Rock Properties Division

AGAT Laboratories

Oil Sands Tailings Analytical Pocket Guide

Geosciences

X-ray Diffraction (XRD)

SAMPLE REQUIREMENTS

- Outcrop, soil, grab, or chip samples
- Core samples
- Drill cuttings
- 0.5 - 1.0 g for bulk XRD, additional 2 g for clay differentiation

Destructive Test: YES

Testing Parameters

- Dry sample is lightly broken-up with mortar & pestle until grains are disaggregated
- Bulk XRD samples are micronized and mounted in a holder for analysis
- For clay analysis, crushed sample is suspended in distilled water and centrifuged to concentrate clays
- Clay fraction is prepped in various ways (dry, glycolated, HCl, high temp) and each analyzed to differentiate clays
- Mineral ID is by phase match to current ICDD database
- Proportional mineralogy by Rietveld Refinement or Relative Intensity Ratio (RIR)
- Cannot quantify non-crystalline material

Deliverables

- X-ray diffractogram
- Mineral identification
- Proportional mineralogy
- Sample prep and interpretation information
- Amorphous material content by internal standard is optional

Energy Dispersive X-ray (EDX) Analysis

SAMPLE REQUIREMENTS

- Outcrop, soil, grab, or chip samples
- Core samples
- Drill cuttings
- 0.2 - 1.0 g

Destructive Test: YES/NO

Testing Parameters

- Dry sample pulverized and homogenized
- Sample loaded into scanning electron microscope and bombarded with electrons
- Electrons cause emission of characteristic x-rays from elements present in sample, detector acquires emissions, proportion of each element present is calculated
- Broad area scan provides bulk elemental chemistry. Spot provides mineral chemistry
- Cannot detect elements lighter than carbon, accuracy improves with atomic mass

Deliverables

- Elemental chemistry of target material
- Area scans useful for scale analysis in conjunction with XRD, interpret non-crystalline material
- Spot analysis applied during SEM study to confirm mineralogy and particle compositions
- XRF profiling with elemental break-downs
- Modeled parameters include mineralogy, hardness, Poisson's Ratio, Young's Modulus

Thin Section (with Descriptions)

SAMPLE REQUIREMENTS

- Outcrop, soil, grab, or chip samples
- Core samples
- Drill cuttings
- 0.2 - 2.0 g

Destructive Test: PARTIALLY

Testing Parameters

- Samples are impregnated with epoxy, mounted to a glass slide and ground to 30µm thickness. Mineral staining can be applied, finish can be with cover slip or fine polish. Epoxy can be blue, clear, or Rhodamine-B for fluorescence petrography
- Petrologist examines slide for mineralogy, rock texture, porosity and mineralization systems, carries out 300-point counting using a mechanical stepping stage, takes photomicrographs micrographs at various scales
- Petrologist develops petrologic interpretation by considering data from all available sources

Deliverables

- Comprehensive petrological report with context-sensitive focus
- Petrographic description, images, point counting, mineralogy, and rock classification
- Reservoir quality and sensitivities
- Mineralization and beneficiation considerations

Scanning Electron Microscopy (SEM)

SAMPLE REQUIREMENTS

- Outcrop, soil, grab, or chip samples
- Core samples
- Drill cuttings
- 0.1 - 0.5 g

Destructive Test: NO

Testing Parameters

- Samples are cut perpendicular to bedding and then mounted to an aluminum base
- Alternatively samples can be prepared as polished thin sections
- Prepped samples are then sputter-coated with conductive gold alloy or carbon to prevent flaring or electrical charge build up which affects the images
- Samples are loaded into SEM instrument
- Samples are studied, with focus on textures, mineralogy, fabric and relative deportment of phases
- EDX spectrometry provides particle/mineral chemsitry
- Micrographs up to 20,000X are taken to illustrate findings

Deliverables

- Reservoir quality analysis
- Pore and pore throat structure
- Mineral deportment and reservoir sensitivities
- Mineralogy by morphology and EDX
- Unknown material ID, particle size distribution

