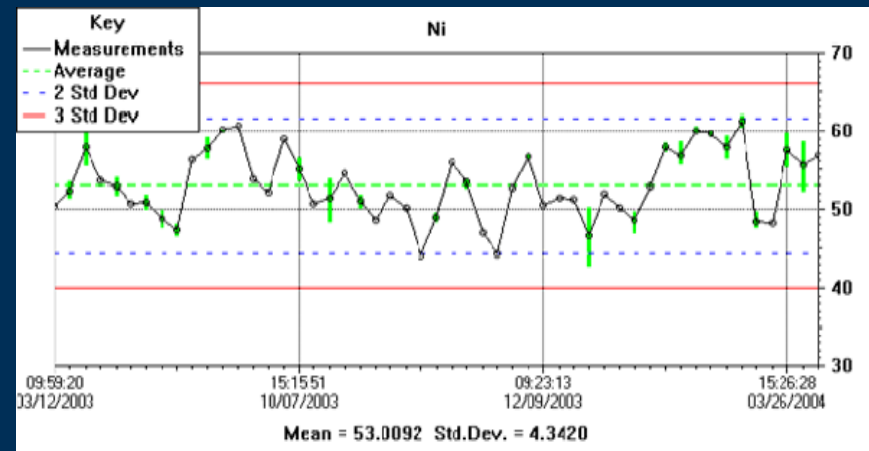
- **Identify necessary element and concentration coverage needed**
 - Typical USGS studies cover up to 86 elements by ICP-OES and ICP-MS
 - Can possibly use multiple QCMs to cover entire range
- **Alternative approach – Composite multiple subsamples of actual sample material collected**
 - More complete matrix match
 - Blend composited source material with other materials to achieve target element concentrations and mineralogical content
 - All elements at measurable concentrations

Example: USGS Contract Lab QCMs

- 60 Elements
- Base material: Sediments from Animas River (CO), Chatfield Reservoir (CO), DGPM-1 (Au ore), and Kesterson sediments (CA, for As and Se), also includes Te, Ti, Pt, Pd and W
 - SAR-L Target Concentrations 2-3X ICP-OES DL
 - SAR-M Target Concentration 10X ICP-OES DL
- Used to track laboratory performance
 - Randomized into every sample batch sent to contract lab for analysis
 - 12-Year performance history

