STANDARD OPERATING PROCEDURE No. 76
SLAKE DURABILITY TEST

REVISION LOG		
Revision Number	Description	Date
76v0	Original SOP – Vanessa Candida Viterbo	03/31/05
76v1	Edits LMK	04/07/05
76v1	Ginger sent this one out to a consultant friend to edit for her	Around the middle of April05
76v2	VCV changed data spreadsheet and added comments on Documentation and QA/QC sections	03/16/2007
76v2	Minor corrections by LMK Finalized by LMK for posting on the Molycorp project website and to send to George Robinson for lab audit. LMK did not re-edit this SOP.	4/4/07
76v3	Editorial by SKA	10/28/08

1.0 PURPOSE AND SCOPE

This Standard Operating Procedure (SOP) is based on ASTM D 4644-87 (Reapproved 1992) Standard Test Method for Slake Durability of Shales and Similar Weak Rocks (see Appendix II) and provides technical guidance and procedures that will be employed determining the slake durability index of weathered rocks for the Molycorp Rock Pile Stability Study project. This SOP addresses equipment, procedures, and personnel responsibilities.

2.0 RESPONSIBILITIES AND QUALIFICATIONS

The Team Leader and Characterization Team will have the overall responsibility for implementing this SOP. They will be responsible for assigning appropriate staff to implement this SOP and for ensuring that the procedures are followed accurately.

All personnel performing these procedures are required to have the appropriate health and safety training. In addition, all personnel are required to have a complete understanding of the procedures described within this SOP, and to receive specific training regarding these procedures, if necessary.

All environmental staff and assay laboratory staff are responsible for reporting deviations from this SOP to the Team Leader.

3.0 DATA QUALITY OBJECTIVES

This SOP addresses objectives 3, 4 and 9 in the data quality objectives outlined by Virginia McLemore for the "Geological and Hydrological Characterization at the Molycorp Questa Mine, Taos County, New Mexico".

These objectives are listed as follows, respectively:

- Determine if the sequence of host rock hypogene and supergene alteration and weathering provides a basis to predict the effects weathering can have on mine rock material.
- Determine how weathering of the rock pile affects the geotechnical properties of the rock pile material.
- Determine if the geotechnical and geochemical characteristics of the bedrock (foundation) underlying the rock piles influences the rock pile stability.

4.0 RELATED STANDARD OPERATING PROCEDURES

The procedures set forth in this SOP are intended for use with the following SOPs:

SOP 01	Data management (including verification)
SOP 02	Sample management (chain of custody)
SOP 05	Sampling outcrops, rock piles, and drill core (solid)
SOP 06	Drilling, logging, and sampling of subsurface materials (solid)
SOP 09	Test pit excavation, logging, and sampling (solid)
SOP 22	Analytical data validation
SOP 35	Volumetric moisture content
SOP 36	Sample preservation, storage, and shipment

The procedures set forth in this SOP are also intended for use with the Molycorp drill plans and sampling plans.

5.0 EQUIPMENT LIST

The following materials are required to perform the slake durability test:

- slake durability device:
  - A test drum comprised of a 2.0 mm (No. 10) standard square-mesh cylinder of unobstructed length of 100 mm and diameter 140 mm, with a solid fixed base. The ends shall be rigid plates, with one removable end. The drum and plates must be sufficiently strong to retain their shapes during use. The drum shall be able to withstand a temperature of 110 ± 5º C.

  - A trough to contain the test drum supported with a horizontal axis in a manner capable of being filled with water to a level 20 mm below the drum axis and which shall allow at least 40 mm of unobstructed clearance between the through and the bottom of the mesh.
  - A motor drive capable of rotating the drum at a speed of 20 rpm (constantly to within 5 per cent for a period of 10 minutes).
  - Woven-wire cloth (conforming to the requirements of Specification E 11, Appendix 1).
- Oven (thermostatically controlled, capable of maintaining a temperature of 110 ± 5º C)
- Weighing balance (sensitive to 1 g and having a 2000 g capacity)
- Timing device, such as a watch or clock with a second hand
- Miscellaneous apparatus (including a brush)
- Gloves, safety glasses, dust masks
- Envelopes and plastic bags for samples
- Weatherproof labels and indelible pen
- Distilled water
- Thermometer
- Brush for cleaning rocks and wire baskets
- Rock hammer
- Forms for recording data (Appendix I)

6.0 TEST SAMPLES

Transport samples from the field in a manner that retains the natural water content, i.e. in ziploc bags. The sample for the Slake Durability test shall consist of ten representative fragments that are intact, roughly equi-dimensional, and weigh 40 to 60 g each. These fragments may be naturally occurring or may be produced by breaking with a hammer. Break off any sharp corners, if possible, and remove all the dust by brushing the sample prior to weighing. The total sample should weigh 450 to 550 g.