QEMScan

SAMPLE REQUIREMENTS

- Outcrop, soil, grab, or chip samples
- Core samples
- Drill cuttings
- 5 - 10 g

Destructive Test: PARTIALLY

Testing Parameters

- Two primary analysis modes: Particle Mineral Analysis (PMA) and Field Scan (FS). PMA is for disaggregated samples (chips, cuttings, grains, crushed rock), and FS is for polished thin sections;
- For PMA the samples are mixed with epoxy, formed into a 30mm puck, and ground and polished. For FS a polished thin section or cut block is used
- The samples are scanned for BSE and EDX spectra using a pixel size appropriate for the rock grain size
- Mineralogy and textural data is interpreted by the QEMSCAN software and operator

Deliverables

- Quantitative mineralogy and size distribution, incl porosity if possible
- Mineral association, elemental deportment, specific or trace search
- Mineral Liberation analysis, porosity size distribution and associated mineralogy
- Mineralogical Field Maps (FS) or Particle Map Analysis (PMA)

Particle Size Analysis (LASER)

SAMPLE REQUIREMENTS

- Clean and dry sediment sample
- Sample crushed (if necessary)
- 3 - 8 g
- Can be run on water with suspended solids, if enough solids present

Destructive Test: YES

Testing Parameters

- Samples are passed through a 2 mm sieve, then disaggregated with a hot water ultrasonic bath, sodium hexametaphosphate, and manual disaggregation
- Subsample placed into test tube with D.I. water and loaded into laser diffractometer
- Sample particles flow in water suspension past laser, diffraction of laser beam due to particles is detected
- Software system interprets diffraction patterns into particle size distribution
- Detector reads from 0.39 to 2000 µm

Deliverables

- Histogram of particle size distribution in standard or custom channel sizes
- Cumulative curves (%-passing or retained)
- Statistical data (mean, median, mode, kurtosis, skewness, D50, other D-values)
- Raw data output and tables

Particle Size Analysis (SIEVE)

SAMPLE REQUIREMENTS

- Clean and dry sediment sample
- Sample crushed (if necessary)
- Minimum 30 g

Destructive Test: NO

Testing Parameters

- Samples are dry or wet (or combined) sieved through various mesh sizes (usually 18, 35, 60, 80, 120, 170, 230, 325, <325)
- Each sieve is then weighed to determine %-retained at each size
- Data is plotted and interpolated to determine D50, average, and other statistical data regarding the particle size distribution

Deliverables

- Histogram of particle size distribution in standard or custom channel sizes
- Cumulative curves (%-passing or retained)
- Statistical data (mean, median, mode, D50)
- Raw data output and tables

Geotechnical Services

Hydrometer Analysis

SAMPLE REQUIREMENTS

- 100 grams of raw, wet sample
- 50 grams of dried sample

Destructive Test: YES

Testing Parameters

- ASTM D422
- The sedimentation specimen is mixed with a dispersion agent and test water
- The slurry is allowed to condition, then is thoroughly mixed
- Readings are taken with a hydrometer at 8 specific time intervals over a 24 hour period
- A gradation curve (size distribution) is calculated for the <75um particle size

Deliverables

- Results are reported as particle diameter and percent finer for 8 points
- Results can be used to calculate silt/clay size fractions

Atterberg Testing

SAMPLE REQUIREMENTS

- 200 grams of raw, wet sample
- 100 grams of dried sample

Destructive Test: NO

Testing Parameters

- ASTM D4318
- The sample is processed to remove any material larger than No. 40 Mesh
- The Liquid Limit is performed by spreading the specimen in a brass cup, dividing the sample into two halves with a grooving tool, and allowed to flow together from the shocks caused by repeatedly dropping the cup
- The Plastic Limit is performed by rolling the soil into a 3.2mm thread, and remixing until the sample moisture is reduced to the point where the thread can no longer hold together
- The Plasticity Index is the difference between the Liquid and Plastic Limits

Deliverables

- Liquid Limit
- Plastic Limit
- Plasticity Index
- Casagrande Plot (upon request)
- Soil Classification (upon request)

