# ASTM D5744 Kinetic Test Method Status and Application

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# ASTM D5744-13: Standard test method for laboratory weathering of solid materials using a humidity cell

- Kinetic test
  - Laboratory mine waste dissolution test
  - Mine waste drainage quality prediction
- Status
  - Most recent modification in 2013
- Application
  - Mine waste management

# OUTLINE

1. Mine waste characterization introduction
2. ASTM D5744 development, rigor
3. ASTM D5744 method description
4. Kinetic test application
   - Reactivity
   - Rates of solute release

Note: Focus on waste rock

# (1) MINE WASTE CHARACTERIZATION
## Introduction

- Solid-phase characterization
  - Ore deposit type (geoenvironmental modeling)
  - Visual examination (drill core logging)
  - Chemical analysis
  - Metal partitioning
  - Mineralogical/petrological analysis
  - Static tests (acid and neutralization potentials)

# (1) MINE WASTE CHARACTERIZATION Introduction

Drainage quality = f(solid-phase characteristics)

- Short-term dissolution tests (soluble salts)
- ***Kinetic tests (long-term dissolution tests)***
  - Soluble salts, other mineral dissolution
- Geochemical modeling

# (2) ASTM D5744 Development

WHAT?

- Detailed laboratory weathering test method

  Kinetic test for waste rock

  8 drafts from 1992-1996
- Reviewed in formal ASTM process

  Practitioners, industry, academia
- Rigorous ruggedness testing
- Revisions 2007, 2013

# (2) ASTM D5744 Development Ruggedness testing

- Intralaboratory and interlaboratory testing

- 68 samples from 6 different rock types
  - Characterized: p. size, chemistry, mineralogy
  - Comparative testing for 20 -265 weeks

- Results in 111 p. research report w/method

# (2) ASTM D5744 Development

WHAT?

Formalized, robust waste rock dissolution test method

WHY?

- Provide detailed description of protocol
  - Based on existing methods (Lawrence 1990)
  - Provide guidance for new practitioners
  - Promote method consistency
  - Increase reproducibility of results

# (2) ASTM D5744 Development

WHAT?

Formalized, robust waste rock dissolution test method

WHY?

- Provide detailed description of protocol
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  - Promote method consistency
  - Increase reproducibility of results

WHO?

Bill White (USBM, USBLM) retired

# (2) ASTM D5744 Development

## Waste Rock

*OBJECTIVES*

1) Determine relative degrees of reactivity

2) Provide rates for modeling solute release in field

Does NOT simulate field drainage.

# (3) ASTM D5744 Description
## Start to Finish

3.1 Rock sample to be tested

3.2 Laboratory test protocol

3.3 Post-test rock analysis

# (3.1) ASTM D5744
## Rock Sample to be Tested

- Collection: methods, amount
- Storage
- Preparation
  - Drying
  - Screening
  - Size reduction
  - Splitting
- Analysis

# (3.1) Sample Preparation Crushing

# (3.1) Sample Preparation
# Sieving -1/4 inch

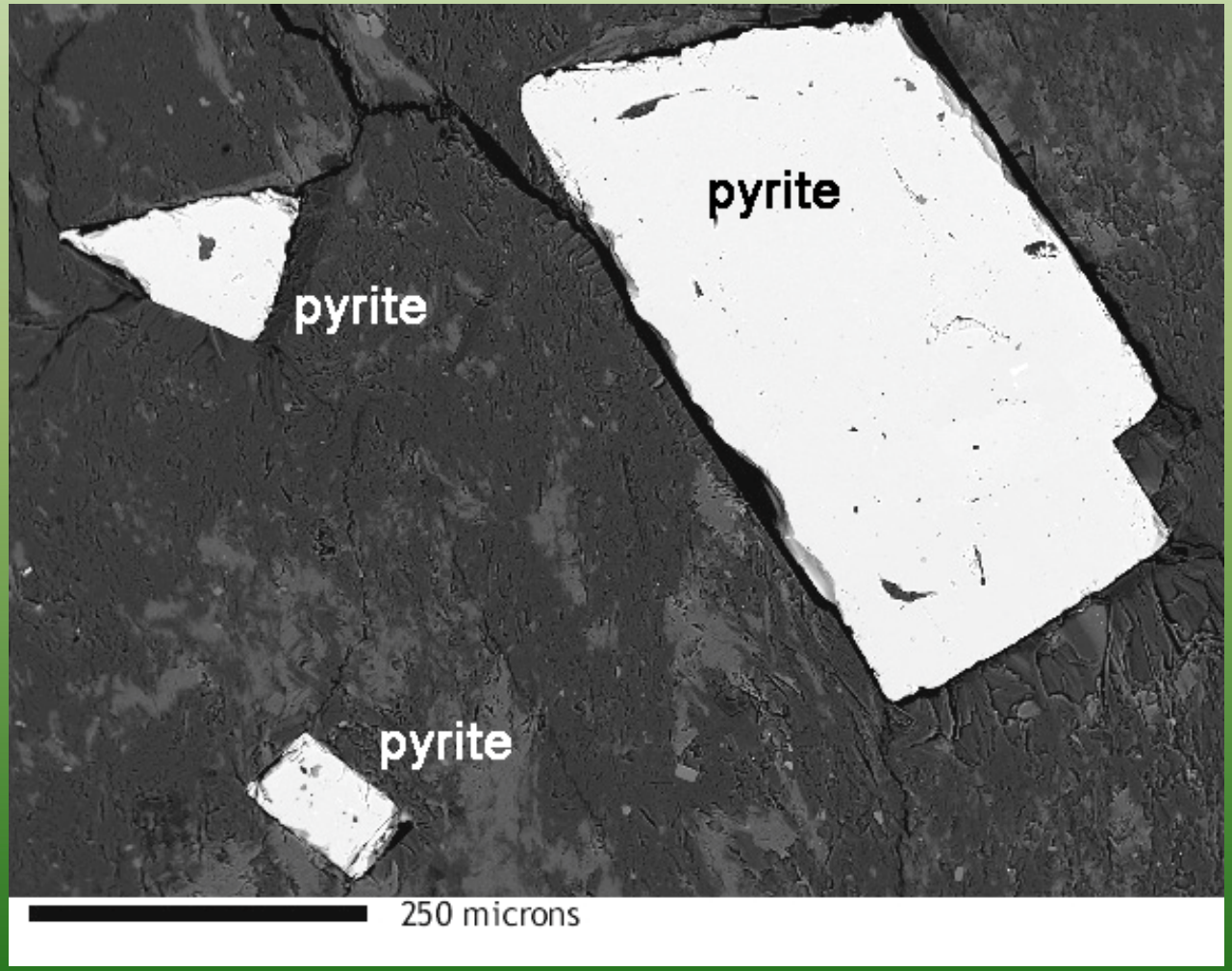
# (3.1) Sample Preparation Splitting

# (3.1) Sample Preparation
# Crushed splits for analysis

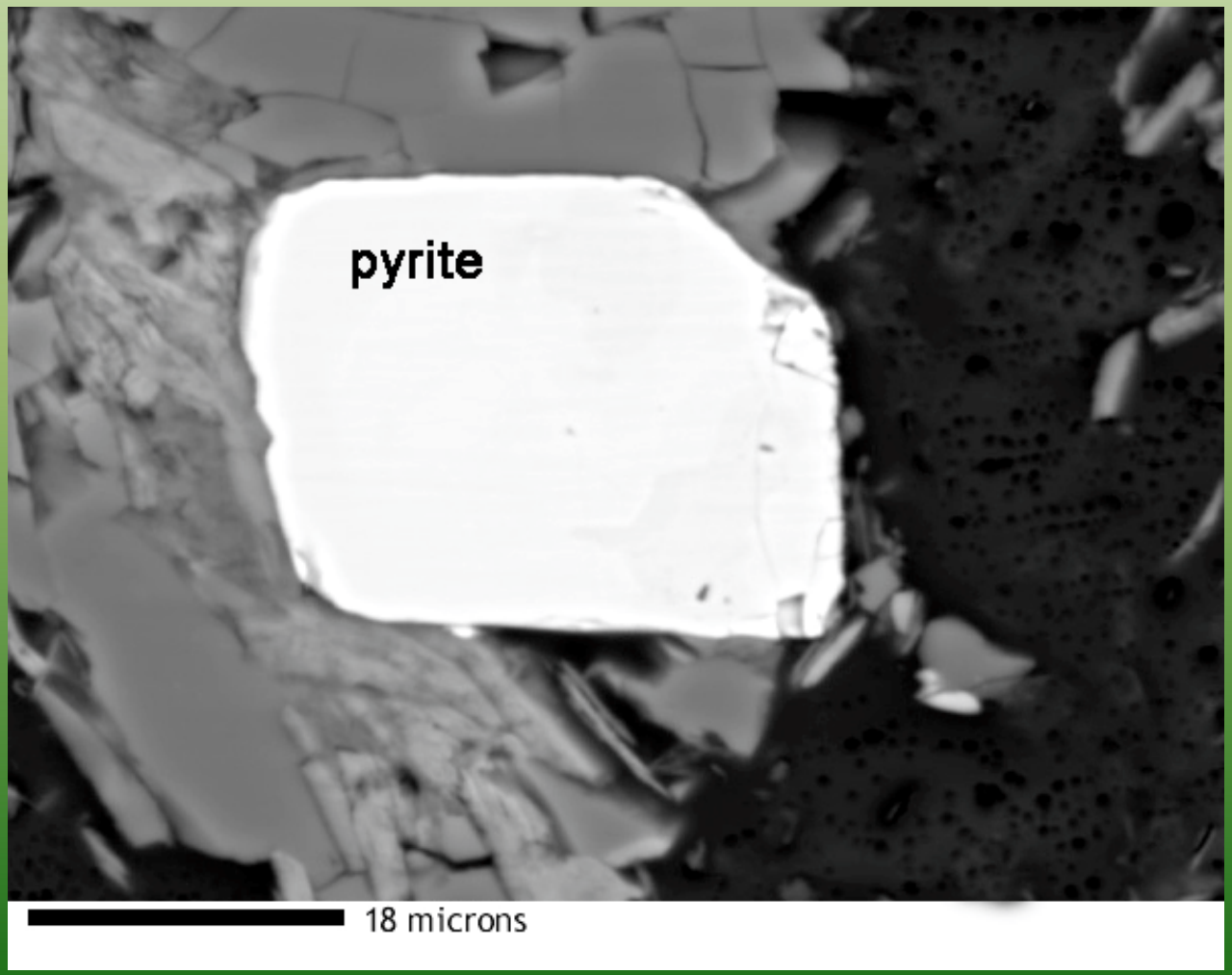
# (3.1) Rock Sample Analysis

- Particle size distribution
- Chemistry
  - S, $S^{2-}$, $CO_3$, whole rock, trace element
- Mineral content & chemistry (e.g. carbonates)
- Size fractions
  - S, $S^{2-}$, $CO_3$
  - $CO_3$
  - AP, NP mineral liberation