7.0 SLAKE DURABILITY TEST PROCEDURE

1. Fill out chain of custody forms.
2. Place the sample fragments in the drum.
3. Weigh the drum containing the sample fragments.
4. Dry the drum with the sample fragments in the oven for 16 hours or to a constant mass. Allow the sample and drum to cool at room temperature for 20 minutes and weigh them again.
5. Calculate the natural water content (formula in Section 10.0).
6. Mount the drum in the trough.
7. Couple the drum mounted in the trough to the motor.
8. Fill the trough with distilled water at room temperature to 20 mm (0.8 inches) below the drum axis.
9. Rotate the drum at 20 rpm for a period of 10 minutes.
10. Record the water temperature at the beginning and end of the run.

11. Remove the drum from the trough and the lid from the drum immediately after the rotation period is complete and dry the drum and the sample by retaining them in the oven for 16 hours at 105 ºC, or to constant mass.
12. After cooling, weigh the drum and sample to obtain the oven-dried mass for the second cycle.
13. Repeat items 5 to 9, then weigh the drum and sample again to obtain a final mass.
14. Brush the drum clean and weigh it to obtain its mass.
15. Retain the sample after testing to archive.
16. Record what type the sample is after the test (Type I, II, or III as described in ASTM Method 4644-87 in Appendix 1).

8.0 DOCUMENTATION

Data are recorded on Slake Durability Test Data Sheets (see Appendix 1) and then the data is entered into the Molycorp database.

Slake durability samples are labeled with their field identification number followed by two sequential numbers for before and after the test is run.. For example, for sample PIT-VCV-0001 the slake durability sample number would be PIT-VCV-0001-86 before the test and PIT-VCV-0001-87 after test.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

NMBGMR will archive all samples for mineralogical, geochemical, petrographic, or other studies as needed. For quality control, sample duplicates will be included for every 20 samples tested. The purpose of duplicate sample analyses is to quantify errors from the procedure and from sample heterogeneity.

10.0 DATA ANALYSIS AND CALCULATIONS

Calculate the natural water content using this formula:

$$w = \frac{(A - B)}{(B - C)} \times 100,$$

where:
w = percentage of water content
A = mass of drum plus sample at natural moisture content (g)
B = mass of drum plus oven-dried sample before the first cycle (g)
C = mass of drum (g)
Calculate the slake durability index (second cycle) as follows:

$$I_2(2) = \frac{W_F - C}{B - C} \times 100$$

where:
I_d (2) = slake durability index (second cycle)
B = mass of drum plus oven-dried sample before the first cycle, g
W_F = mass of drum plus oven-dried sample retained after the second cycle, g
C = mass of drum, g

11.0 REFERENCES

1. ASTM, 2001, American Society for Testing Materials. Procedures for testing soils (1964). Standard Test Method for Slake Durability of Shales and Similar Weak Rocks: D4644-87 (Reapproved 1992). Annual Book of ASTM Standards, West Conshohocken, PA.
2. Franklin, J. A. and Chandra, R. The Slake Durability Test. *Int. J. Rock Mech. Min. Sci.,* **9**, 325-341 (1972).

APPENDIX I. FORMS

Figure 1. Slake Durability Test Data Sheet

Slake Durability Test Data Sheet			
Name:	Vanessa C. Viterbo - VCV	**Date:**	
Project:	Molycorp Mine Project	Mass of Drum:	823.27

Specimen Nº	Sample 1	Sample 1	Sample 1	Sample 1
Begin and End Date				
Drum #	1	2	3	4
Mass of Sample (g)				
Oven-dried Sample before the 1st cycle (g)				
Moisture Content (%)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
Water Temperature at the beginning of the 1st run (ºC)				
Water Temperature at the end of the 1st run (ºC)				
Oven-dried sample retained before the 2nd cycle (g)				
Water Temperature at the beginning of the 2nd run (ºC)				
Water Temperature at the end of the 2nd run (ºC)				
Oven-dried sample retained after the 2nd cycle (g)				
Slake Durability Index (second cycle) (%)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!

