

Chemical Analysis

Solids / Waters / Leachates

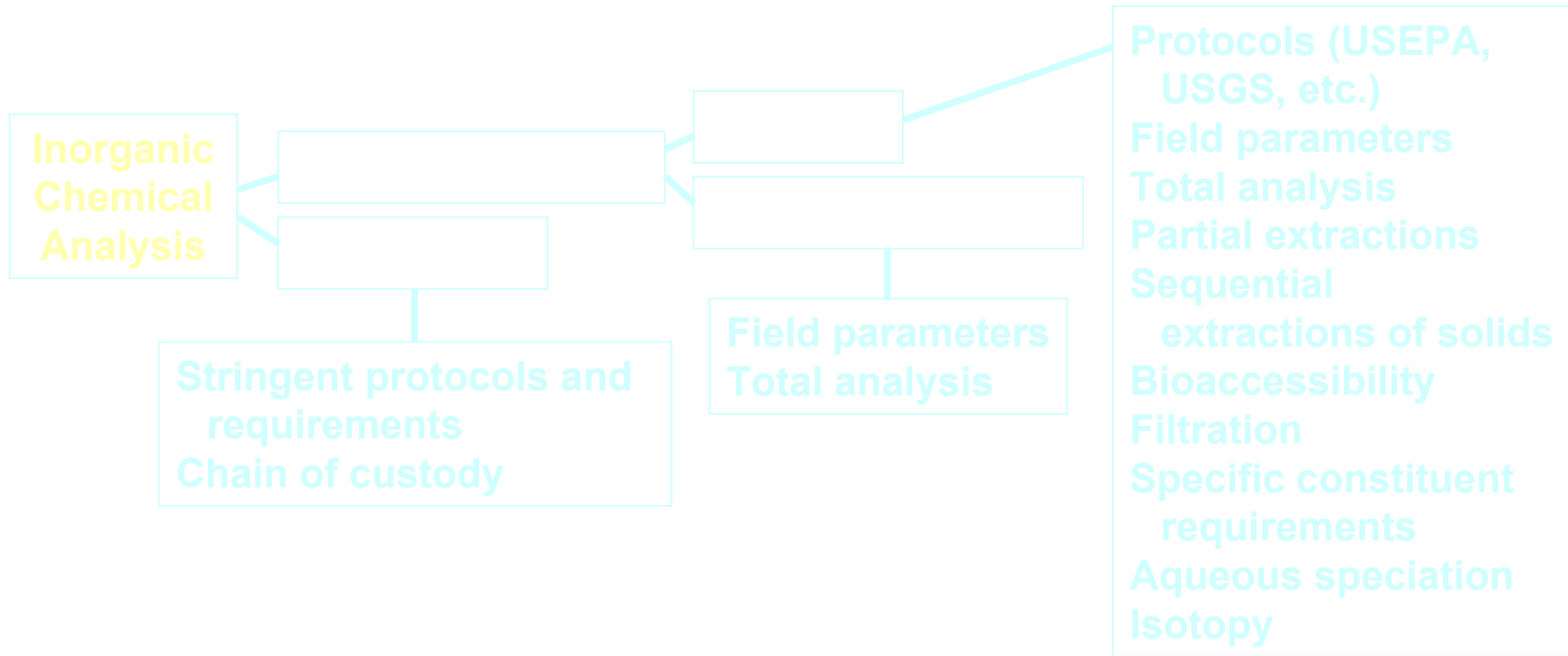
Kathleen S. Smith, USGS

Billings Symposium / ASMR Annual Meeting

**Assessing the Toxicity Potential
of Mine-Waste Piles Workshop**

June 1, 2003

Flow Chart for Ranking and Prioritization



Scale



Chemical Analysis of Mine-Waste Material

What is the question?

Need to choose appropriate methods for your needs

- Constituents of interest
- Precision
- Detection limits

Remember, analytical data are only as good as the sample

Chemical Analysis of Mine-Waste Material

- **Sample collection**
- **Sample preservation**

- **Sample preparation**
- **Laboratory subsampling**
- **Sample digestion / decomposition**
- **Analytical methods**
 - **Standard reference materials**
 - **QA/QC**
- **Sample archival**

Analytical Error

- **Sample preparation and processing**
- **Laboratory subsampling**
- **Digestion or extraction**
- **Analytical method**
- **Instrument calibration**

Sample Digestion

“Total” Digestion Methods

- **Acid(s)**
 - USGS methods
 - USEPA methods
 - Aqua regia ($\text{HNO}_3/\text{HCl}=1:3$; problem with silicates)
- **Fusion (lose an element)**

