About the National Minerals Laboratory……

The National Minerals Testing Lab (AKA the Worland Field Office Mineral Testing Laboratory; the Worland Clay Lab, and others) was originally set up to test bentonite samples for mineral patent examinations. The lab has since then become an excellent national resource for agency use, and its services are historically intra-agency cost free. The acquisition of new equipment and software have expanded and adapted the focus and capabilities of the laboratory to meet the growing needs of the Bureau. This memorandum provides a summary as to the type of tests and analyses presently conducted in the laboratory, services offered, and types of equipment available for analytical work.

Tests described below can be modified to meet additional industry/academic criteria, as needed or requested. The laboratory may/might be capable of conducting special sample-specific tests/procedures upon request depending on the complexity of the desired test, the type of apparatus or equipment required, and/or the chemicals needed. Equipment/supplies costing over $1500.00 can be cost shared. Questions concerning the laboratory and its potential testing abilities should be addressed to Marilyn Wegweiser, email: marilyn_wegweiser@blm.gov. The laboratory phone number is 307-347-4135.

INSTRUMENTS FOR MINERAL/SPECIMAN IDENTIFICATION

Rigaku Miniflex X-Ray Diffractometer Model 2005: Two x-ray tubes are available for analyses: CuKα and Co. The X-ray tube fixed voltage is 30kV and the fixed current is 10mA. The XRD is computer driven using MDI Data Scan and Jade Search-Match software. A powder diffraction file (PDF-2) is used in conjunction with the Jade software for mineral identification. The PDF-2 data is obtained from the International Center for Diffraction Data and contains nearly 138,000 XRD patterns. Included in the PDF files are mineral and organic patterns. A quantitative analysis is possible if more than two mineral phases are present in the sample.

Optical Instruments

a) Leica Zoom 2000 Stereo Microscope: Stage scope; magnification range 10.5-4.5x.
b) Meiji EMZ Boom Stereo Microscope: Boom stereo microscope with 10x and 20x oculars available. Magnification range 0.07-4.5x.
c) American Optical Compound Microscope: This microscope is a rotating stage transmitted light scope with 4, 10, 45, and 100x objectives and has a 35 mm camera attachment.

Measuring Instruments

a) Mettler Balance P2010: readability 0.1g, capacity 2000g.
b) Mettler Balance PN1210: readability 0.01g, capacity 1200g.

c) Mettler Balance H80 (2 units): Readability 0.0001g, capacity 160g.
d) AMETEK TCL Strobe Light to adjust rpm spindle speed on mixers. Hooked to a rheostat and used to calibrate centrifuge as needed.
e) Brookfield LVT Viscometer
f) Fann 35A direct indicating variable speed viscometer
g) NL Baroid direct indicating variable speed viscometer
h) Methylene Blue Clay tester
i) Orion Benchtop pH/mv Meter

Sample Prep Instruments

a) Ro-Tap Sieve Shaker
b) Sorvall Model RC-3B Centrifuge
c) Waring Commercial Blender (20,000 rpm)
d) Waring Multimixers (800 rpm)
e) US Standard Sieves
f) Cincinnati Muller
g) Sand Rammer
h) Bico-Braun Chipmunk Jaw Crusher
i) Bico-Braun Direct Drive Pulverizer
j) Jones Sample Splitter

Ovens & Furnaces

a) Thelco Model 18: up to 450°F (consistent)
b) Despatch Double Oven: up to 550°F (consistent)
c) Thermolyne Type 1500 Furnace (for melting stuff): maximum 2000+°F

Mineral Identification (additional)

UV light: long & short wave

Reagents

For use of reagents not discussed below, call.

Rock & Mineral Specimens Prepared for Examination or Display

a) Hillquist 20-24 Slab Saw: This saw can accommodate rock sizes for cutting up to 15-inches by 9.5 inches. Larger rocks can be cut, depending on the rock shape.
b) Diamond Pacific Vibro Lap with a 20” bowl: after cutting, surfaces may be put through 3 stages of polishing using a series of grit designed to produce a highly polished face on a flat surface.