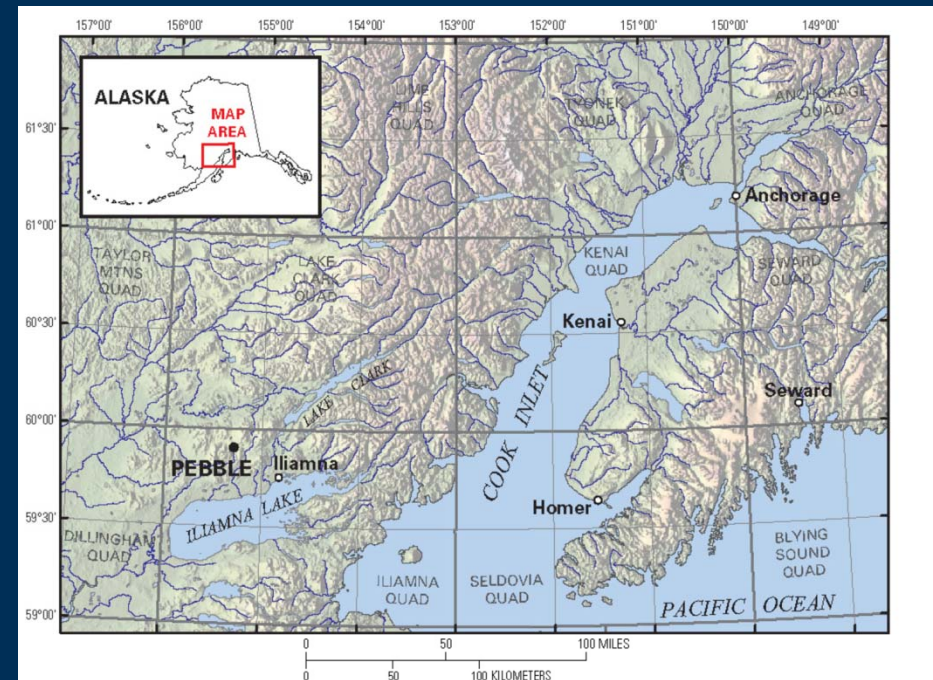
SAR-L and SAR-M Performance

- Results for QCMs compiled for each batch of data
- Data only accepted and entered into LIMS if QCM results acceptable
 - Unacceptable results will trigger redigestion and reanalysis



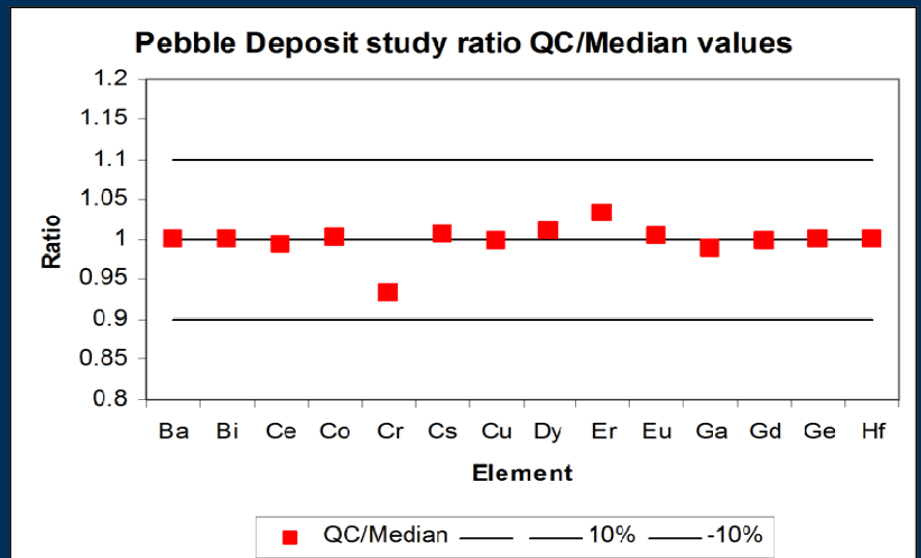
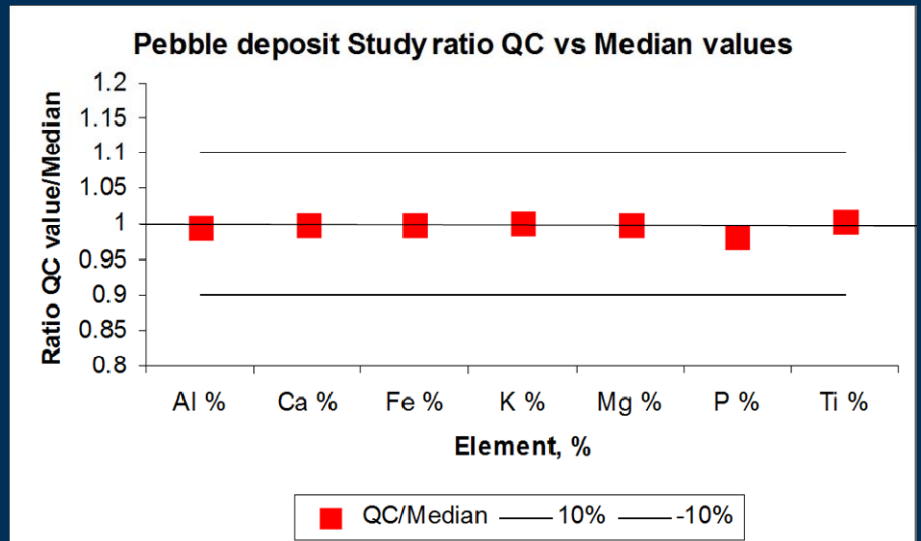
Regional QCM: Pebble Mine Deposit