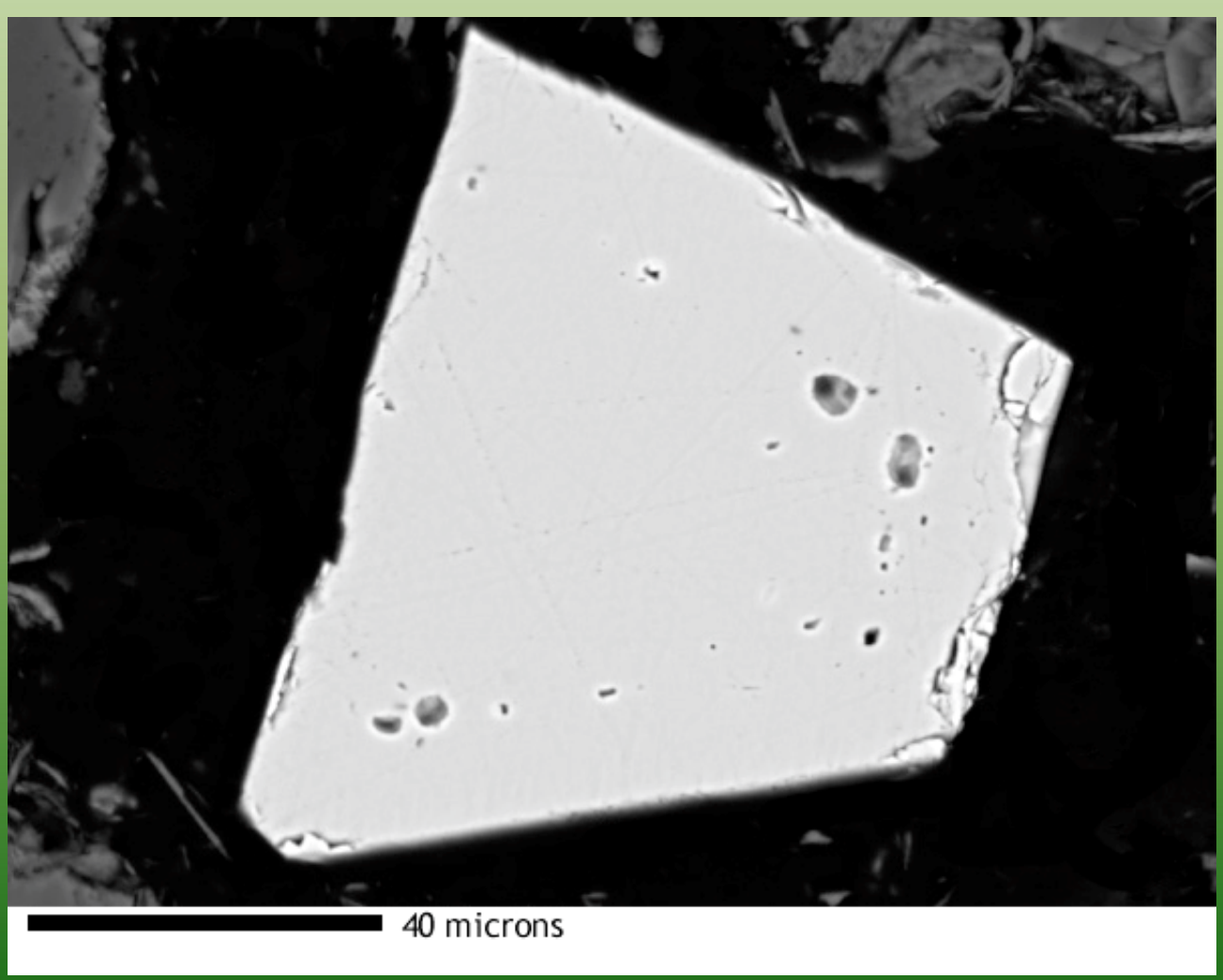
# (3.1) Pyrite included:
# +2000 µm Archean greenstone rock



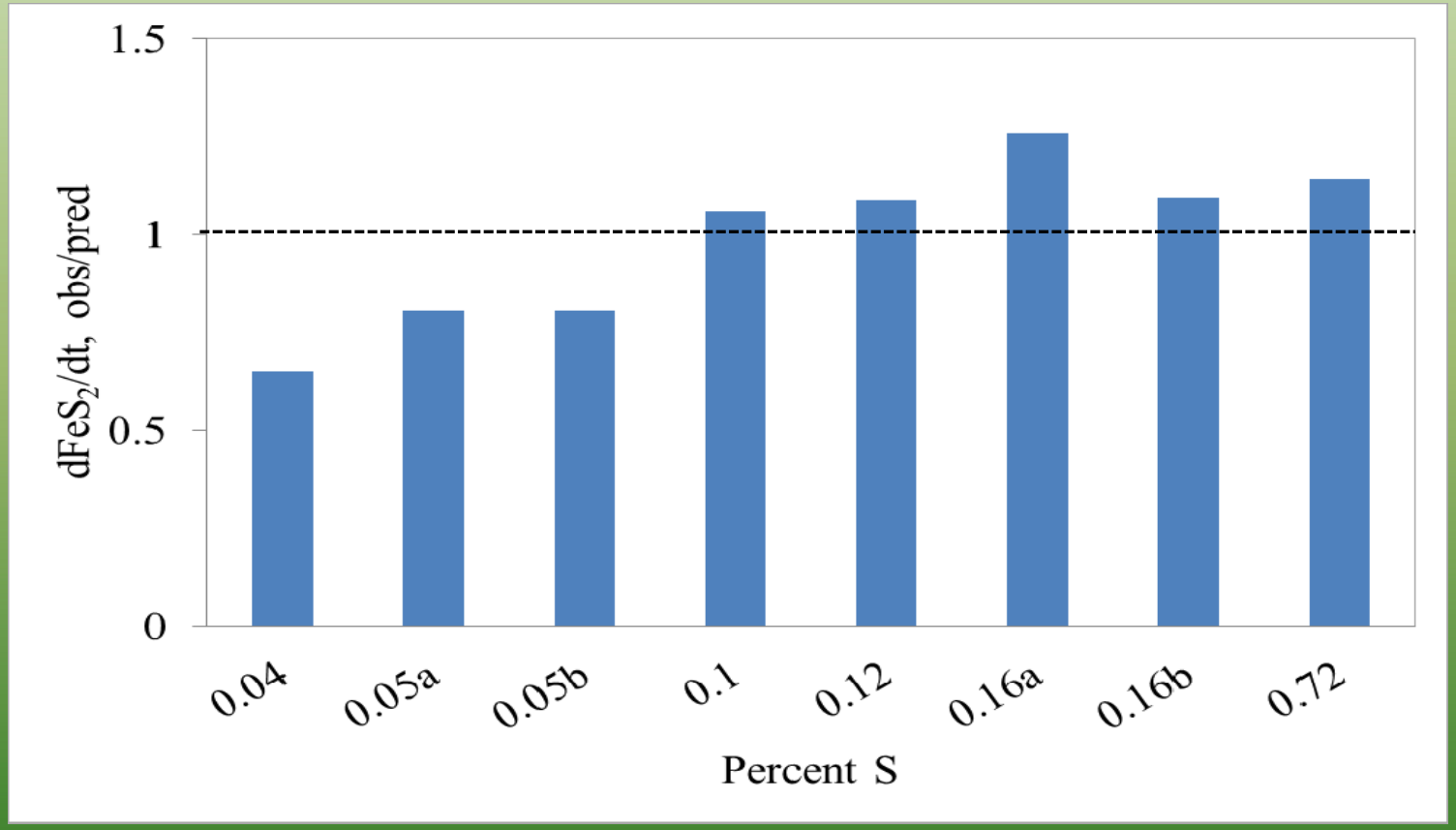
# (3.1) Partially exposed pyrite: 75-150 µm Archean greenstone rock



# (3.1) Liberated pyrite:
# 75-150 um Archean greenstone rock.



# (3.1) Archean greenstone $FeS_2$ oxidation rate: Observed/Predicted (Williamson & Rimstidt 1994).



# (3.2) ASTM D5744 Test Protocol
# 1 kg Archean greenstone rock, d < 6.25 mm

# (3.2) ASTM D5744 Protocol

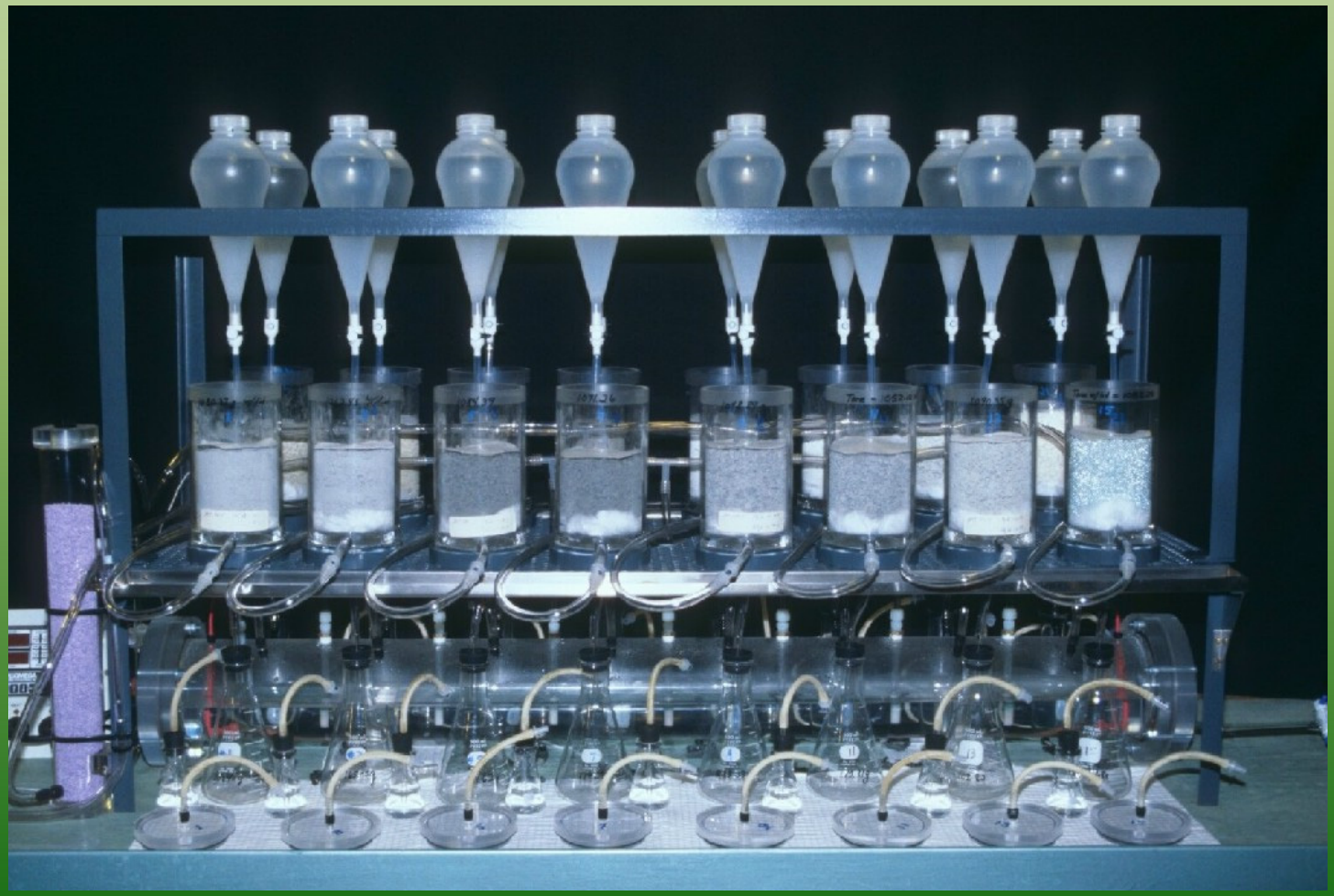
- Load cell, initial rinse (500 or 1000 mL)
- Rock reacts with water air for 1 week
- Controlled T, R.H.
- Rinse on seventh day (500 or 1000 mL)
- Determine drainage volume
- Analyze drainage