Proctor Analysis

SAMPLE REQUIREMENTS

- Samples passing 3/8" sieve require 16 kg of raw sample
- Sample not passing 3/8" sieve require 29 kg of raw sample

Destructive Test: YES

Testing Parameters

- ASTM D698
- A soil at a selected molding water content is placed in three layers into a mold
- Each layer is compacted by 25 or 56 blows
- The resulting dry unit weight is determined
- The process is repeated for a sufficient number (5) of molding water contents to establish a relationship between water content and dry unit weight

Deliverables

- Up to 5 sub-tests each with a moisture content and a calculated dry unit weight
- A calculation of the maximum dry density and the moisture content at which that state is achieved

Cation Exchange Capacity (CEC) / Methylene Blue Index (MBI)

SAMPLE REQUIREMENTS

- 10 grams of hydrocarbon free soilds or paste

Destructive Test: YES

Testing Parameters

- Modified ASTM C837 (most recent version
- Methylene blue (MB) is a cationic dye that is adsorbed on clay mineral surfaces by a cation exchange reaction
- By acidifying the clay prior to titration with MB, the clay surfaces are of constant charge and the amorphous Fe_2O_3 retains positive surface charge and will not adsorb the cationic dye
- The amount of clay present is determined by the amount of MB adsorbed during the test. Non-clay minerals (i.e. quartz, feldspare, calcite, etc) have low CEC (cation exchange capacity)
- End point is determined by blotting a drop of the endpoint solution on filter paper and the resulting "halo" demarcation is an indication that the endpoint is reached

Deliverables

- Values are reported in miliequivalence (meq)

Hydraulic Conductivity (constant or falling head)

SAMPLE REQUIREMENTS

- A soil core of 7cm length by 3" diameter is required. Sample must be submitted in a Shelby tube for analysis. Typically 3ft long tubes are submitted. Lab will identify a suitable section of core and make the cuts for analysis

Destructive Test: PARTIALLY

Testing Parameters

- Lab will identify a 7cm section from Shelby tube that is suitable for analysis and cut/extract this section
- The chosen section of core is fastened into a permeameter cell. A Mariotte's tube is attached to the cell and delivers water to the soil core. A drain line is attached to the other end of the core
- The core is allowed to fully saturate for 5 days minimum, after which the water level on the Mariotte's tube is monitored
- Darcy's law is used to calculate the hydraulic conductivity of the soil core

Deliverables

- Hydraulic conductivity, k, of a soil core

Important Information

General Information

This pocket guide is specific to AGAT Laboratories Western Canadian Sedimentary Basin operations and lists some of the most commonly requested analyses. Please note that AGAT does provide customized programs based on our clients requirements and input. Should you have any questions or concerns, please don't hesitate to contact us at one of the phone numbers provided or email us at **info@agatlabs.com** for more assistance.

Sampling Guidelines

Each test listed in this pocket guide contains information regarding the sampling requirements for each test. It is always a good idea, if you're able, to get additional sample for possible QA/QC and additional testing you may want to do in the future.

Destructive Testing Categories

NO — Material can be reused for further testing.

YES — Material cannot be reused for further testing and may need to be disposed of after test completion.

PARTIALLY — Sample material where some of the rock is lost and what is prepped is irreversibly modified.

Rock /Fluid Evaluation

Capillary Suction Time (CST)

SAMPLE REQUIREMENTS

- Core or cuttings
- Sample crushed
- 5g for each fluid tested

Destructive Test

YES

Testing Parameters

- Sample is crushed and introduced to a fluid
- Clays that break down and swell will clog and inhibit the mixture's ability to flow, resulting in a higher CST value
- Fluids that minimize or prevent clay swelling will result in a lower CST value

Deliverables

- Capillary suction times in each fluid tested
- Graphical comparisons of each fluid and the CST values

Roller Oven Dispersion (RO)

SAMPLE REQUIREMENTS

- Core samples or large cuttings
- Sample crushed and sieved between 5 and 10 mesh screens
- 20 grams for each fluid tested