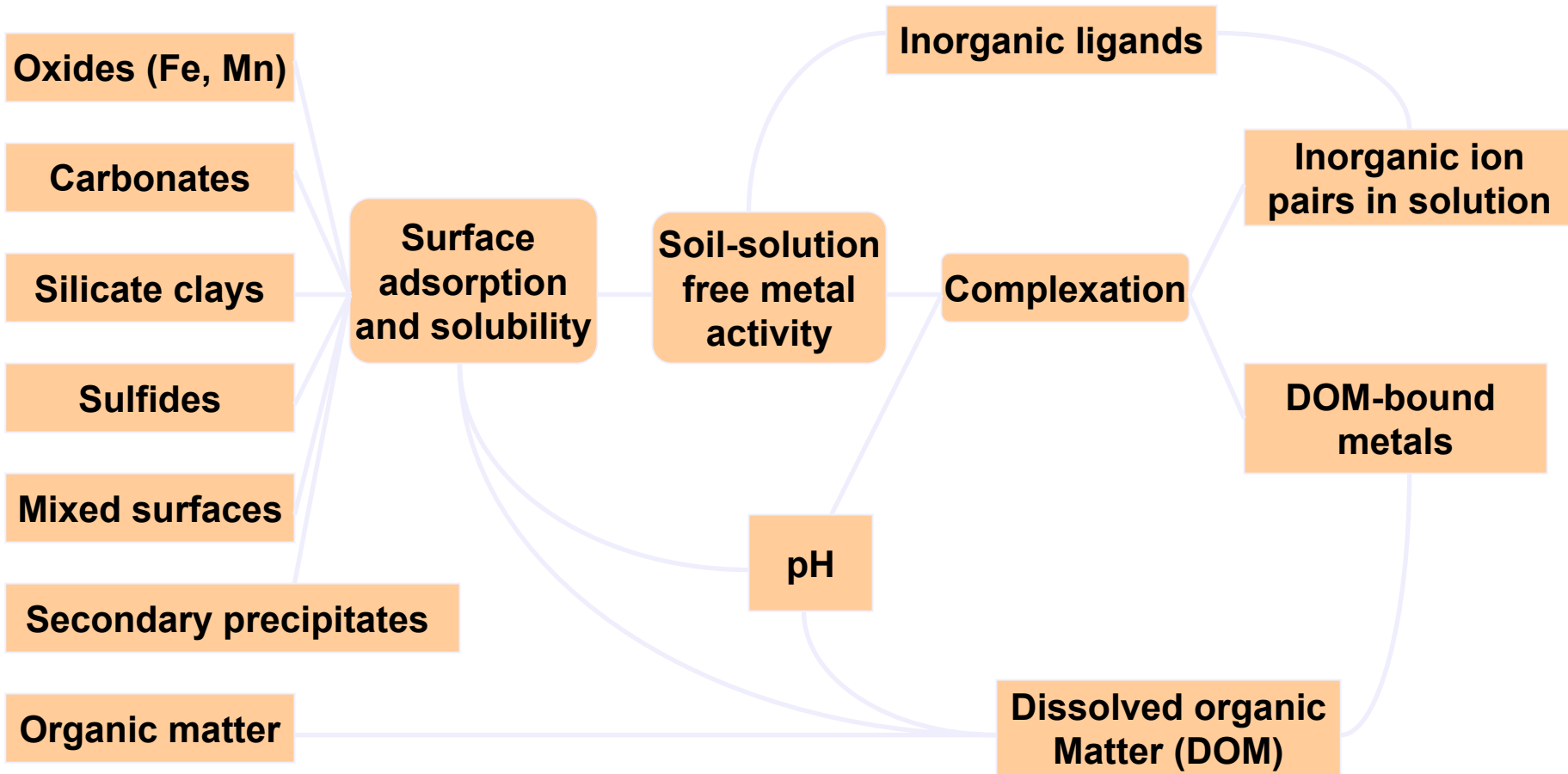
Partial Extraction Methods

- **USEPA methods**
- **Custom methods**
- **Sequential extraction methods**
 - **Operationally defined phases**

Metal Pools

Solid-phase fractionation

Solid-solution chemical speciation



Common Extraction Methods

- **Water**
- **Salt solutions**
 - e.g., CaCl_2 , MgCl_2 , NaNO_3 , NH_4NO_3
 - Simulate soil solutions; exchangeable
- **Chelating agents**
 - e.g., EDTA, DTPA
 - Potentially labile portion
- **Oxidizing or reducing agents**
- **Acids**
 - e.g., HCl, HNO_3 , aqua regia
 - Fairly rigorous treatment

Common Analytical Methods

- **Inductively coupled plasma-atomic emission spectroscopy (ICP-AES)**
- **Ion chromatography (IC)**
- **Inductively coupled plasma-mass spectrometry (ICP-MS)**
- **Atomic absorption spectroscopy (AAS)**
- **X-ray fluorescence (XRF)**
- **Instrumental neutron activation analysis (INAA)**
- **Laser ablation**

Common Waste-Rock Methods

- **Leaching methods**
 - **Static tests**
 - **Kinetic tests (incl. humidity cells)**
- **Acid-base accounting**
- **Mineralogy**
 - **X-ray diffraction**
 - **Petrographic studies**
- **Specific gravity**

Which Analytical Method?

- **Detection limits**
- **Precision requirements**
- **Constituent(s) of interest**
 - **Multiple or single**
(e.g., Hg requires special methods)
 - **Specific method necessary**
- **Freedom from interferences**
- **Established methods vs new methods**
- **Resources**

QA / QC

- **Standard Reference Materials (SRMs)**
 - NIST
 - USGS
 - Canada
- **Standard Operating Procedures (SOPs)**
- **Replicates**
- **Blanks**
- **Spiked samples**
- **Documentation**