# (3.2) ASTM D5744 Protocol Option A

## AIRFLOW

- Set at 1-10 L/min $\pm$ 0.5 L/min
- 3 day dry air (R.H. < 10%)
  - DO NOT DRY CELL COMPLETELY
- 3 day humidified air (R.H. ~95%)

# (3.2) ASTM D5744 Option A Array



# (3.2) ASTM D5744
# Option B

- No air flow
- Controlled T, R.H.
- More consistent and higher water content
- Enhanced acid neutralization for some rocks

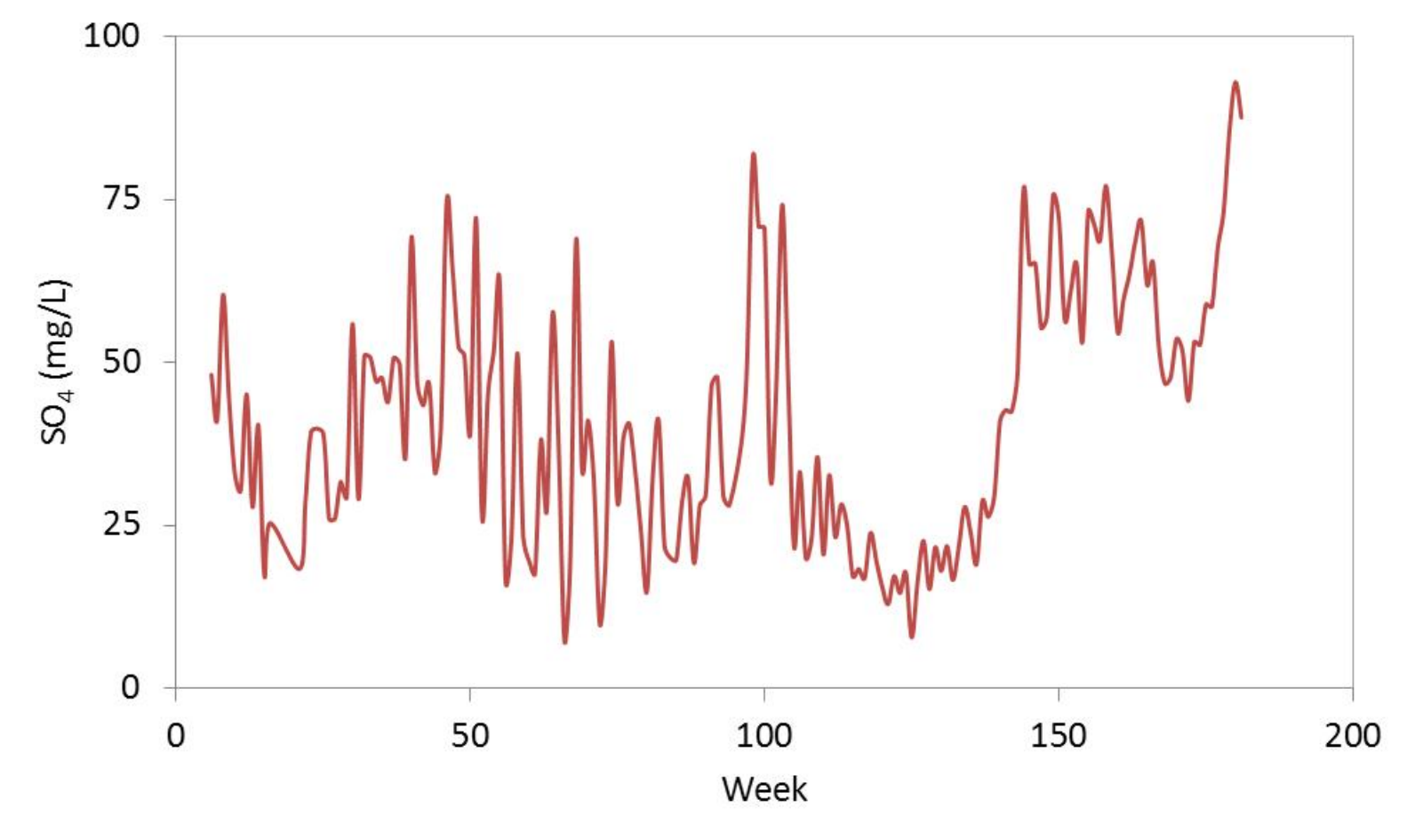
# (3.2) ASTM D5744 Option B

# (3.2) ASTM D5744 Option B

# (3.2) ASTM D5744 Humidity Cell

- Dissolution test procedure
  - Apparatus
  - Solid addition to cell
  - Airflow
  - Reaction T, RH, water retention
  - Leachant
    - Volume
    - Quality
  - Leachate analysis (solutes, frequency)

# (3.2) Uncontrolled Temperature $[SO_4]$ oscillates ~ factor of 10



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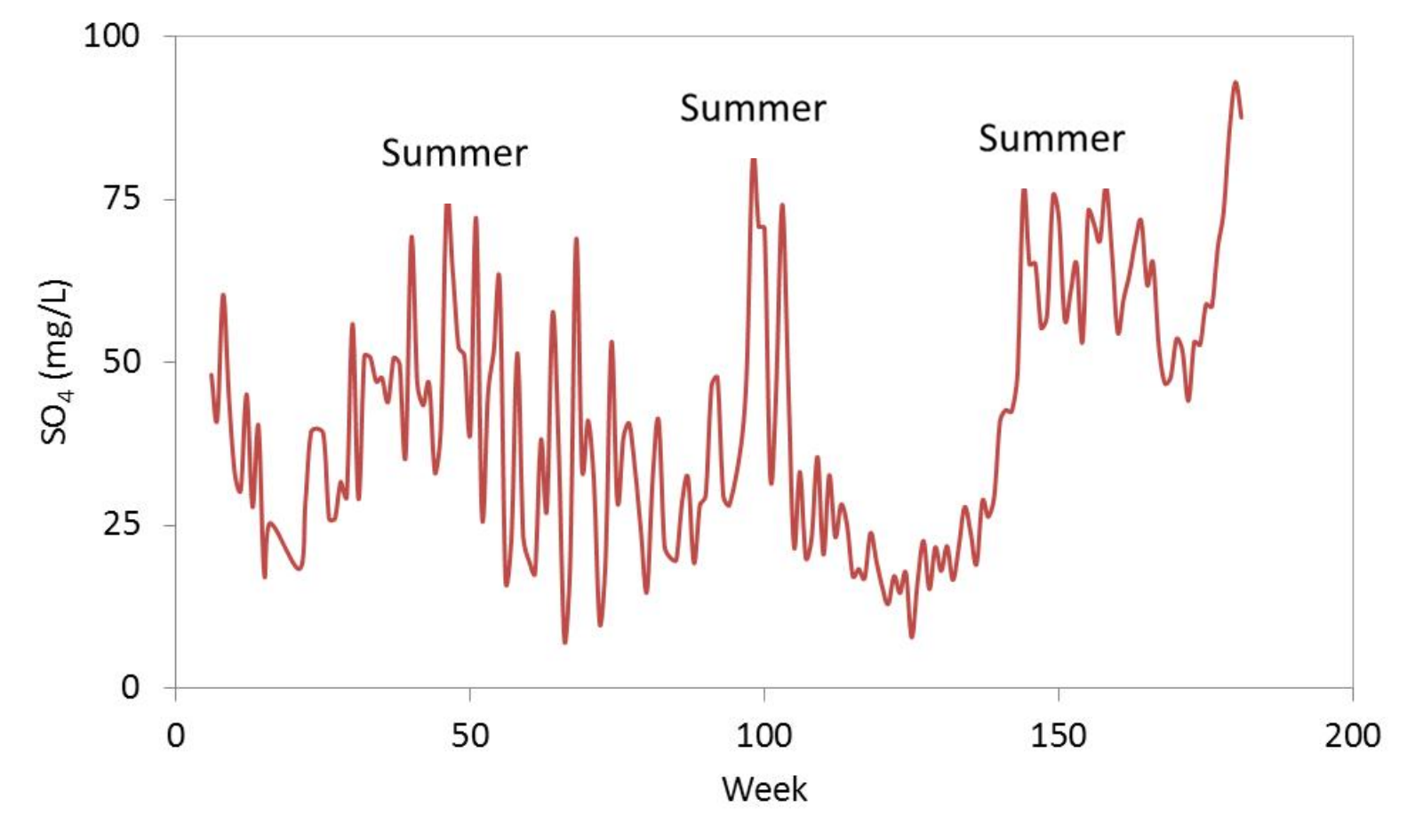
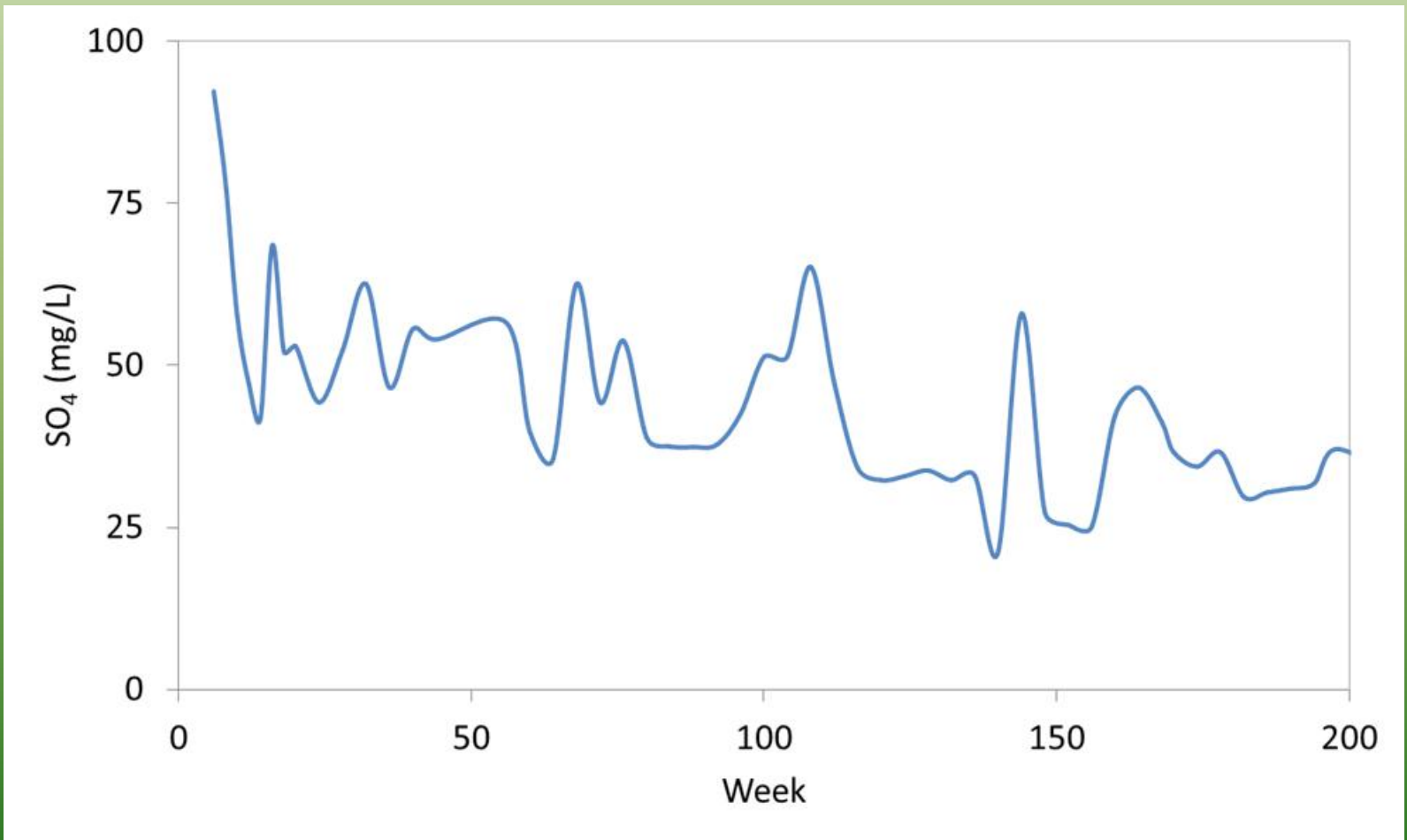