Visual Description for the retained material: Type I:Pieces remain virtually unchanged Type II:Consist of large and small pieces Type III:Exclusively small fragments **Index Classification:** 0-25: Very low 25-50: Low 50-75:Medium 75-90: High 90-95: Very high 95-100:Extremely high				
Observation:				

Checked by: _____ _____ **Date:** _____

APPENDIX II. ASTM Method D 4644-87

Designation: D 4644 - 87 (Reapproved 1992)

Standard Test Method for Slake Durability of Shales and Similar Weak Rocks[1]

This standard is issued under the fixed designation D 4644; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the slake durability index of a shale or other similar rock after two drying and wetting cycles with abrasion.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[2]

3. Terminology

3.1 *Description of Term Specific to This Standard:*

3.1.1 *slake durability index*—the percentage by dry mass retained of a collection of shale pieces on a 2.00 mm (No. 10) sieve after two cycles of oven drying and 10 min of soaking in water with a standard tumbling and abrasion action.

4. Significance and Use

4.1 The test method is used to estimate qualitatively the durability of weak rocks in the service environment.

4.2 This test method is used to assign quantitative durability values to weak rocks. A primary example is the Franklin Rating System (1).[3]

5. Apparatus

5.1 *Slake Durability Device*—The drum (Fig. 1) shall be made of 2.00 mm (No. 10) square-mesh, woven-wire cloth, conforming to the requirements of Specification E 11. It shall be cylindrical in shape, with a diameter of 140 mm (5.5 in.) and a length of 100 mm (3.9 in.). The ends shall be rigid plates, with one removable end. It must be sufficiently strong to retain its shape during use, but neither the exterior of the mesh nor the interior of the drum shall be obstructed by a support. The drum shall be able to withstand a temperature of 110 ± 5°C (230 ± 9°F). A trough shall support the drum in a horizontal manner such that the drum is free to rotate about its axis. The trough shall be capable of being filled with slaking fluid to 20 mm (0.8 in.) below the drum axis, and shall allow at least 40 mm (1.6 in.) unobstructed clearance between the trough and the bottom of the mesh. The drum shall be rotated by a motor capable of maintaining a speed of 20 rpm, constant to within 5 %, for a period of 10 min. Devices conforming to these requirements are commercially available.

5.2 *Drying Oven*, thermostatically controlled, capable of maintaining a temperature of 110 ± 5°C (230 ± 9°F).

5.3 *Balance*, sensitive to 1 g and having a 2000-g capacity.

5.4 *Miscellaneous Apparatus*, including a brush.

5.5 *Distilled Water.*

6. Test Specimen

6.1 The specimen shall consist of ten representative, intact, roughly equidimensional shale fragments weighing 40 g to 60 g each. These fragments may be naturally occurring or may be produced by breaking with a hammer. Such fragments may be obtained from rock cores or from test pits, and their sizes will vary with the method of sampling. Break off any sharp corners, if possible, and remove any dust by brushing the sample just prior to weighing. The total sample shall weigh 450 to 550 g.

6.2 Transport and store the sample in such a manner as to retain the natural water content.

7. Procedure

7.1 Place the shale fragments in the drum. Weigh, and dry in the oven for 16 h or to constant mass. Allow the shale and drum to cool at room temperature for 20 min and weigh again. Calculate the natural water content as follows:

$$w = [(A - B)/(B - C)] \times 100$$

where:

w = percentage water content,
A = mass of drum plus sample at natural moisture content, g,
B = mass of drum plus oven-dried sample before the first cycle, g, and
C = mass of drum, g.

7.2 Mount the drum in the trough and couple to the motor. Fill the trough with distilled water at room temperature to 20 mm (0.8 in.) below the drum axis. Rotate the drum at 20 rpm for a period of 10 min. Record the water temperature at the beginning and end of the run.

7.3 Remove the drum from the trough immediately after the rotation period is complete and dry the drum and the sample retained in the oven for 16 h, or to constant mass.

7.4 Weigh the drum and sample to obtain the oven-dried

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.12 on Rock Mechanics.

Current edition approved Feb. 2, 1987. Published April 1987.