Destructive Test

YES

Testing Parameters

- Determines the dispersive properties of different fluids on a rock sample.
- The higher the mixed layer clays present in a sample the lower the sample recovery

Deliverables

- Rock recoveries in each fluid tested
- Graphical comparisons of each fluid and the roller oven values

Linear Swell Meter (LSM)

SAMPLE REQUIREMENTS

- Core samples or cuttings
- 1" diameter core plugs, trimmed
- Cuttings crushed to a powder (minimum 10 grams)

Destructive Test

YES

Testing Parameters

- This test is an effective way of analyzing the interaction between water based fluids in motion and crushed or intact rock samples
- The swelling characteristics are used to predict and manage the issues encountered during drilling and stimulating rock formations that are often unpredictable
- The higher the mixed layer clays present in a sample the higher the percent swell

Deliverables

- Percent swell in each fluid
- Short-term (0-5 minutes) and/or long-term (several hours) swelling due to clay-fluid interactions can be measured

Fluid Analysis

Soluble Ions Analysis (SI)

SAMPLE REQUIREMENTS

- 100 grams oil sands ore, or MFT material

Destructive Test

YES

Testing Parameters

- The oil sand is extracted with boiled (hot), de-ionized water (1:1 w/w extraction) and stirred for 5 minutes with an impeller stirrer
- The slurry is filtered through specially hardened filter paper (Whatman # 50) using a mud press
- A portion of the resulting aqueous filtrate is analyzed for pH and electrical conductivity (EC)
- The remaining filtrate is post-filtered through 0.1 µm Millipore filter and the final extract analyzed for anions (Cl^-, $SO4^{2-}$) by IC, and cations (K^+, Na^+, Mg^{2+}, and Ca^{2+}) by ICP/AES
- Electrode probes are used to measure the pH and EC of the sample extracts

Deliverables

- Cation (Na, K, Mg, Ca), anion (Cl and SO4) concentrations. Bicarbonate and carbonate concentrations can also be analyzed if required

Water Chemistry (includes Naphthenic Acids)

SAMPLE REQUIREMENTS

- Water Chemistry: 250mL of water
- Naphthenic Acid: 250mL of water

Destructive Test

YES

Testing Parameters

- Water Chemistry: pH, EC and alkalinity (including bicarbonate and carbonate) are reported by an autotitrator. These parameters can also be measured manually for highly contaminated samples. Cations concentrations are determined via ICPOES analysis and anions by IC analysis
- Napthenic Acids: Naphthenic Acid by FTIR

Deliverables

- Water Chemistry: pH, EC, alkalinity (inc CO_3, HCO_3), dissolved Na, Ca, K, Mg, Fe, Mn, Cl, F, NO_2, NO_3, SO_4, Hardness, calculated TDS and Ion Balance can be calculated and reported if required
- Naphthenic Acids: Naphthenic Acids only.

Polyacrylamide Analysis

SAMPLE REQUIREMENTS

- A 250 mL water sample contained in a glass container

Destructive Test

YES

Testing Parameters

- A standard curve of known concentrations of polyacrylamide is first made and the linear relationship is used to determine the unknown concentration of polyacrylamide in the sample

Deliverables

- Polyacrylamide concentration in the sample

Specific Gravity by Water Pycnometry

SAMPLE REQUIREMENTS

- Sample can be dry or wet;
- The minimum amount of dry material a sample must contain is 45 grams; optimal: 100 grams

Destructive Test

NO

Testing Parameters

- ASTM D854-14.
- The sample is processed to remove any material larger than 4.75 mm
- Distilled water is mixed with the sample to form a slurry. If the as-received sample is dry, it is mixed in a calibrated volumetric flask. For a wet sample, it is mixed prior to loading into the flask
- The slurry is placed on a hot plate to boil for at least 2 hours to remove entrapped air.
- The slurry is cooled to a stable temperature. Distilled water is added to the flask to the volumetric mark. Mass of the pycnometer is measured
- The mass of dry sample is determined by drying the above slurry at 110±5°C

Deliverables

- Specific Gravity of soil passing 4.75 mm sieve at 20 °C
- Percent of sample passing 4.75 mm sieve
- Mass record