# (3.2) Controlled Temperature

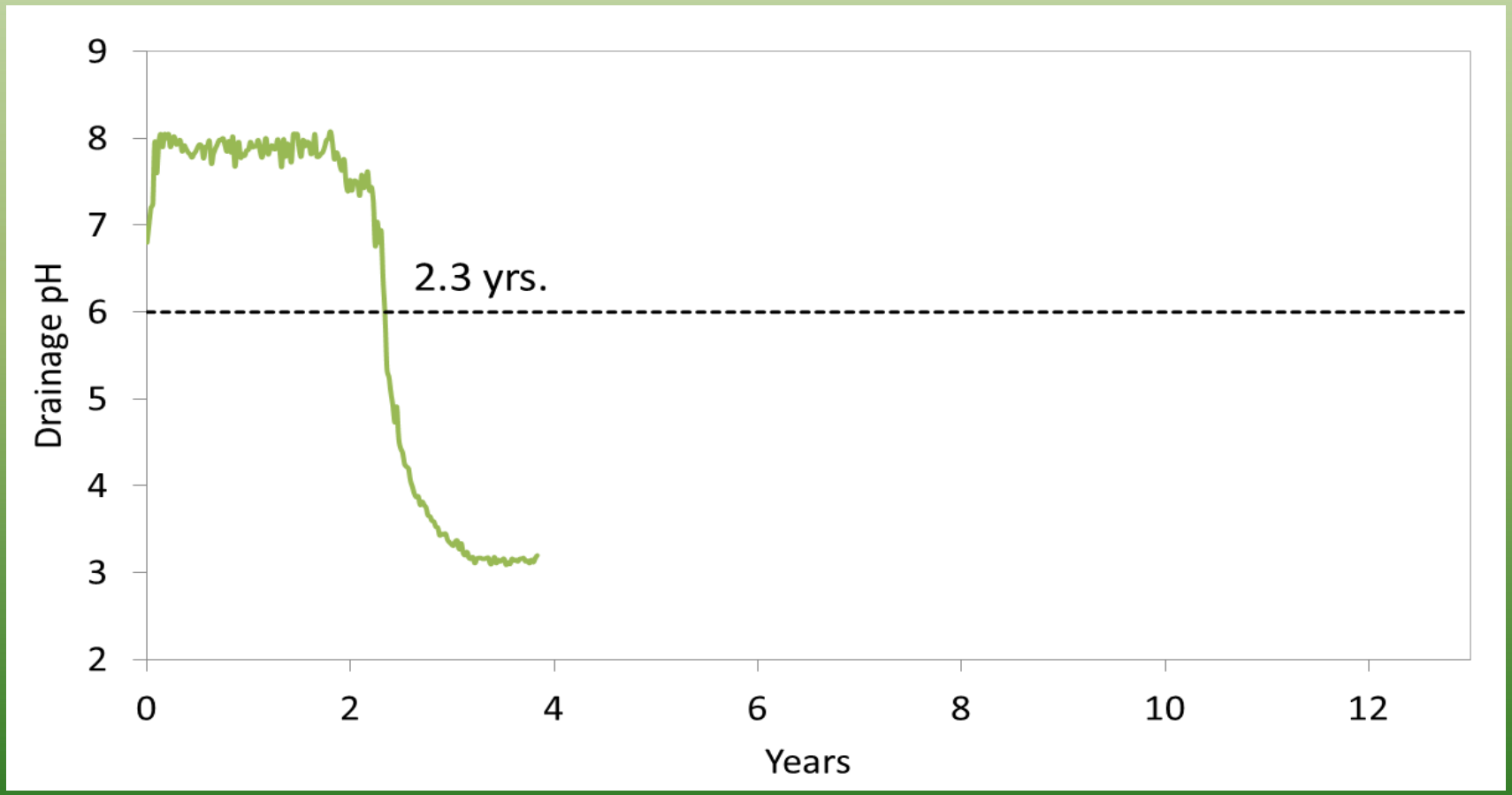


# (3.2) Test Duration Dependent on Test Objectives.

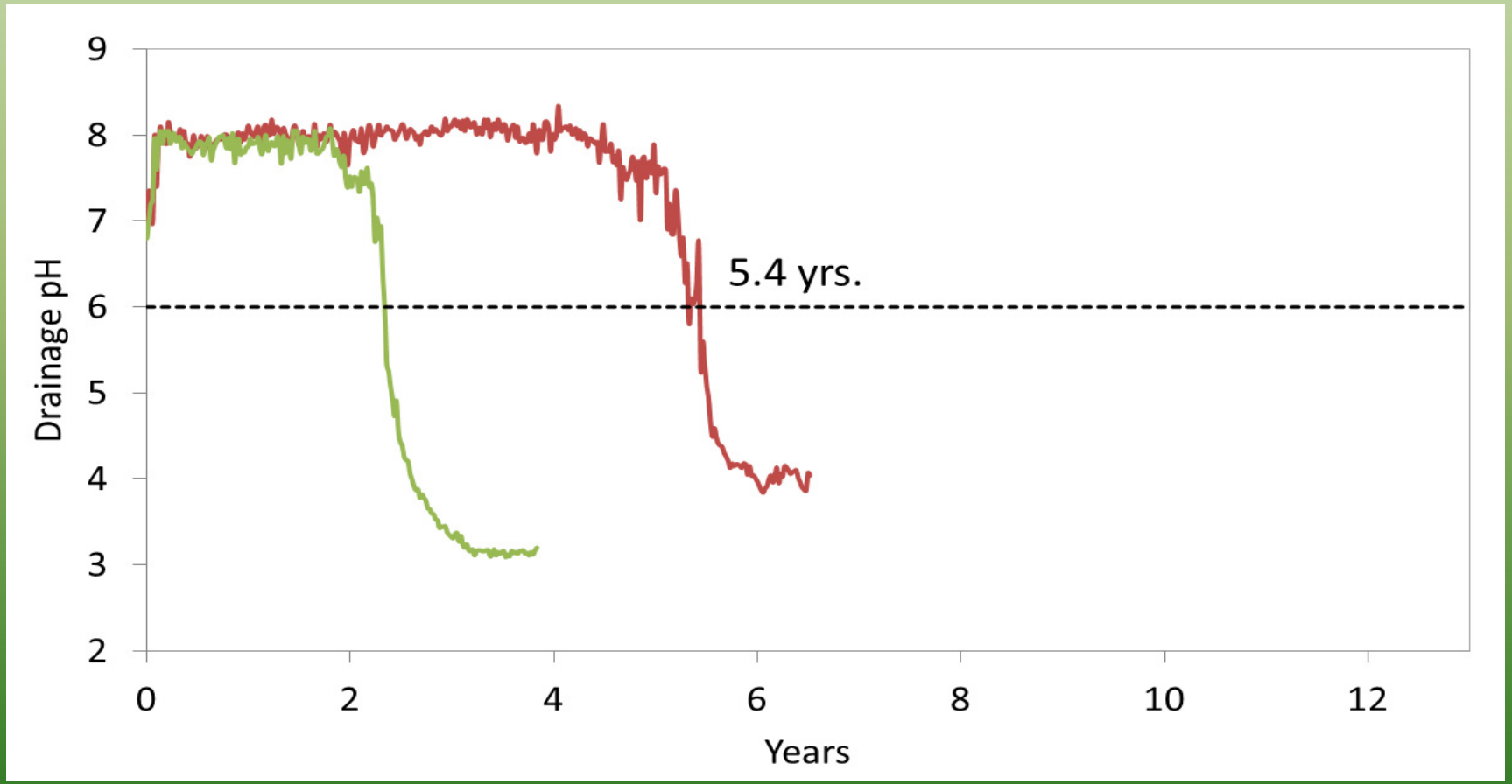
- No prescribed duration
- Clearly define objectives with test design
- Examine data at regular intervals to see if objectives met.
- If (Ca + Mg)$CO_3$ content high, determination of reactivity might require long test

