NMS

ASTM Standardization of Methods for Environmental Characterization of Metal Bearing Wastes and Ores

C. H. Bucknam, Newmont Metallurgical Services,
K. A. Lapakko, Minnesota DNR, and
W. W. White III, BLM Salt Lake Field Office

Presentation Overview

- Historical Review
- Humidity Cell Method D5744
- Carbon-Sulfur Methods E1915
- Water Leaching Methods
  - Synthetic Precipitation Leaching Procedure (SPLP) D6234
  - Meteoric Water Mobility Procedure (MWMP) E2242
- Future Plans – Acid Neutralization Potential Acidity (ANPA) Titration and Ore/Metals Environmental Testing (O/MET) Proficiency Testing Program (PTP)

Historical Review of Standards

- American Society for Testing and Materials (ASTM) International Committees
  - E01 on Analytical Chemistry of Metals, Ores and Related Materials
    - Merged Committees E02 on Spectroscopy, E03 on Metals and E16 on Metal Bearing Ores and Related Materials
  - D34 on Waste Management
- US Bureau of Mines Humidity Cells
- Society of Mineral Analysts Methods Committee Review of Carbon-Sulfur Methods
- Existing Methods Were Developed For Soil and Coal Overburden

Humidity Cell Test (HCT) Method

- ASTM D5744 Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell
- Protocol Designed to Assess Relative Potential of Mine-Waste Samples to Produce Problematic Drainage - Does Not Simulate Field Conditions
- Protocol Provides Laboratory Conditions Conducive to Oxidation of Sample Constituents & Enhances Transport of Weathering-Reaction Products
- Enables Measurement of Weathering-Product Mass Release
- Method Can Be Used for Meeting Regulatory Requirements for Kinetic Testing

HCT Method Overview

- 1-kg Test Charges of Well Characterized -6.3-mm (-1/4”) Material
- Cylindrical Cell 10.2-cm ID 20.3-cm H
- Weekly Cycle (20 Week Minimum)
  - Week 0 First Leach with 0.5-L or 1-L Flood or Drip-Leach (Two to Three Hours Duration)
  - Next Day Conductivity, Eh, pH, Alkalinity, Acidity, Preserve for Metals and Anion Analyses
  - Three Days Dry Air 1-10 L/min
  - Three Days Wet Air Humidifier 30^{o}C
  - Leach Ends Weekly Cycle
  - Track Weights at Each Stage of Weekly Cycle

Interpretation of HCT Test Results

- Calculate Loading of Sulfate, Alkalinity, Acidity, etc., From Effluent Volume and Concentrations
- Plot Assays, Loads and Cumulative Loads with Respect to Time (Weeks)
- Identify Inflection Points on Cumulative Plots, Use Slopes Between Points to Calculate Release Rates, µg/g/week

Within Laboratory Repeatability

(see White and Lapakko 2000 for details)

Weeks	Replicate 1, Sulfate µg/g/week	Replicate 2, Sulfate µg/g/week
0-20	523	559
20-40	887	868
40-60	1986	1481
60-80	1835	1292
80-100	1783	1248
100-120	3165	1661

HCT Method Revision Underway

- Method B Proposed Revision
  - Controlled Temperature and Humidity Room
  - Lower Cost and Simplified Method
  - Extensive Comments Being Addressed
- Between Laboratory Reproducibility Needed
  - 8 Volunteer Labs Needed
  - ASTM Provides Administrative Support for Interlaboratory Studies, but Participation is Voluntary
  - Funding is Needed To Support Some Possible Participating Labs

ADTI-CMS Column Method Development

- Acid Drainage Technology Initiative Coal Mining Sector (ADTI-CMS) Column Methods Under Development With Funding by United States Environmental Protection Agency (USEPA) and Office of Surface Mining (OSM)

Carbon-Sulfur Methods

- ASTM E1915 Standard Test Methods for Analysis of Metal Bearing Ores and Related Materials by Combustion Infrared Absorption Spectrometry
- Acid-Base Classification Based on Sulfide Sulfur and Carbonate Carbon
- Same Methods Used for Metallurgical Characterization of Non-Ferrous Ores

Carbonate Carbon Estimation

- Total Carbon
  - **Contains No Organic Carbon Nor Graphite**
- Hydrochloric Acid Insoluble Carbon
  - **Isolates Organic Carbon and Graphite**
  - **Carbonate Carbon Estimated By Difference**
- Pyrolysis Residual Carbon
  - **550^{o}C Drives Off Organic Carbon and Graphite**
  - **Some Carbonate Carbon May Be Driven Off**
  - **Tends to Be Lower Estimate**
- Acid Neutralization Potential (ANP) = 8.33 Carbonate Carbon, % $CaCO_3$

Sulfide Sulfur Estimates

- Total Sulfur
  - No Sulfate Present
- Pyrolysis Residual Sulfur
  - Sulfides Are Driven Off At 550^{o}C
  - Sulfur May Be Adsorbed By Carbonates
  - Metal Sulfides May Require Higher Temperatures
  - Sulfide Estimated by Difference