Specific Gravity

SAMPLE REQUIREMENTS

- A minimum of 50 mL of sample is required for this analysis

Destructive Test

NO

Testing Parameters

- This analysis determines the density of the water by use of a hydrometer.
- Alternative methodology is available through useage of an automatic densitometer

Deliverables

- Absolute density and specific gravity of the sample

Geochemistry

ED-XRF

SAMPLE REQUIREMENTS

- Outcrop, soil, grab, or chip samples
- Core samples
- Drill cuttings
- 1-5g

Destructive Test

NO

Testing Parameters

- Material is scanned using a handheld XRF instrument
- For core a scan can be taken at regular intervals along the core length, often 5cm spacing
- Cuttings and loose material can be mounted for scanning non-destructively
- Lightest detectable element is sodium
- Best practice and highest data quality are achieved with control samples analyzed by WD-XRF and XRD for precision geochemistry and mineralogy
- Cuttings samples should be crushed and homogenized prior to testing

Deliverables

- Elemental chemistry of each data point scanned
- For cores an elemental profile along the well is generated
- Mineralogy, geomechanical properties and other petrophysical properties can be modeled

WD-XRF

SAMPLE REQUIREMENTS

- Outcrop, soil, grab, or chip samples
- Core samples
- Drill cuttings
- 10-12 g

Destructive Test

YES

Testing Parameters

- Concentrations of major elements are measured on beads prepared with thermal fusion methods
- Concentrations of trace elements are measured on pressed pelletes prepared with a hydraulic press on powdered samples

Deliverables

- Elemental chemistry
- Major elements: Na, Mg, Al, Si, P, S, K, Ca, Ti, Mn, Fe (wt %)
- Trace elements: Sc V Cr Co Ni Cu Zn Ga As Rb Sr Y Zr Nb Mo Sn Sb Cs Ba La Ce Pb Th U Cd (ppm)
- Loss on Ignition (LOI)

Bitumen Characterization and Quanitification

Bitumen Characterization (Viscosity)

SAMPLE REQUIREMENTS

- 50 mL of oil

Destructive Test

YES

Testing Parameters

- Determination of viscosity by Brookfield Rheometer at a temperature range up to 190°C

Deliverables

- Viscosity at desired temperature profile

Bitumen Characterization (API and Densities)

SAMPLE REQUIREMENTS

- 50 mL of oil

Destructive Test

YES

Testing Parameters

- Density and Relative Density of liquids by pycnometer method

Deliverables

- Density in g/cm^3

Oil Extraction and Quantification (Dean Stark Analysis)

SAMPLE REQUIREMENTS

- 100 grams of sample is used for Dean Stark Analysis

Destructive Test

PARTIALLY

Testing Parameters

- The samples are weighed and then placed into glass pots containing boiling toluene solvent. Vaporized water is condensed and measured.
- The toluene will continue to reflux until all hydrocarbons are removed from the sample (minimum 12 hours).
- The residual sample weight is used to calculate the oil and water saturations

Deliverables

- Oil and water saturations

Our Locations

Oil Sands Tailings Analytical Laboratory
3801 21st Street NE
Calgary, Alberta
T2E 6T5
403-299-2077

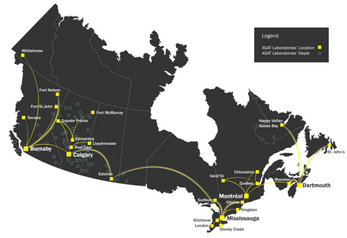
Geosciences and Geotechnical Laboratory
106, 2730 39th Ave NE
Calgary, Alberta,
T1Y 7H6
587-391-2540

Environmental Sciences Laboratory
2910 12th Street NE
Calgary, Alberta
T2E 6V6
403-299-2000

For more information:
Email: Info@agatlabs.com
Website: www.agatlabs.com

PROUDLY CANADIAN
100% CANADIAN OWNED AND OPERATED

With over 40 locations from Burnaby, BC to St. John's, NL we are Canada's largest laboratory network.



Version 3 - Jan 2022