- USGS regional study for baseline data of concealed Cu, Au, and Mo deposit area
- QCM required for laboratory performance evaluation
 - Existing USGS Reference Materials a poor matrix match
- Developed by compositing subsamples of all samples collected
 - ~ 100 pounds of QCM material needed for study duration



QCM Suitability testing

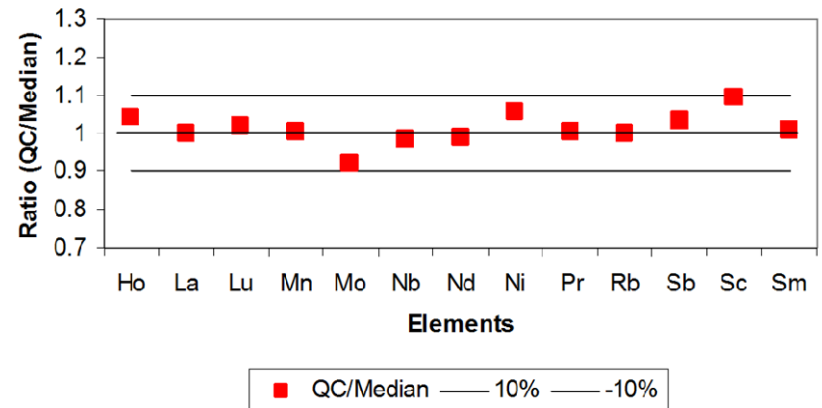
- Compare median element concentration to QCM element concentration
- QCM values determined by:
 - INAA, XRF, ICP-OES, ICP-MS, HG-AAS/AFS, etc.
- Perfect match when $[QCM]/[Median] = 1$
 - $\pm 10\%$ window for analytical precision



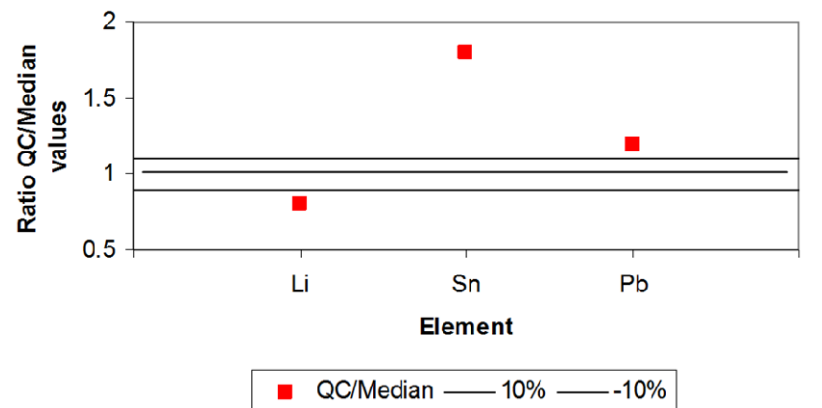
Pebble QCM Material

- Most elements show good agreement
- Li, Pb, and Sn show poor agreement
 - Li issues related to significant figures
 - Pb related to a single outlier skewing median
 - Results within $\pm 10\%$ with outlier removed
 - Sn related to mineralogy
 - Incomplete sample digestion or mineral type inhomogeneity across deposit

Pebble deposit study QC values vs Medians



Pebble deposit study ratio QC vs Median value



QCM for Continental Geochemical Study

- ~14,000 samples
 - 600 sample analysis batches over 4 years
- Large quantity of single QCM needed
 - 600 pounds
 - Monitor laboratory performance
- 42 elements by ICP-OES and ICP-MS
 - As, Se, Hg by HG-AAS