GENERAL SAMPLE PREPARATION AND ANALYSES

1. *General Sample Preparation*

a) Drying Ovens: The lab has three drying ovens: a Thelco Model 18, a Despatch Double Oven, and a Thermolyne Type 1500 Furnace. Samples can be heated to a variety of desired, timed, consistent temperatures ranging from 100°F to 550°F in the ovens, and from 0°-2200°F in the furnace.
b) Crushing: Bico-Braun Chipmunk Jaw Crusher
c) Pulverizing: A Bico-Braun Direct Drive Pulverizer with adjustable grinding plates; particle size can be reduced to less than 200 mesh.
d) Sample Volume Reduction: A Jones Sample Splitter having twelve ½" riffles is used to reduce large samples to workable test size samples as needed.
e) Distilled water: Distilled water is used in all analyses that require an aqueous solution. The lab manufactures its own distilled water.

2. *Bulk Density*

This test determines the density in lb/ft^3 or kg/m^3 of a granulated or powdered dry substance in its loose state. The test can be run on material as received, or on designated size fraction.

3. *Sieve analysis (grain size analysis)*

This test is used to obtain particle size distribution/fractions for analytical purposes. The number of particles that will pass through a given sieve is time dependent. Subsequent analysis can be either quantitative or qualitative. The lab has settling tubes that can be employed for the sedimentation analysis of sand-size particles, and a rotap machine is utilized to analyze gravel-and sand-size distributions. Sedimentation analysis requires that all the samples must be pre-treated and disaggregated to remove flocculating agents, cements, organic matter, soluble minerals, etc., in order to obtain an accurate reading of the grain size distribution of the residue of interest. USA Standard Test Sieves are used in sizes ranging from ¾" to a #500 or 25µ maximum diameter. Calculated data is compared with the data from known depositional environments. A comparison of the mean-median-mode grain size, sorting, skewness, and kurtosis is presented graphically. The purpose of this test is to determine what depositional environment your sample appears to be from.

4. *Sphericity (texture)*

The numerical values from the calculations for this quantitative measurement do not fall in to visually distinct categories, but become apparent when plotted on a Zingg diagram.

5. *Roundness (texture)*

A quantitative measurement taken from a 2-dimensional projected image When possible a consistent orientation is attempted with the sample, to provide a maximum cross-section.

6. ***Specific gravity***
   This measurement is first taken in air, followed by weight in water method using a beam balance. Weighing accuracy is to 0.01 gram.

7. ***Chemical fingerprinting***
   The laboratory is beginning collaborative projects that attempt to chemically fingerprint sediments so that provenance and stratigraphic locality can be determined. Call the lab for an update of what we can do.

BENTONITE AND SPECIALTY CLAYS

1. *Drilling Mud Properties*

Testing is done according to standard procedures set forth in American Petroleum Institute Specification 13A. "Bentonite" is used universally in all descriptions in this section, however, these tests can be run on any the clays used for drilling mud including but not limited to for example bentonite, attapulgite, and sepiolite.

a) Viscosity Test: This test measures the swelling properties of bentonite. The instrument used is a Fann 35A direct indicating variable speed viscometer or an NL Baroid direct indicating variable speed viscometer. Rotational speeds available on both instruments are 3, 6, 100, 200, 300 and 600 rpm. Using the viscometer readings the barrel yield, plastic viscosity, yield point/plastic viscosity ratio and gel strength of the material being tested can be calculated.

b) Filtrate or Water Loss Test: This test measures wall building properties of bentonite to prevent fluid loss. The test is conducted using a filter press consisting of cylindrical mud cells having an inside diameter of 3-inches and a height of at least 2.5 inches. A maximum of 120 psi of air pressure can be applied to the cell.

c) Grit: This test determines the amount of "non-clay" in a sample that is indicated by the +200-mesh (sieve screen; 200 squares/inch) or 75 micron (75 µ) material in a sample. Any screen size can be used to determine the percentage of grit in a sample if different parameters are desired. This test can be run on pulverized or non-pulverized (as received) samples. The major components of the grit can be determined using other tests available in the lab, if requested.

The property of bentonite viscosity indicates a sample's ability to aid in preventing well cuttings from sinking to the bottom of the hole during drilling. The cuttings suspend in the bentonitic drilling mud rather than sinking to the bottom of a well bore. The geochemical property of bentonite of "wall building" is that its ability to infiltrate formational strata intercepted while

drilling a well aids in the prevention of a loss of circulation during that drilling. Grit maximum in a sample is 4%.