# Data from different method used to demonstrate test duration.

# Test Duration: Time to acidification



# Test Duration: Time to acidification



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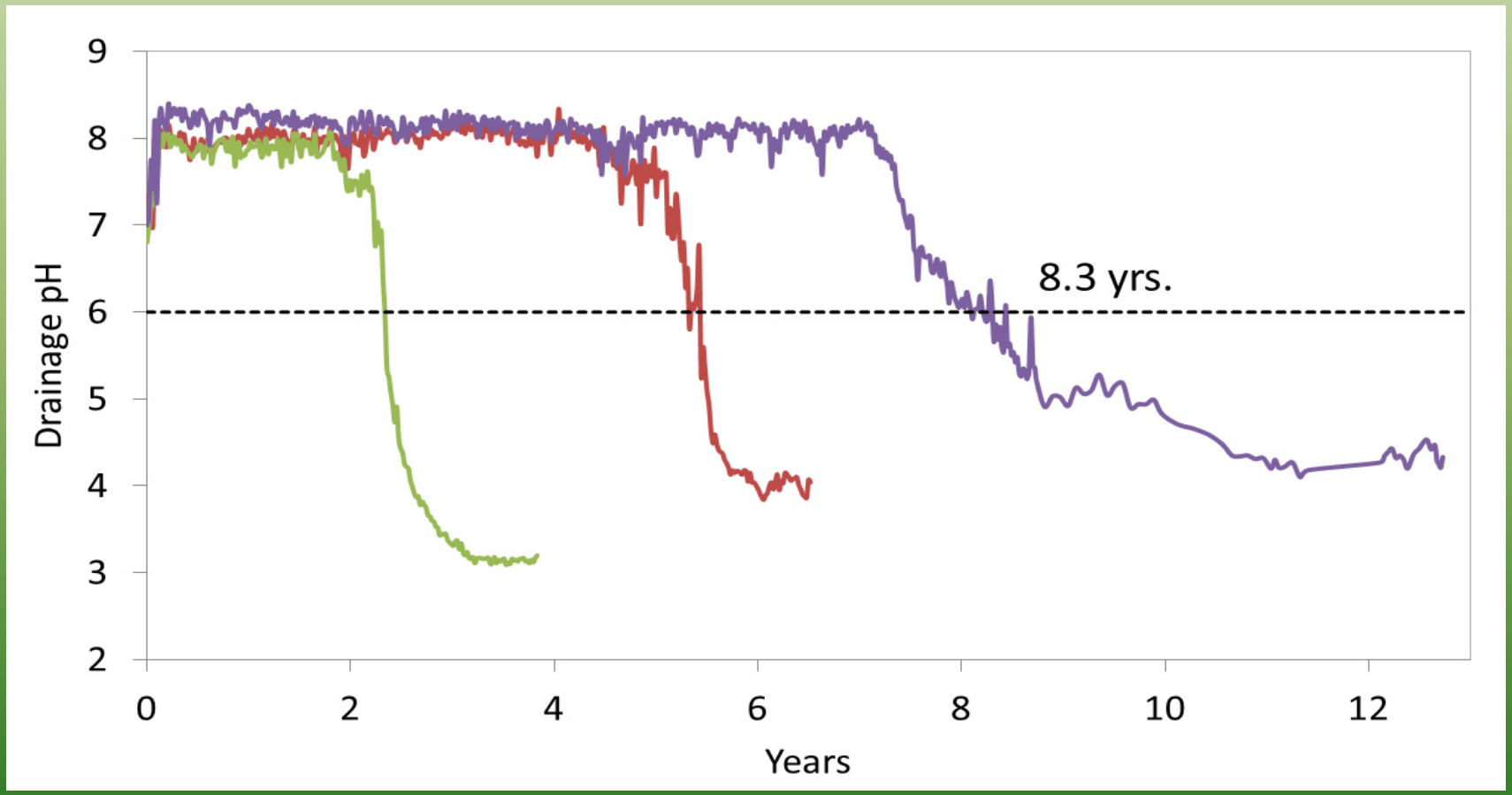

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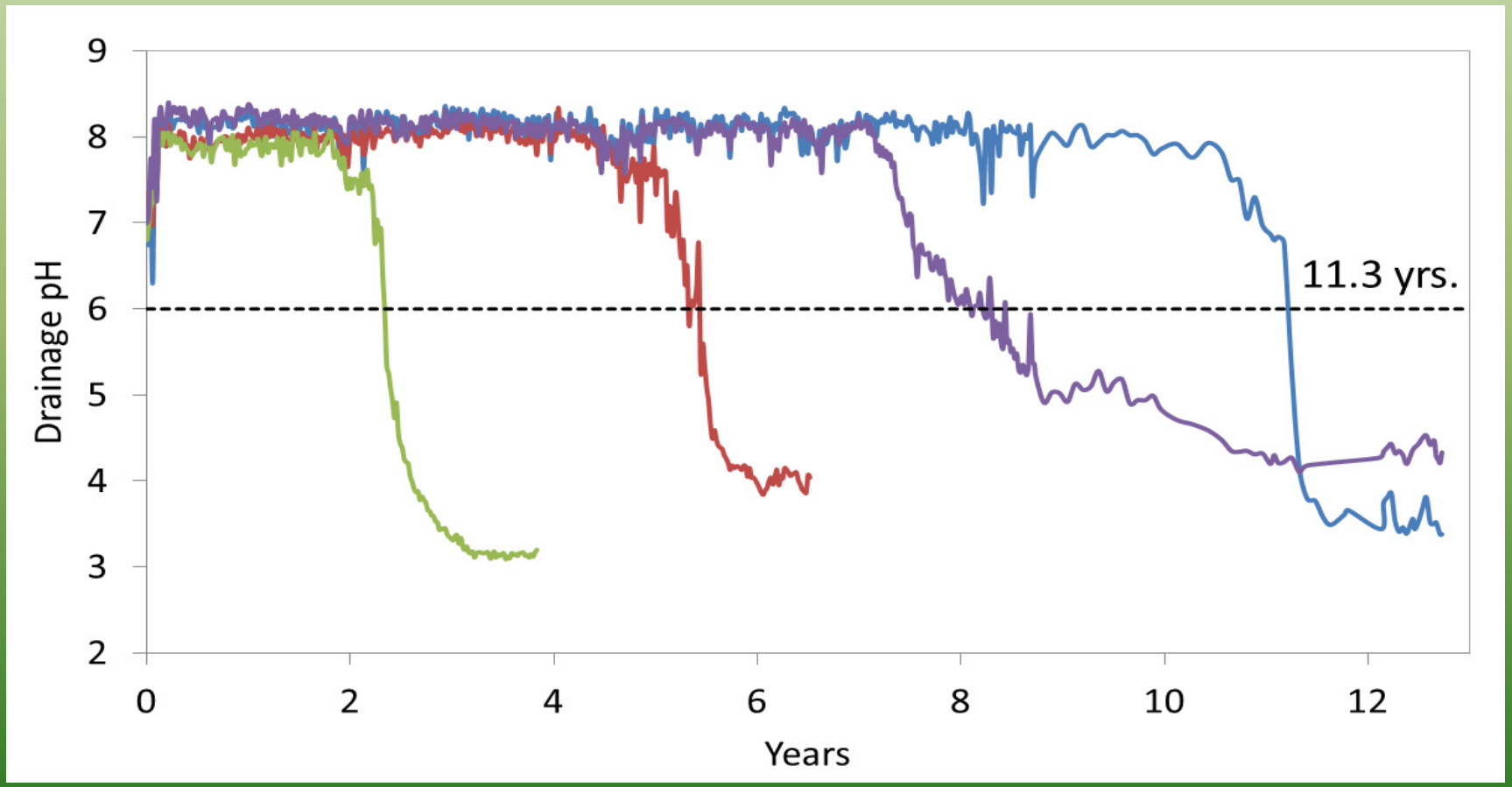