[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

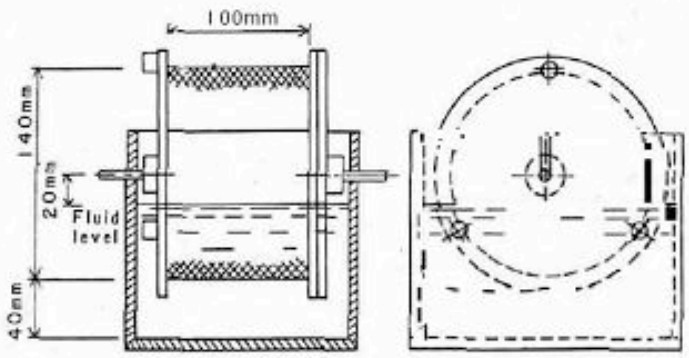

FIG. 1 Critical Dimensions of Slake Durability Equipment

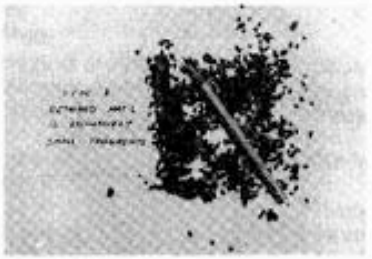

FIG. 2 Illustration of Fragment Types Retained

mass for the second cycle. Repeat 7.2 and 7.3. Again weigh the drum and sample to obtain a final mass.

7.5 Photograph materials retained and file a copy of the photo with the laboratory report, or record standard verbal descriptions, as follows:

7.5.1 *Type I*—Retained pieces remain virtually unchanged.

7.5.2 *Type II*—Retained materials consist of large and small pieces.

7.5.3 *Type III*—Retained material is exclusively small fragments.

7.6 See Fig. 2 for representative photographs of the three types.

8. Calculation

8.1 Calculate the slake durability index (second cycle), as follows:

$$I_d(2) = [(W_F - C)/(B - C)] \times 100$$

where:

$I_d(2)$ = slake durability index (second cycle),
B = mass of drum plus oven-dried sample before the first cycle, g,
W_F = mass of drum plus oven-dried sample retained after the second cycle, g, and
C — mass of drum, g.

9. Report

9.1 The report shall include the following:

9.1.1 Description of the material and where it was obtained.

9.1.2 Slake durability index (second cycle) to the nearest 0.1 %.

9.1.3 Range and average value of the water temperature.

9.1.4 Natural water content.

9.1.5 Description of the appearance of the fragments retained in the drum (see 7.5).

10. Precision and Bias

10.1 Due to the nature of shale and other similar rock tested by this test method it is either not feasible or too costly at this time to produce multiple specimens which have uniform physical properties. Any variation observed on the data is just as likely to be due to specimen variation as to operator or laboratory testing variation. Subcommittee D18.12 welcomes proposals that would allow for development of a valid precision statement. There is no accepted

reference value of shale or weak rock for this test method; therefore, bias cannot be determined.

11. Keywords

11.1 abrasion resistance; shale; slake durability; weal rocks

REFERENCES

(1) Franklin Trow Associates, "Field Evaluation of Shales for Construction Projects," Research and Development Project No. 1404, Ministry of Transportation and Communications, Research and Development Branch, **Downsview,** Ontario, March **1979.**

(2) Chapman, D. R., "Shale Classification Tests and Systems: A Comparative Study," **MSCE** Thesis and Joint Highway Research Project No. 75-11, **Purdue** University, West Lafayette, IN, June 1975, 90 pp.

(3) Deo, P., "Shales as Embankment **Materials,"** Ph.D. Thesis and Joint Highway Research Project No. 45, Purdue University, West Lafayette, IN, December 1972, 202 pp.

(4) International Society for Rock Mechanics, "Suggested Methods for Determining **Slake-Durability** Index Properties," Commission on Standardization of Laboratory and Field Tests, November 1972.

(5) Lutton, R. J., "Design and Construction of Compacted Shale Embankments, Volume 3, Slaking Indexes for Design," Report No. **FHWA-RD-77-1,** Federal Highway Administration, Washington, DC, February **1977.**

(6) **Surendra,** M., "Additives to Control Slaking in Compacted **Shales,"** Ph.D. **Thesis** and Joint Highway Research Project No. **80-6,** Purdue University, West Lafayette, IN, May **1980,** 277 pp.

(7) Oakland, M. W., and **Lovell,** C. W., "Classification and Other Standard Tests for Shale Embankment," Joint Highway Research Project No. **82-4,** Purdue University, West Lafayette, IN, February 1982, 171 pp.