Sulfide Sulfur Estimates- Continued

- Hydrochloric Acid Insoluble Sulfur
  - Determined With Insoluble Carbon
  - Pyrhottite Will Be Lost
  - Isolates Pyrite
- Nitric Acid Insoluble Sulfur
  - USEPA Pyritic Sulfur By Difference
    - **Six Hour Leach Ineffective**
    - **Samples Must Be Boiled With Nitric Acid**

Sulfide Sulfur Estimates- Continued

- Sodium Carbonate Insoluble Sulfur
  - Sulfate Minerals Are Dissolved
  - Orpiment and Realgar Are Dissolved (Sulfides)
  - Alunite and Jarosite May Not Dissolve (Sulfates)
- Acid Generation Potential (AGP) = -3.1 Sulfide Sulfur,% $CaCO_3$

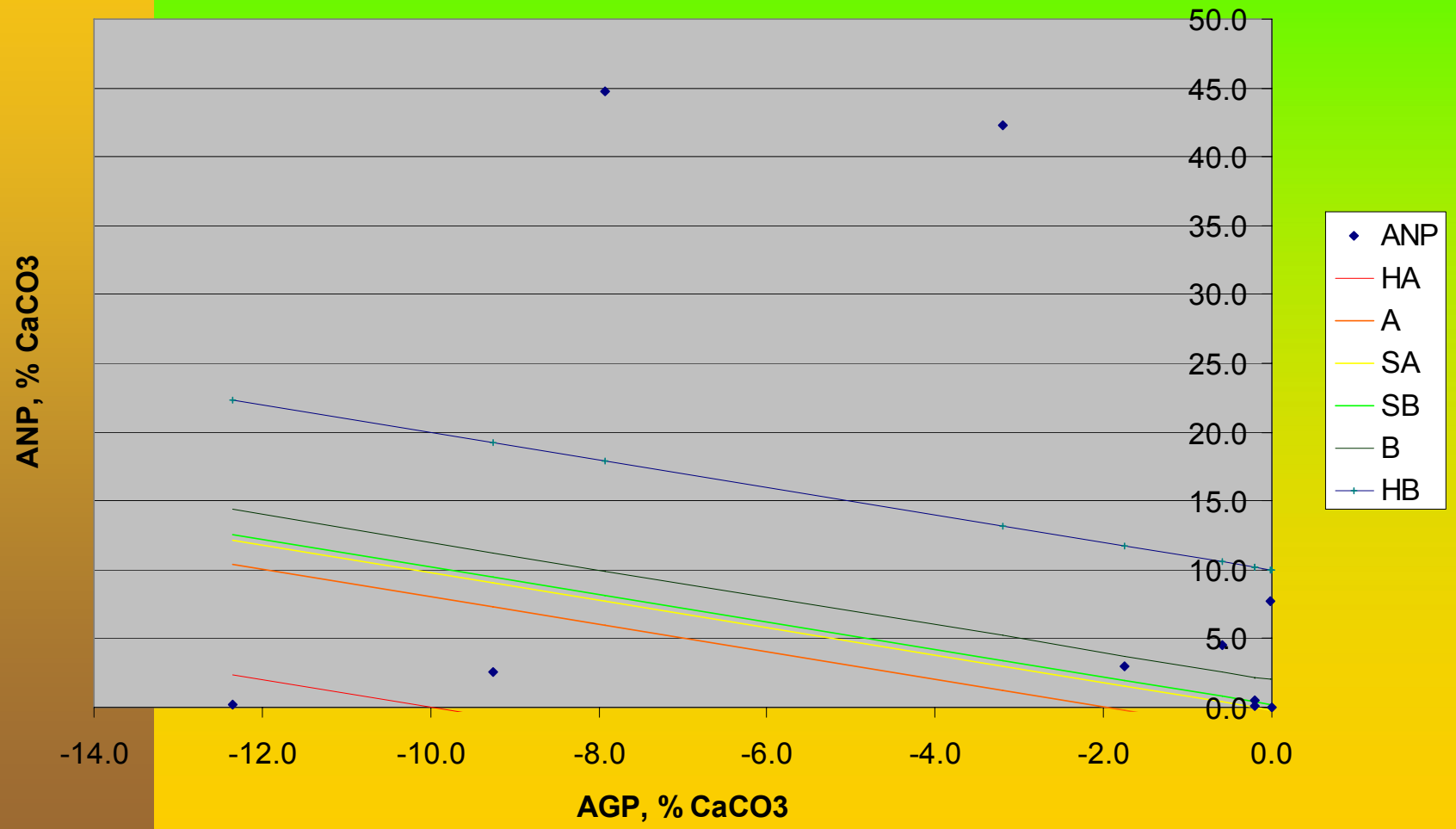
Net Calcium Carbonate

- Correct Positive AGP and Negative ANP Results to Zero
- Net Carbonate Carbon (NCC) = ANP + AGP, % $CaCO_3$
- Classify Material According to Geology and NCC
- Perform NCC Confirmation
  - **Mineralogy**
  - **Other Carbonate and Sulfide Estimates**
  - **Biological Acid Production Potential (BAPP), Humidity Cell Tests (HCTs) and Site Columns**
  - **SPLP or MWMP**
  - **Toxic Release Inventory (TRI) Characterization to Lowest Detection Limits**

