Chemical Analysis

Solids / Waters / Leachates

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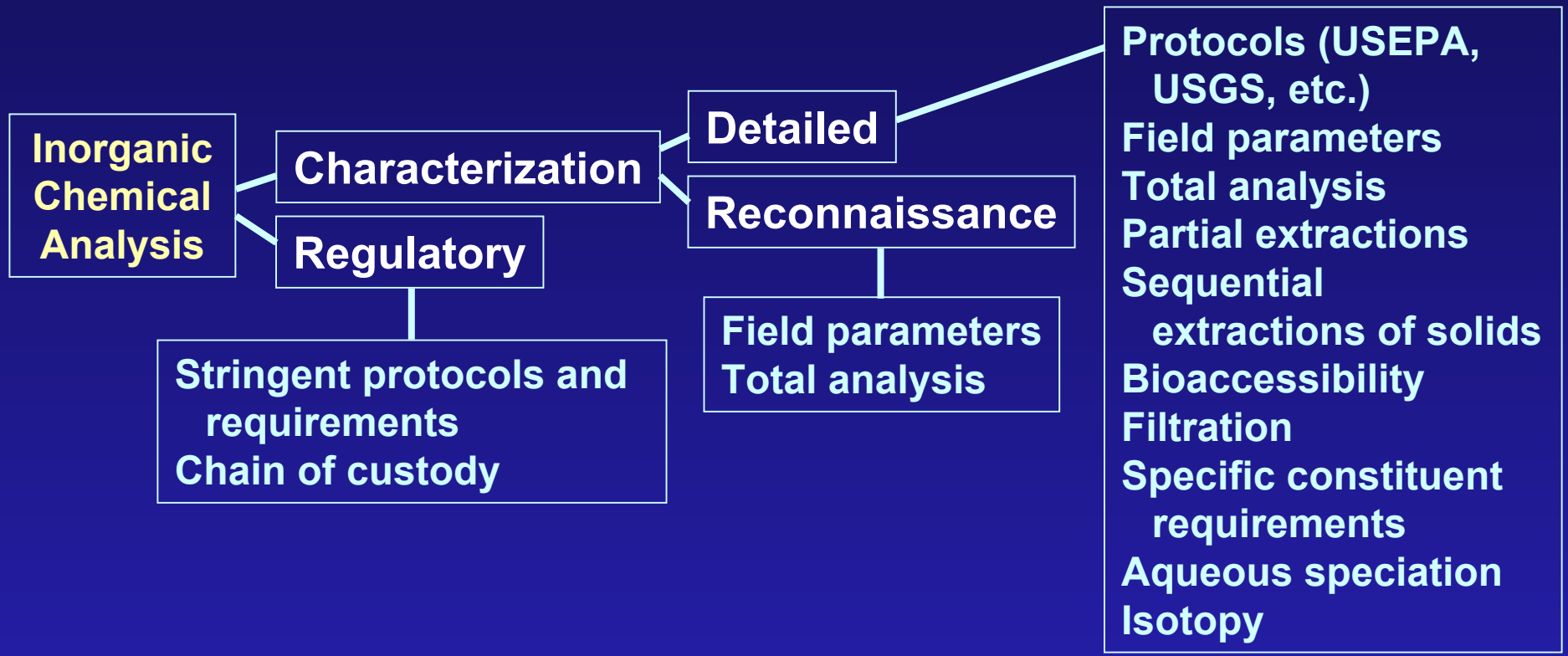
Billings Symposium / ASMR Annual Meeting

Assessing the Toxicity Potential
of Mine-Waste Piles Workshop

June 1, 2003

U.S. Department of the Interior
U.S. Geological Survey

Flow Chart for Ranking and Prioritization



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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

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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

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Analytical Error

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

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Sample Digestion

"Total" Digestion Methods

- Acid(s)
  - USGS methods
  - USEPA methods
  - Aqua regia (HNO_3/HCl=1:3; problem with silicates)
- Fusion (lose an element)