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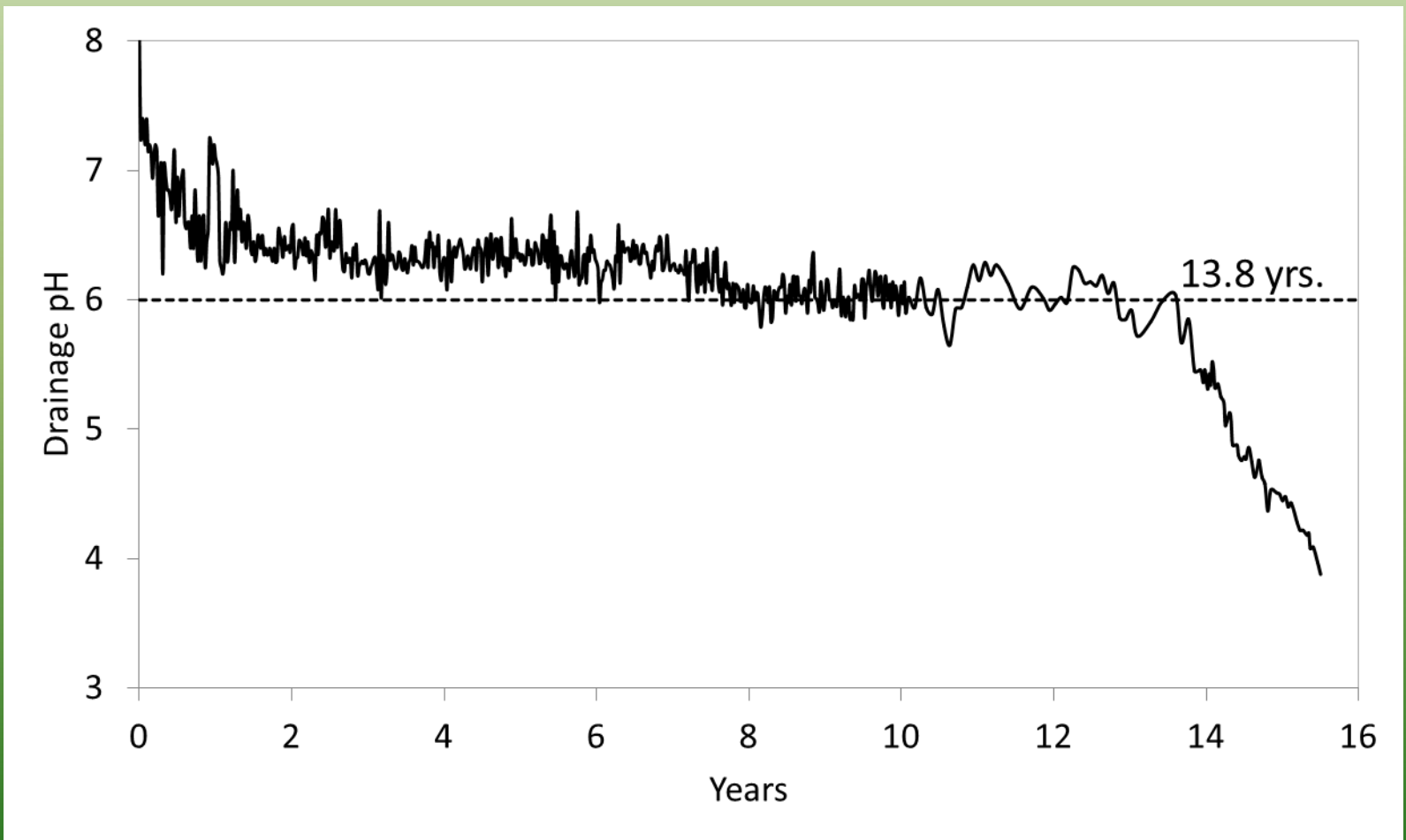
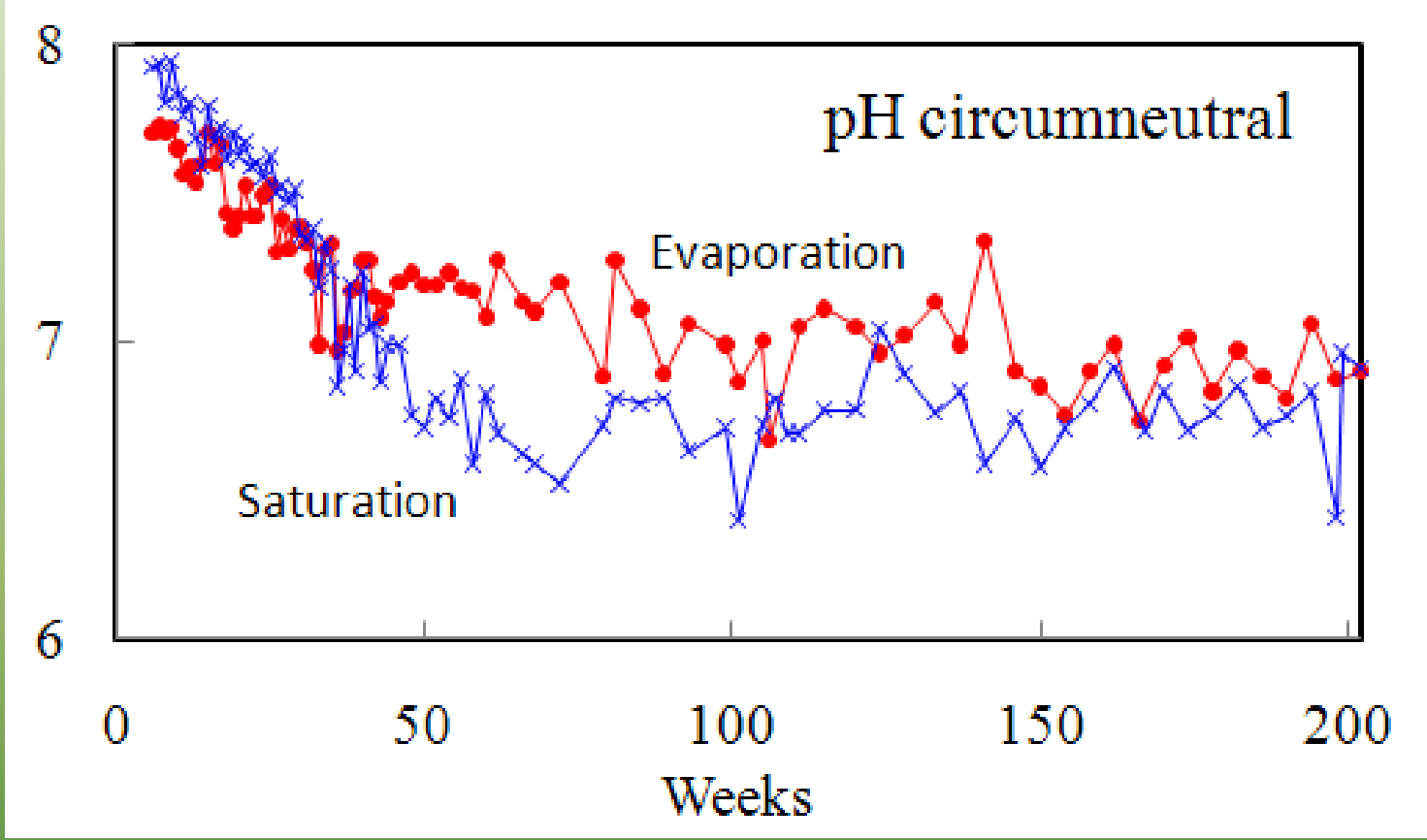


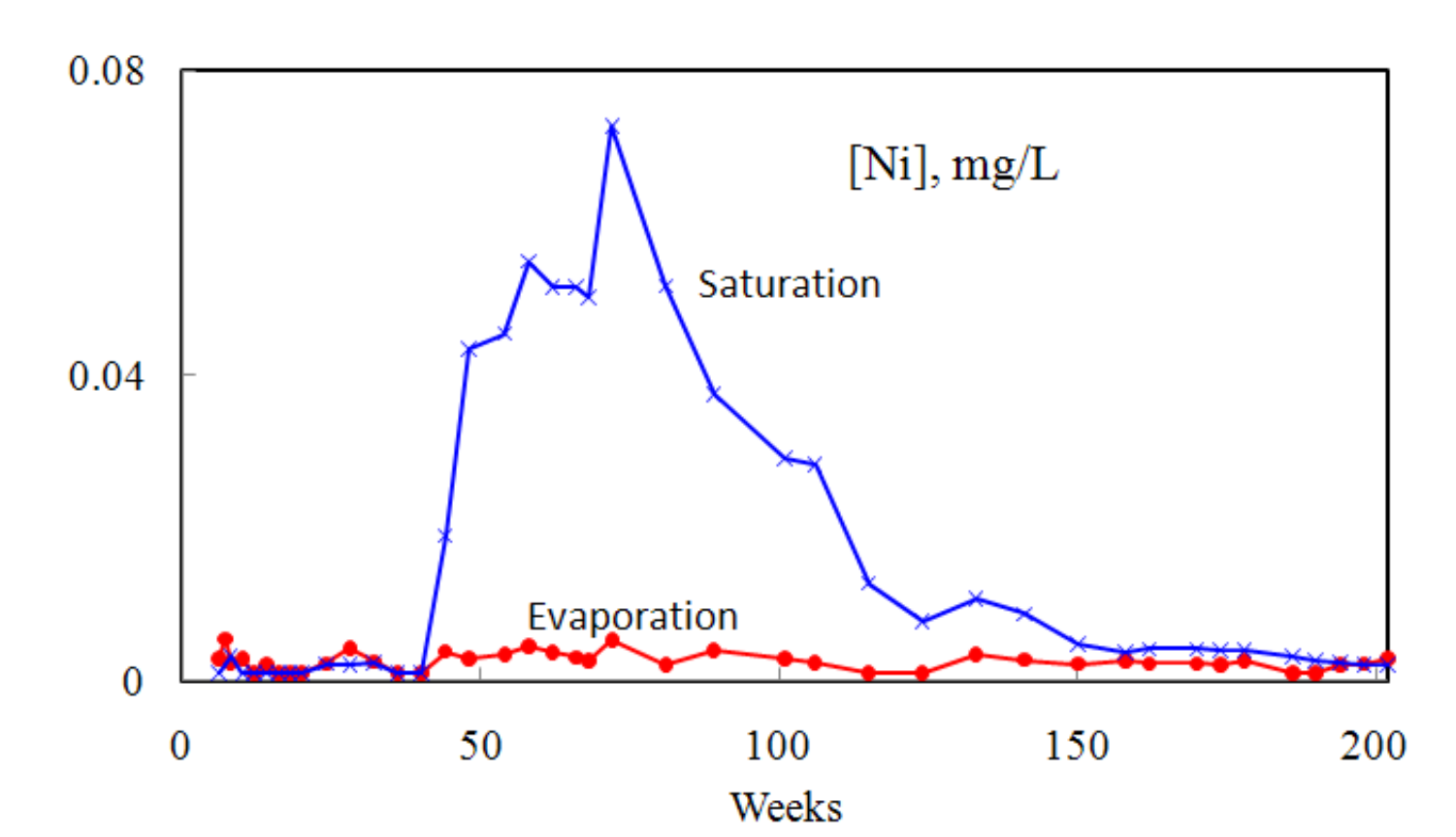
# Test Duration
# Describe Dissolution Behavior

DISCOVERY

How will rock dissolve?




Minimum pH: saturated ~ 0.2-0.3 units lower than evaporating




Max rates: dNi/dt (sat) ~ 20-25 dNi/dt (evap)