NCC Classifications

Highly Acidic	NCC $\leq$ -10
Acidic	-10 < NCC $\leq$ -2
Slightly Acidic	-2 < NCC < -0.2
Neutral/Inert	-0.2 < NCC < 0.2
Slightly Basic	0.2 $\leq$ NCC < 2
Basic	2 $\leq$ NCC < 10
Highly Basic	10 $\leq$ NCC

NCC Classification Plot For Interlaboratory Studies (ILS)



SPLP Standard Method

- ASTM D6234 Test Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure
- Method Extracted from Sequential Batch Extraction Method For one Material
- Simplified Version of USEPA 1312, No Zero Headspace Extraction nor Filtration
- Only One Mine Waste Sample Tested in ILS
- Similar to USEPA 1311, Toxicity Characteristic Leaching Procedure (TCLP)

SPLP Method Overview

- Crush Sample To Pass 9-mm Sieve
- 100-g Sample Charge, Dry Basis
- 2-L Water, pH May Be Adjusted For Acid Rain Nitric/Sulfuric Acid Dropwise
- 16 Hours End-Over-End Mixing
- Filter and Preserve for Water Analyses
- Immediate pH

SPLP ILS Results For Mine Waste

Metal	Mean, µg/mL	Reproducibility Index (R), µg/mL	R, %
Ba	1.9	0.8	42
Ca	1815	666	37
Mg	107	34	32
Mn	37	15	42
Pb	63	22	35
Si	55	58	105*
Zn	297	98	33

* R > 50% Non-Quantitative

MWMP Standard Method

- E 2242 Test Method for Column Percolation Extraction of Mine Rock by the Meteoric Water Mobility Procedure
- Developed by State of Nevada
- Column Test Method For Coarse Rock
- Single Pass of Solution Through Rock
- Uses Deionized Water

MWMP Method Overview

- Crush Sample To Pass 50-mm (2”) Sieve
- 5000-g Sample Charge, Dry Basis
- 5-L Deionized Water
- 24 Hours Peristaltic Pump
- Filter and Preserve for Water Analyses
- Limited By Percolation Rate
- Bottle Roll and Filtration Options Under Development

MWMP ILS Hard Rock Mine Waste

Metal	Mean, µg/mL	R, µg/mL	R, %
Ba	0.025	0.021	84*
Mg	18	7.5	42
Mn	3.8	1.7	43
Pb	2.7	0.73	27
Zn	22	10	47

* R > 50% Non-Quantitative

MWMP Results For Heap Spoils

Metal	Mean, µg/mL	R, µg/mL	R, %
Ba	0.041	0.085	209*
Mg	4.5	1.5	28

* R > 50% Non-Quantitative

Proposed ANPA Automatic Titration

- Uses Low Fizz Sobek Conditions
- Sulfuric Replaces Hydrochloric Acid
- pH > 4 Selects Higher Acid Range
- Filtrate Reacted With H_2O_2 To Fully Oxidize Metals
- Boil to Remove Excess Peroxide
- Back Titrate With Standard Base
- Report Negative Results for Acidity Present
- Includes Calibration Mixtures ($CaCO_3$ and $FeSO_4$) to Check Titrator Set-up

ILS Pilot Program Study

- Committee E01 Test Plan
- 13 Participating Laboratories
- 12 Reference Materials Donated
- Contractor To Distribute Samples
- ASTM to Compile Results, Statistics and Research Report
- Section E01.02.05 To Review and Ballot

ASTM Proficiency Testing Program

- Approved by Committee E01
- O/MET = Ores and Metals Environmental Testing
- Planned to Start With 30 Labs, Twice/Year
  - Two Materials (~300-kg)
    - Neutral to Highly Acidic
    - Inert to Highly Basic
- Delayed Reporting for Kinetic Tests

Material Distributed by USGS

- 250-g @ -100 mesh
- 1-kg @ -6.3-mm
- 2 x 100-g @ -9-mm
- 5-Kg @ -50-mm
- Carbon-Sulfur, BAPP and ANPA
- Humidity Cells
- SPLP and TCLP
- MWMP

ASTM Report to O/MET Labs

- 6 Weeks To Report Results On-Line
  - BAPP and HCT Report 6 Month Delayed
- Youden Plots for Two Materials
- Labs Identified By Number
  - Labs Can Disclose Identification Number to Auditors
- Reports Downloaded in Electronic Format
- Complies With Certification Requirements
- Section E01.02.05 To Review Program

NMS

7th ICARD

7th International Conference on Acid Rock Drainage

St. Louis Missouri, March 26-30, 2006

http://www.smenet.org/meetings/AnnualMeeting2006