

STANDARD OPERATING PROCEDURE No. 32

BULK DENSITY FOR UNDISTURBED SAMPLES

REVISION LOG		
Revision Number	Description	Date
0.0	Original SOP	11/6/03
32.1	Revisions by PJP	5/19/2004
32.2	Revisions by JMS	8/31/04
32.3	Revisions by PJP as per JMS	9/24/04
32.4	Minor edits JRM, approved VTM	01/14/2005
32v5	Edits & Comments LMK	2/7/05
32v6	Re-edit LMK and repost to Utah site	6/27/06
32v6	Finalized by LMK for posting to project website and to send George Robinson for lab audit (no new edits)	4/2/07
32v7	Editorial by SKA	10/24/08

1.0 PURPOSE AND SCOPE

This Standard Operating Procedure describes a laboratory procedure for determining the bulk density of an undisturbed sample of rock pile material. This method is only appropriate for samples which have been extracted from the field without any changes in density, shape, or volume. Bulk density can also be estimated from field measurements using a nuclear density gauge (see SOP 61) or measured with a sand cone (see SOP 65) or sand replacement (see SOP 70) test. Bulk density measurements of the same material with all three methods will not necessarily yield the same results because each method interrogates a different volume and has a different set of principle assumptions.

Bulk density, ρ , is the ratio of sample mass to sample volume for a dry sample. Bulk density can be calculated from the particle density (dry mass of all solid particles in a sample divided by the volume of those solid particles) or the specific weight of a dry soil given the sample porosity. Bulk density or sample volume is needed to convert gravimetric moisture content (SOP 40) to volumetric moisture content (SOP 35). Bulk density is routinely required in numerical codes for modeling variably saturated flow and transport, e.g., FEHM [Zyvoloski, 1997] and HYDRUS-2D [Simunek et al, 1994].

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 they will 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 1 and 7 in the data quality objectives outlined by Virginia McLemore for the "Geological and Hydrological Characterization at the MolyCorp Questa Mine, Taos County, New Mexico".

- Determine how the hydrogeochemistry and water balance dynamics influence rock pile weathering and stability.
- Determine if pyrite oxidation, moisture content, and microbe populations affect rock pile weathering and stability.

4.0 RELATED STANDARD OPERATING PROCEDURES

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

- SOP 1 Data management (including verification and validation)
- SOP 2 Sample management (including chain of custody)
- SOP 4 Taking photographs
- SOP 35 Volumetric moisture content
- SOP 40 Gravimetric moisture content
- SOP 42 Porosity
- SOP 61 Nuclear density gauge
- SOP 65 Sand cone

- SOP 70 Sand replacement

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

5.0 EQUIPMENT LIST

The following materials are required for collecting undisturbed samples from the field, whether for determination of bulk density (this SOP) or for volumetric moisture content (SOP 35):

- Aluminum foil or plastic wrap
- Plastic or metal coring cylinders of known volume and density (if needed)
- Ziploc bags
- Weatherproof labels
- Bubble wrap and tape
- Boxes or coolers to store and transport wrapped samples in an undisturbed state
- Thermometer
- Indelible marker
- Field sample forms (see section 9)

The following materials are required for analyzing undisturbed samples for bulk density using the wax clod laboratory method (Blake et al., 1986):

- Scale with hook for suspending samples beneath the scale
- Weights to calibrate scale
- Paraffin wax and deep-walled container to hold melted paraffin
- Heat source to melt paraffin wax
- Thin copper or similar metallic wire (32 gauge or narrower)
- Glass vials to measure paraffin bulk density
- Beaker sufficiently wide to immerse suspended sample completely in water without touching the beaker's sides
- Field sample forms (Appendix 1)

6.0 COLLECTION OF SAMPLES

Samples are collected from split spoon samples from drill holes, test pits, and trenches. Samples should only be collected from materials if those materials are undisturbed by cracks, compaction, or shearing. Photographs with a scale bar should be taken before the sample is collected, if possible.

Sample must be quickly sealed in aluminum foil or similar wrapping, sealed again in zip lock bag, wrapped with bubble wrap or plastic foam, and stored in a cooler to prevent disturbance of sample during transport. It is essential that the initial sample volume and shape be maintained throughout the collection, transport, and laboratory analysis steps.

Coring of outcrop walls or sub-coring of split spoon samples may be required to collect a sample from poorly cohesive material. Great care must be taken to not disturb sample density during the coring procedure and to accurately measure the internal volume of the coring device.

Sample wrapping must be labeled with sample ID, date and time of collection, and collector's initials using indelible marker and weatherproof labels.

Refer to SOPs 1 and 2 for relevant requirements regarding the collection, labeling, preservation, and transportation of samples.

7.0 PROCEDURES

Field procedures are the same for this SOP as they are for SOP 35 (volumetric moisture content).

Bulk density is the mass of a dry sample (mass of solids) divided by the volume of the sample. If the sample is collected using a container of known volume, determining the sample's volume and calculating bulk density is straightforward: divide the sample's dry mass by its volume. If, however, the sample volume is not known and cannot be accurately determined from the sample's dimensions, the volume must be measured using a suitable method. Of the many methods available, the procedure below describes the wax clod method, which is best suited for samples which are not easily deformed or disaggregated.