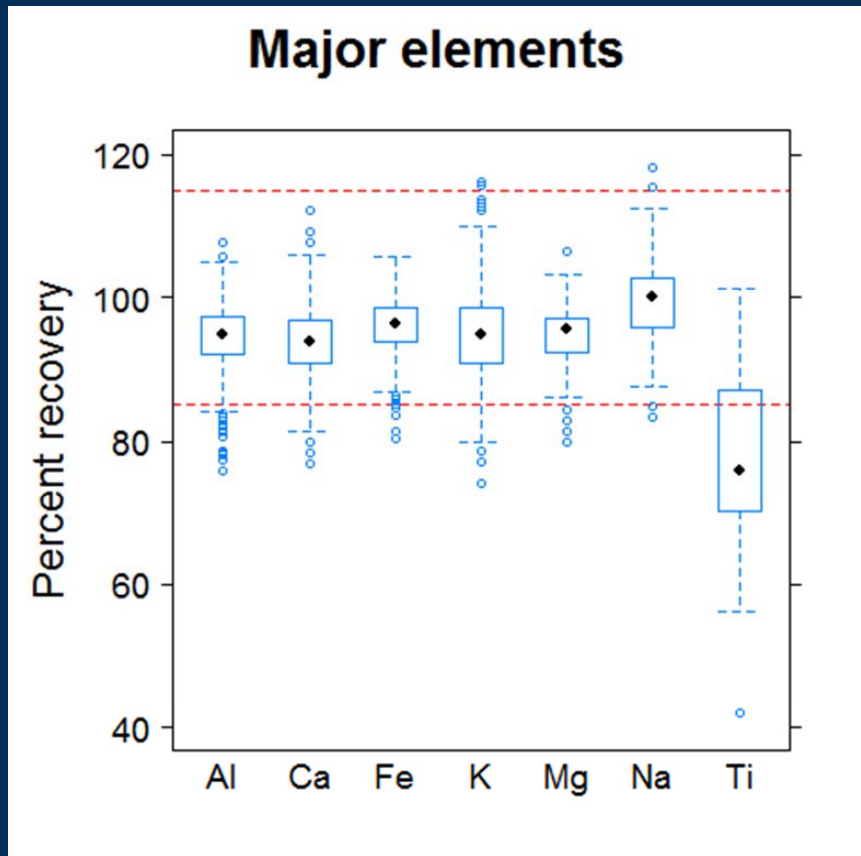


4,800 sites sampled from 2007-2010

**See Thursday's Session on North
American Geochemical Study for results
1:15 – 5pm in Regency ABC**

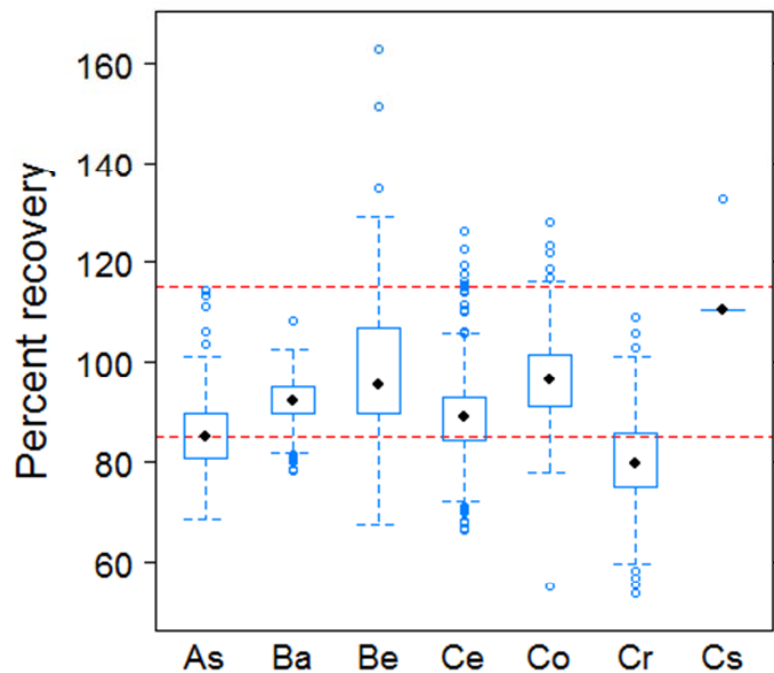
SoNE-1 QCM Soil Developed

- **Base material: Sharpsburg soil series**
 - Deep, well drained upland soil formed in loess
 - Collected in Lancaster County, NE
- **Used to assess laboratory precision and accuracy**
- **Use by Canadian, Mexican and U. S. geological surveys to assess data/method comparability**

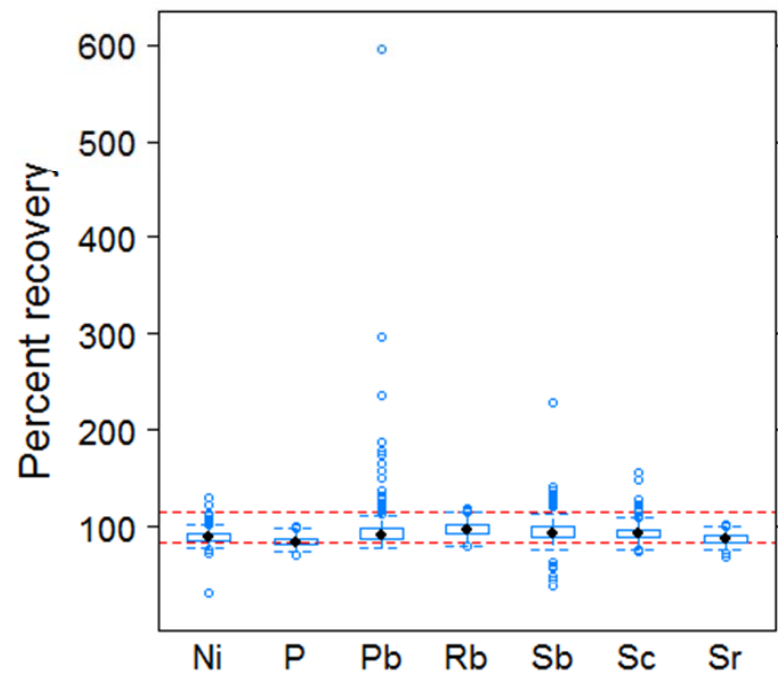


SoNE-1 Trace Element Performance

Trace elements



Trace elements



USGS Reference Materials Project

- Can provide consultative services for custom QCM development
- Can collect, grind, homogenize base material plus specific augmentations to meet requirements
- QCMs produced, bottled, and delivered to meet end use requirements
- Can also provide QCM concentration information from USGS in-house and contract laboratory analysis
 - INAA, XRF, ICP-OES, ICP-MS, etc.



History: USGS Reference Materials

- Began in 1951
 - Developed G-1 (granite) and W-1 (diabase) with MIT
- Over 42 standards developed
 - Natural matrix materials
 - Soils, sediments, silicate rocks, manganese nodules
 - Make 20 year supply
 - ½ - 1 ton of material
- Also develop standards for other organizations
 - NIST (2709, 2710, 2711, etc.)
 - NASA – simulated lunar soil
 - EPA – asbestos standard
 - Foreign government organizations
- Certification analyses done in-house and via round-robin



Steve Wilson preparing Cr VI soil for NIST

New Microanalytical Reference Materials

- Designed for direct solid analysis
 - Laser Ablation ICP-MS
 - SEM
- Concentrations obtained via international round-robin testing using microanalytical and bulk chemistry techniques
- Distributed in polished epoxy mounts to fit laser ablation holders



Contact Information

- **Websites:**

- http://minerals.cr.usgs.gov/geo_chem_stand/index.html
- http://minerals.cr.usgs.gov/icpms/reference_materials.html
- <http://pubs.usgs.gov/fs/2007/3056/>

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