2. Testing Effects of Adding Chemical or Organic Polymers

The purpose of adding chemicals or polymers to bentonite or other clays is to enhance the quality of low grade bentonite and/or to meet customer specifications. Amounts added are in accordance with industry guidelines. Reagents are on hand in the laboratory are sodium carbonate, the polymers Alcomer 1771 (for viscosity) and Alcomer 507 (for filtrate loss). Other reagents may be obtained if needed.

3. Blending

Blending is the mixing of different grade bentonite or other clays to produce a desired quality not inherent without the blending process. Information needed to make this a meaningful test is a final product with required specifications, blending ratio, and a sample of the bentonite or clay the test sample is to be blended with.

4. Bonding Properties

a) Plate Water Absorption: This test is used mainly by the taconite industry to determine if a bentonite will serve as a binder in the production of taconite pellets. Pulverized bentonite is placed on a filter paper which is then placed on a partially submerged sintered alumina plate in a closed container.

b) Methylene Blue Titration: This test is used primarily by the foundry industry where bentonite acts as a binder for holding sand molds together. Apparatus that is used is a titration setup called a Methylene Blue Clay tester consisting of a 50 ml buret, mixer, and ultra sound.

c) Green and Dry Core Strengths: This is a foundry test and involves mixing bentonite and sand to a certain moisture content, pressing the mixture into a core and then placing the cores on a strength measuring device which measures the force at which the core ruptures. The equipment consists of a Cincinnati Muller, Sand Rammer to make 2-inch diameter cores and a compression machine.

5. Hole Plug Material

Clay meeting certain criteria is used mainly for filling geophysical or water well holes.

a) Swell Test: A pulverized sample is used that has been screened to the required size fraction. The only equipment used is a 100 ml graduated cylinder and sieves.

b) Attrition Test: This test determines the toughness of granular material (final product) for packaging, handling, and transport. See the following test description for a discussion of the equipment used.

6. Attrition Test

This test determines the breakdown of granular substances such as clays, zeolites, etc. Results can be applied to floor absorbents, agricultural carriers, and cat litter. Test parameters, such as size of granular material and sieve sizes can vary between companies. Equipment necessary for this test includes a mechanical sieve shaker (Ro-Tap), variable screen sizes (US Standard Sieve), and chromium balls that measure ¼" and $^{5}/_{8}$" in diameter.

7. Specialty Clay Testing

This type of clay is generally defined as those clays used in a situation where montmorillonite (bentonite) does not meet the rheological or chemical properties that are required by the application. A Brookfield LVT Viscometer is used to accomplish testing. The instrument has four spindles (#1-4) and 8 speeds of 0.3, 0.6, 1.5, 3, 6, 12, 30, and 60 rpm. Sample preparation, slurry concentration, and spindle parameters are generally provided by industry.

8. Water or Oil Absorption Test

This test determines the fully saturated absorptive capacity of granular material and is applicable to floor absorbents and cat litter. Equipment presently used consists of a glass tube, 30 mm inside diameter, with an 18 mesh screen on the bottom, graduated cylinder, and a tripod stand. Test parameters such as size fraction to be tested can vary greatly as they are determined by industry.

9. Percent Clay Recovery

This procedure is used to separate small amounts of clay from gangue material for purposes of determining a recovery factor that is associated with calculating mining costs. The process uses a Waring Commercial Blender capable of 18,000-22,000 rpm and mixing can be done in either glass or stainless steel containers. Gangue and clay are separated using a Sorvall Model RC-3B centrifuge (refrigerated) which is equipped with four 1-liter buckets. The maximum speed is 6,000 rpm. Chemicals can be used to deflocculate the clay if necessary.

10. pH Determinations

The lab equipment for pH testing includes an Orion Benchtop pH/mv Meter, with digital readout using a Triode combination pH/automatic temperature control probe.

11. Moisture Determinations

This procedure calculates the % moisture in clay samples as received. The samples are heated at 220°F to a constant weight, cooled in a desiccator, and then weighed again. Samples are weighed on a Mettler PN1210 Balance with a 1/100th of a gram accuracy.

12. Gypsum

Gypsum purity is determined by using the Land Plaster Method. During the final stages of the test the sample is heated to 450°F, cooled in a dessicator, weighed, and converted to % gypsum using a land plaster conversion table.