Partial Extraction Methods

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

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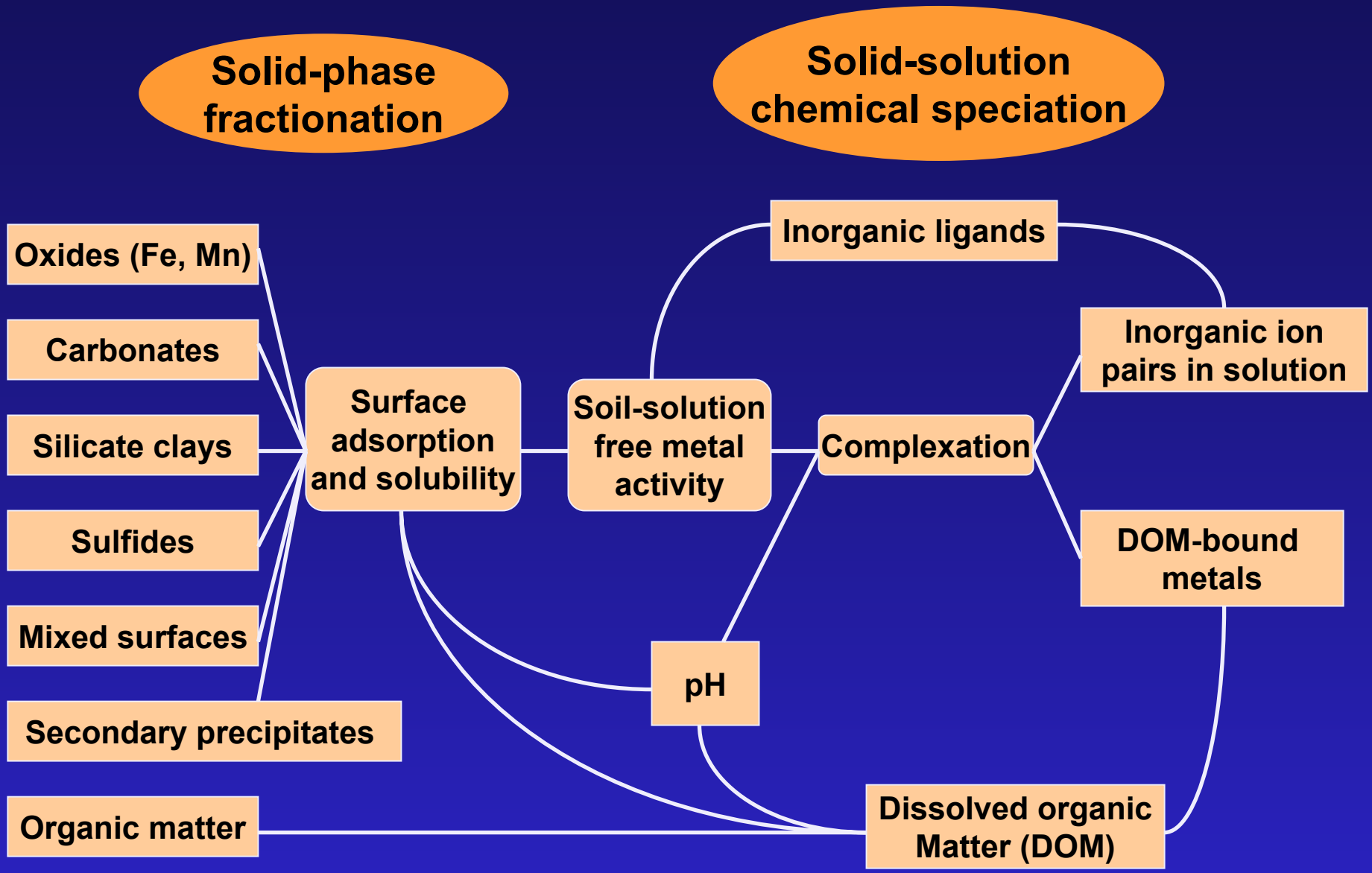
Metal Pools



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(Sauve, 2002)

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

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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

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Common Waste-Rock Methods

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

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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

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QA / QC

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

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