# (3.3) ASTM D5744
# Post-test rock analysis

- Visual examination
- Leach extractions
- Mineralogical examination

# (4) Data Analysis

Kinetic test data contributions

4.1) Determine relative degrees of reactivity

4.2) Provide data to quantifying solute release rates

# (4) Data Analysis:
# (4.1) Reactivity Examples

- Waste rock lithology
  - negligible carbonate
  - S present as sulfide

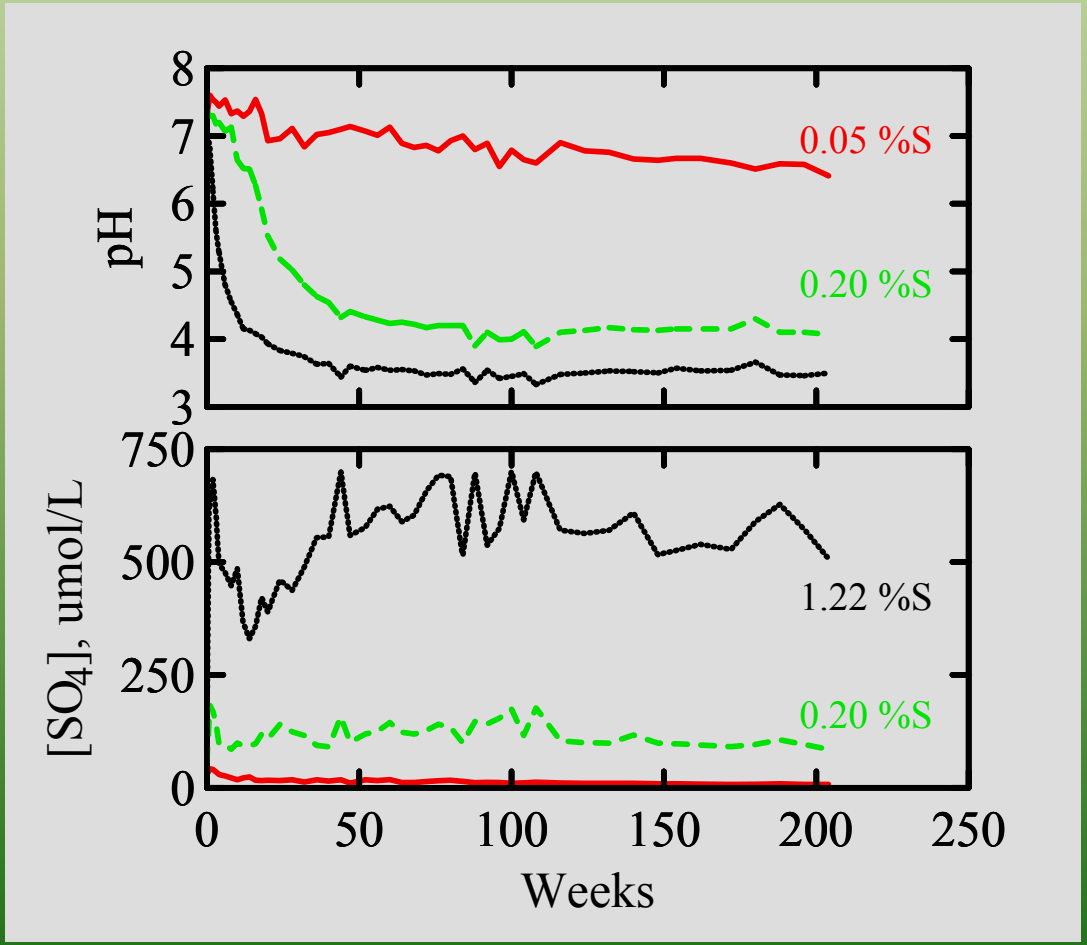
- **KINETIC TEST PROGRAM DESIGN**

  - Select multiple samples of variable S content

  - Subject to long-term (kinetic) dissolution testing

  - Compare results as f(%S)

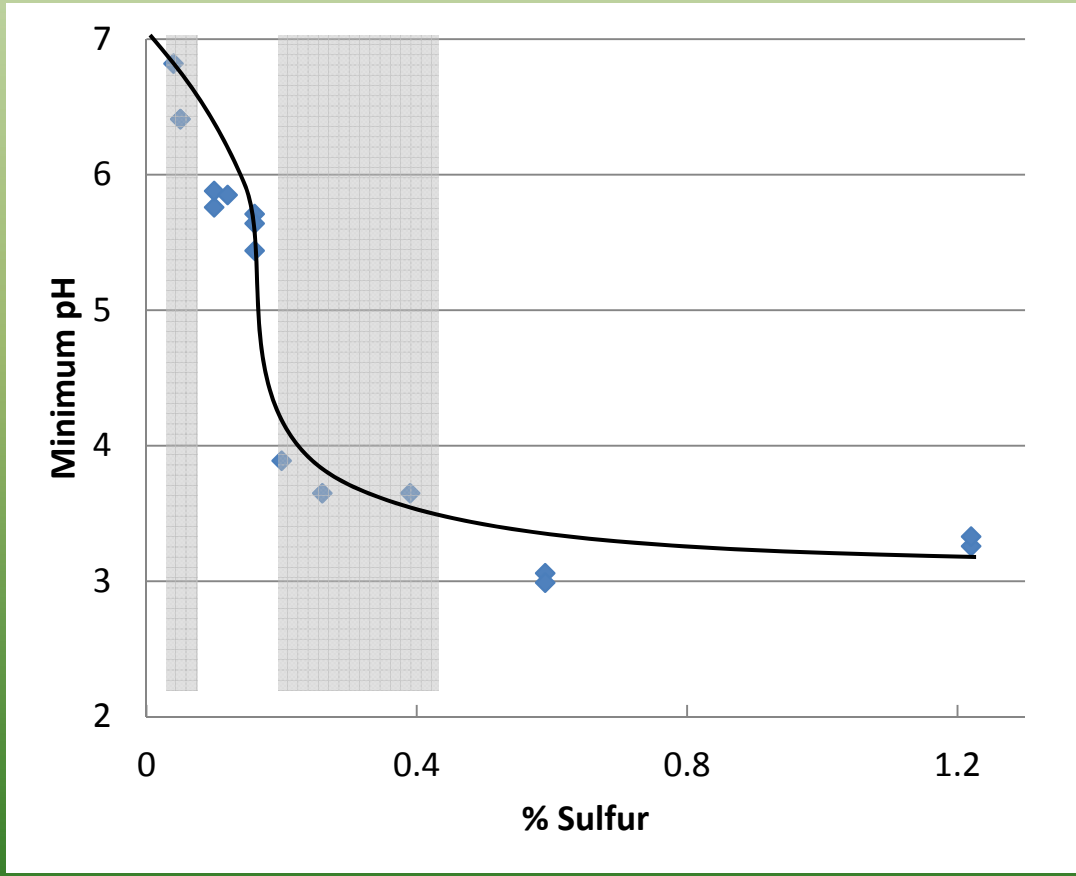
# (4.1) Archean Greenstone: As S content increased: drainage pH decreased and [$SO_4$] increased. (“Reactivity” increases with S)



# (4.1) Archean Greenstone min pH (Reactivity) vs %S

154 – 204 week record

| % S | min pH |
|---|---|
| 0.04 – 0.05 | 6.4 – 6.8 |
| 0.10 – 0.16 | 5.4 – 5.9 |
| 0.20 – 0.40 | 3.6 – 3.9 |
| 0.59 – 1.22 | 3.0 – 3.3 |



# (4.1) Minimum pH vs. %S (Reactivity) Informs waste rock management

Archean greenstone

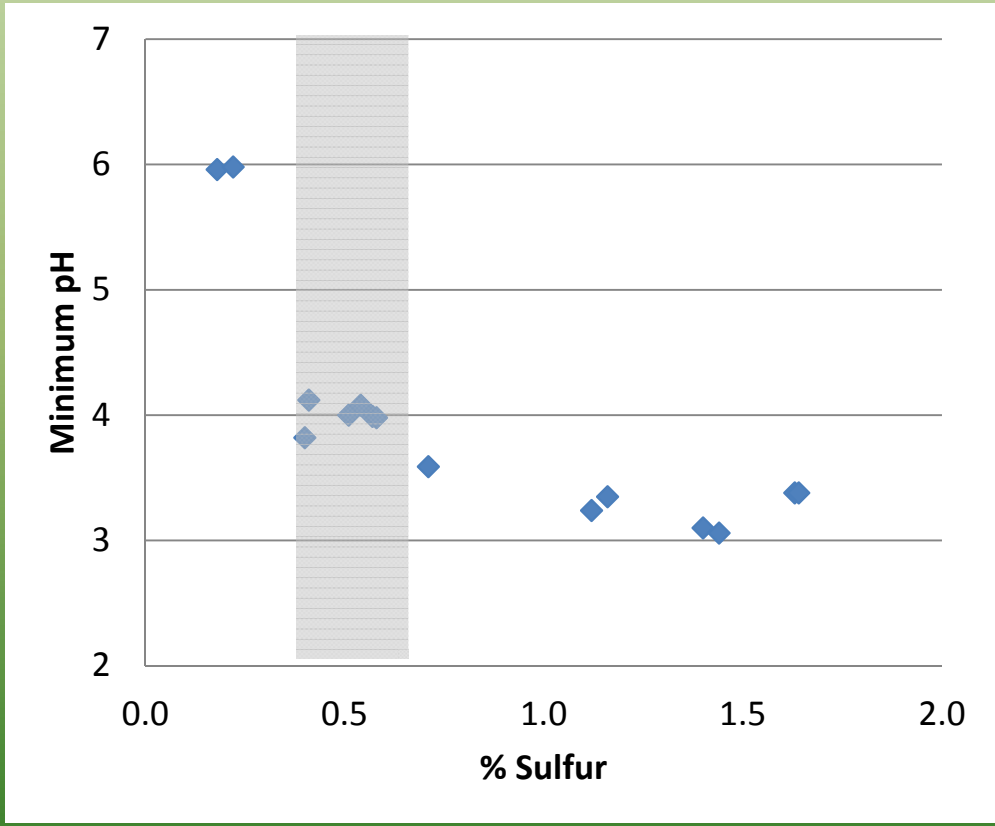
| %S | min pH | Management |
|---|---|---|
| 0.04-0.05 | 6.4-6.8 | Meets pH standards[1] |
| 0.10-0.16 | 5.4-5.9 | Slight pH adjustment? |
| 0.20-0.40 | 3.6-3.9 | Substantial mitigation |
| 0.59-1.22 | 3.0-3.3 | Rigorous mitigation |

[1] *Must check for potential problematic solutes*

# (4.2) Troctolitic rock reactivity, min pH vs %S

324 – 726 week record

| % S | min pH |
|---|---|
| < 0.22 | ~ 6.0 |
| 0.40 – 0.58 | 3.8 – 4.1 |
| 0.71 – 1.64 | 3.1 – 3.6 |