1. Photograph the core, pit, or trench with a scale bar.
2. Collect intact sample by hand or with coring device. Wrap with aluminum foil or plastic wrap. Seal in Ziploc bag and wrap with bubble wrap or plastic foam. Label appropriately. Complete sample form (Appendix 1). Transport to the laboratory. Log samples into laboratory, following Chain of Custody procedures SOP 2.
3. Calibrate scale using standard weights.
4. Remove wrapped sample from Ziploc bag. Weigh wrapped sample to within 0.01 g and record.
5. Weigh aluminum weighing tare to within 0.01 g and record as tare weight.
6. Carefully remove sample from wrapping and weigh sample to within 0.01 g in a weighing tare. Subtract tare weight and record the result as sample wet weight.
7. Weigh sample wrapping to within 0.01 g. Compare the sum of this weight and the sample wet weight to the wrapped sample weight for quality control.
8. Oven dry sample at 105 degrees C for 24 hr and cool in a desiccation chamber.
9. Weigh the sample to within 0.01 g and record as the sample dry weight.
10. Indicate the quality, if known, of the sample volume measurement on the bulk density form. Examples include "lab measurement", "estimated from sample dimensions", "sample compacted and sheared", etc. Divide sample dry mass by sample volume and record as bulk density (g/cm^3). If the sample volume is not known to within 1 cm^3 and the sample is reasonably competent, follow steps 11-23.
11. Melt the paraffin so that it forms a liquid bath sufficiently deep to contain the entire

sample.

12. Determine the paraffin density and record it. This can be achieved by filling vials (with known volumes and dry weights) with the melted paraffin, weighing the filled vials, determining the weight of the paraffin within each vial, and then dividing those weights by the volume occupied by the paraffin. Paraffin density measurements should be repeated enough times to determine a mean and variance for the paraffin density.
13. Cut a 20 to 25 cm piece of very thin copper wire, weigh it to within 0.01 g, and record its dry weight.
14. Tie one end around the sample's center of mass so that the sample will not slip out while it is suspended from the bottom of the scale.
15. Weigh the sample and wire to within 0.01 g and record the weight
16. Dip the sample with the wire into the paraffin bath so that the sample (not the wire stem) is completely coated by paraffin. This may require multiple immersions. The objective is to obtain a waterproof coating.
17. Cool the sample, weigh it to within 0.01 g by suspending it from the scale's bottom hook, and record the weight as the sample + paraffin weight in air.
18. Weigh to within 0.01 g the paraffin-coated sample once again from the scale's bottom hook, but with the sample completely immersed in the water bath. The sample must not touch the walls of the water bath. Record the weight as sample + paraffin weight in water.
19. Observe whether the sample weight remains steady. If it remains steady, remove the sample from the water bath and dry it. If sample weight increases with time while suspended in water, water is imbibing into the sample through an opening in the paraffin coating, and the measurement is compromised. Carefully remove the paraffin coating and repeat steps 9-21.
20. Calculate sample volume (cm³): [(weight of sample and paraffin in air) – (weight of sample and paraffin in water) – (weight of paraffin/density of paraffin)]/water density
21. Calculate bulk density (g/cm³) = sample dry mass / sample volume

8.0 QUALITY ASSURANCE/QUALITY CONTROL

The laboratory scale should be calibrated using standard weights prior to using. Oven temperature should be checked and maintained to within 5 degrees C. Minimize sample handling and record all losses of sample mass (spalled grains, sample tipped and spilled, etc.)

9.0 DOCUMENTATION

Data fields to be captured using the bulk_density form in the database should include the following:

Hole_ID
Test_pit_ID
Field_ID

Elevation (ft)

Depth (m)

Date sample collected

Time sample collected

collector's initials

location (feature ID, UTM coords, description, etc.)

sampling method description

disturbed or undisturbed sample?

repacked sample? Y/N

sample dry weight (g)

sample dry weight w/ paraffin in air (g)

sample dry weight with paraffin in water (g)

paraffin weight (g)

paraffin density (g/cm³)

paraffin volume (cm³)

sample volume (cm³)

source of volume (measured in lab, estimated from bulk density, etc.)

sample bulk density (g/cm³)

source of bulk density (measured in lab, estimated from nuclear gauge, estimated from sand cone or other field method, etc.)

Lab ID

Date analyzed

SOP number

Deviation from SOP

Analyzed by (initials)

Comments

Reference

10.0 REFERENCES

Blake, G.R., and K.H. Hartge, 1986, Bulk Density, in *Methods of Soil Analysis Part 1. Physical and Mineralogical Methods*, Agronomy Monograph no. 9, Soil Science Society of America, Madison, WI, USA, p. 363-373.

Simunek, J., M. Sejna, and T. van Genuchten, The HYDRUS-2D software package for simulating the two-dimensional movement of water, heat, and multiple solutes in variably saturated media, Version 2.0, U.S. Salinity Lab, Agricultural research Service, U.S. Dept. of Agriculture, Riverside, CA, USA, April, 1999.

Zyvoloski, G. A., B. A. Robinson, Z. V. Dash, and L. L. Trease, Summary of the models and methods for the FEHM application – A finite-element heat-and mass-transfer code, Los Alamos National Lab. Rep. LA-13307-MS, Los Alamos, NM, 1997.

APPENDIX 1. FORM

Example of the current bulk_density_form:

bulk_density_form								
BULK DENSITY TABLE								
Sample_id	Sample_volume	Sample_wet_weight	Sample_dry_weight	Bulk_density	Analyst	SOP_number	Deviation_SOP	Laboratory_id
<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>

Record: 1 of 1