# (4.2) Data Analysis: Solute release rates

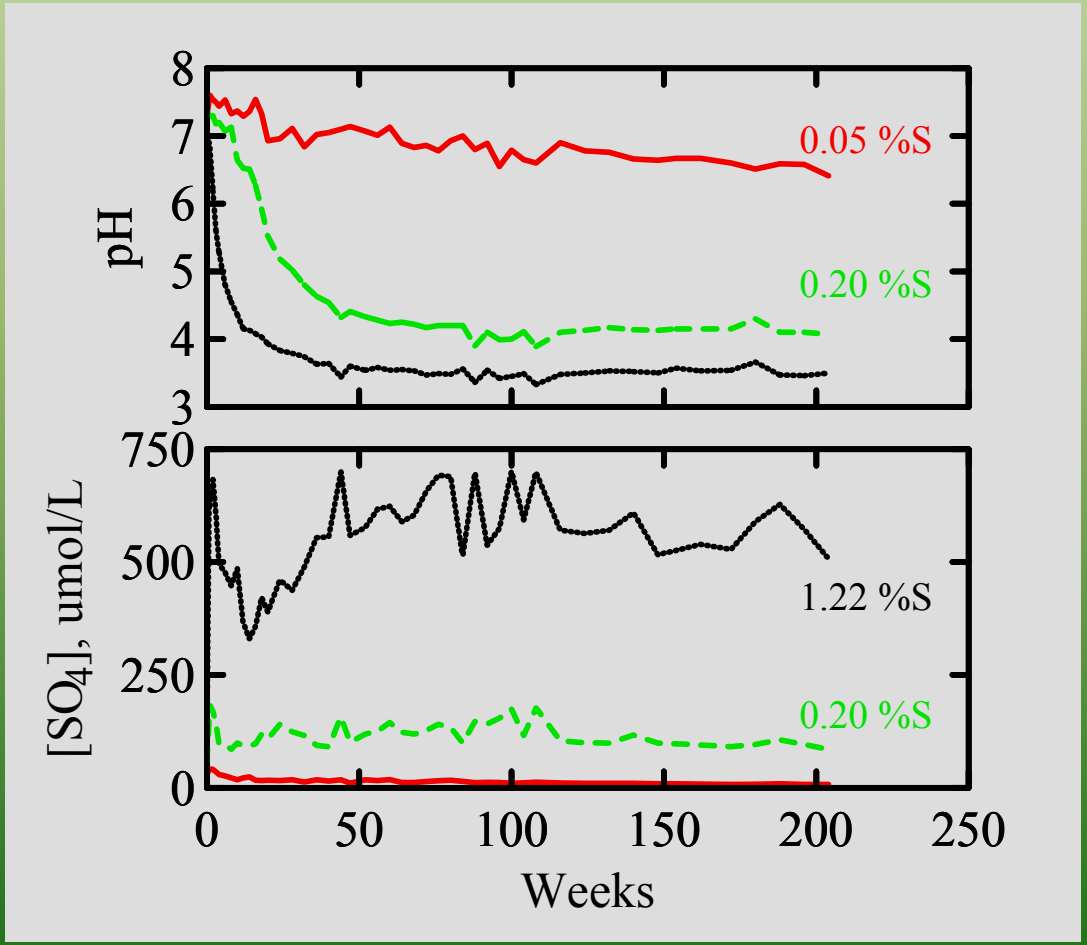
- Observed rates to determine field behavior
- Scale lab to field (NO SMALL TASK)
- Modeling to determine source terms

# (4.2) Data Analysis: Solute release rates

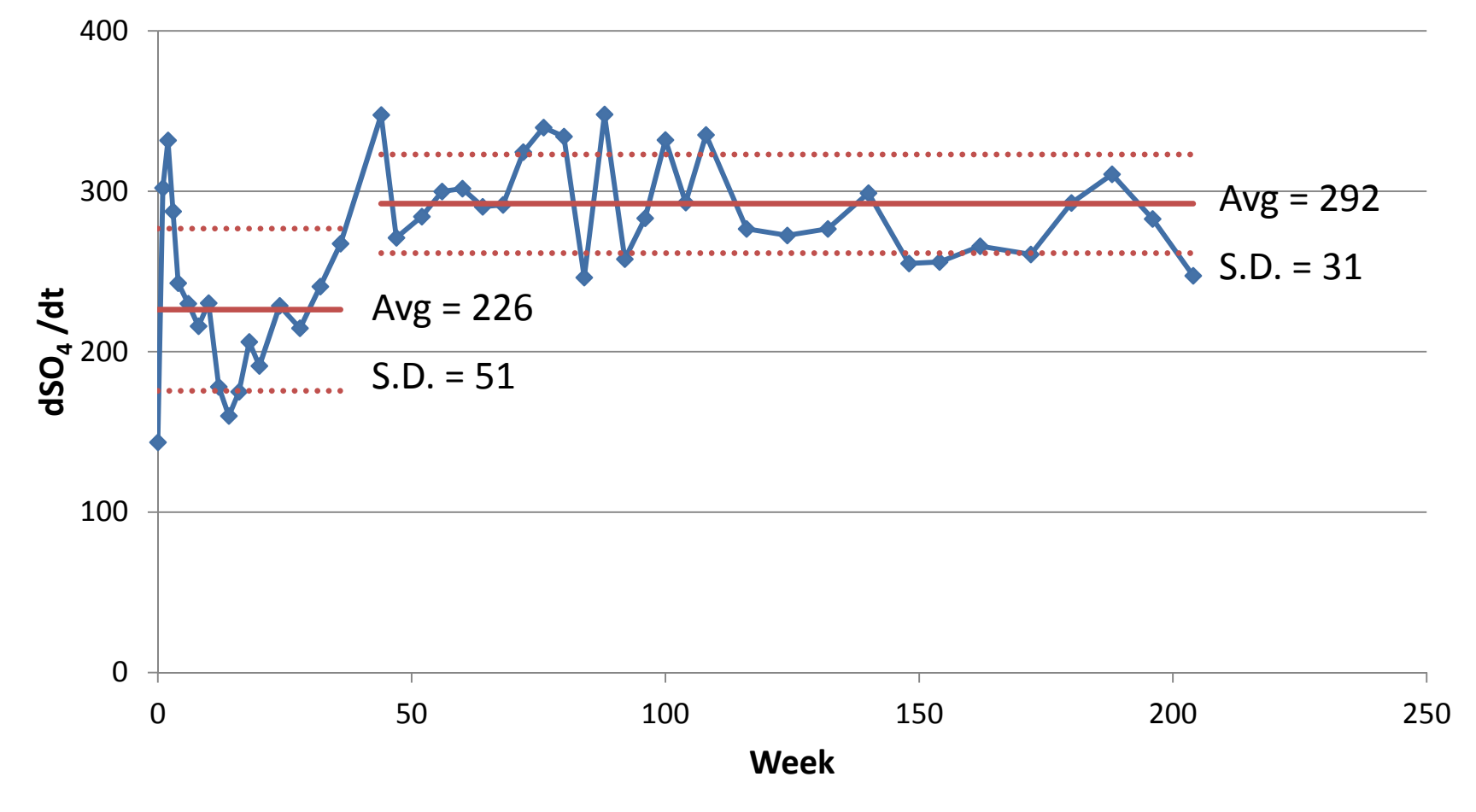
- Humidity cell test yields
  - Solute concentrations
  - Drainage volume
- Can calculate solute release rates
  - Statistical description of all weekly rates or
  - For periods of relatively constant release
    - Average release rate and
    - Standard deviation

# (4.2) Determine $SO_4$ release rates

## Archean Greenstone, 1.22% S



# (4.2) Average sulfate rates: µmol(kg•wk)$^{-1}$



# Summary

- ASTM D5744-13 detailed protocol
  [and 27 explanatory notes]
- Rigorous review process
- Approved in 1996, revised 2007, 2013
- Robust replication testing

# ASTM D5744-13 Humidity Cell

- Provide detailed description of protocol
  - Provide guidance for new practitioners
  - Promote method consistency
  - Increase reproducibility of results
- Contributes to environmentally sound management of mine wastes today
- Generates data to improve understanding of mine waste dissolution tomorrow.

# Helpful References

Method Description

ASTM International. 2013a. D5744-13, Standard test method for laboratory weathering of solid materials using a humidity cell. *In* Annual book of ASTM Standards, 11.04. American Society for Testing and Materials International, West Conschohocken, PA. 23 p. (http://www.astm.org/Standards/D5744.htm?A).

Bucknam. C.H., White III, W., Lapakko, K.A. 2009. Standardization of Mine Waste Characterization Methods by ADTI-MMS. *In* Proc. Securing the Future and 8$^{th}$ICARD, June 22-26, 2009, Skellefteå, Sweden (CD ROM). 12 p.

Lapakko, K.A. 2003. Developments in humidity cell tests and their applications. *In* Environmental Aspects of Mine Wastes, J.L. Jambor, D.W. Blowes, and A.I.M. Ritchie, Eds., *Mineralogical Association of Canada Short Course Volume 31*, 2003, p. 147-164.

To order method: service@astm.org

# Helpful References

Robustness Testing

Lapakko, K.A., White, W.W. III. 2000. Modification of the ASTM 5744-96 kinetic test. In Proceedings from the Fifth International Conference on Acid Rock Drainage. SME, Littleton, CO. p. 631-639.

Lapakko, K.A., White III, W.W. 2013. Research Report D34-1019: Interlaboratory study to establish precision statements for ASTM D5744-13, Test method for laboratory weathering of solid materials using a humidity cell. Committee D34 on Waste Management, Subcommittee D34.01.04 on Waste Leaching Techniques, 1 September 2013. ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. 111 p. (D34-1019 available upon request through ASTM Customer Service at service@astm.org.)

White III, W.W., Lapakko, K.A. 2000. Preliminary indications of repeatability and reproducibility of the ASTM 5744-96 kinetic test for drainage pH and sulfate release rate. In Proceedings from the Fifth International Conference on Acid Rock Drainage. SME, Littleton, CO. p. 621-630.

# Helpful References

Surface area available for reaction

Lapakko, K.A., Antonson, D.A. 2006. Pyrite oxidation rates from humidity cell testing of greenstone rock. In Proc. 2006, 7th ICARD, March 26-30, 2006, St. Louis MO. Published by ASMR, 3134 Montavesta Rd., Lexington, KY 40502. p. 1007-1025.

Lapakko, K. A., Engstrom, J.N., Antonson, D. A. 2006. Effects of particle size on drainage quality from three lithologies. In Proc. 2006, 7th ICARD, March 26-30, 2006, St. Louis MO. Published by ASMR, 3134 Montavesta Rd., Lexington, KY 40502. p. 1026-1050.

http://www.imwa.info/docs/imwa_2006/1026-Lapakko-MN-2%20.pdf

# Helpful References

Test Duration

Lapakko, K. A., Berndt, M. 2009. Laboratory dissolution of tailings under three different test conditions. In Proc. Securing the Future and 8th ICARD, June 22-26, 2009, Skellefteå, Sweden (CD ROM). 11 p.

Lapakko, K. A., Wessels, J. N. 1995. Release of acid from hydrothermal quartz-carbonate hosted gold-mine tailings. In Sudbury '95, Conference on Mining and the Environment, Sudbury, Ontario, May 28th - June 1st, 1995. p. 139-148.

# Helpful References

Good Prediction References


Maest A.S., Kuipers, J.R., Travers, C.I., Atkins, D.A. 2005. Predicting water quality at hardrock mines: methods and models, uncertainties and state-of-the-art. Kuipers & Associates and Buka Environmental.

http://www.ceaa-acee.gc.ca/050/documents_staticpost/cearref_3394/hearings/SM09.pdf

Morin, K., Hutt, N. Mines, Mining, and the Environment, Case studies. http://www.mdag.com

Price W.A. 2009. Prediction manual of drainage chemistry from sulphidic geologic materials. MEND Report 1.20.1.

http://wman-info.org/resources/technicalreports/MENDPredictionManual-Jan05.pdf/file_view