

IOSI Laboratory Facilities and Equipment

Contact for current availability, service or training:
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1. IOSI laboratory covers a broad range of research support to oil sands projects from standard techniques for routine analysis to advanced techniques at molecular level for fundamental research.
2. Example applications utilizing some of the equipment in oil sands research are listed in the next a few slides. Please note that the examples are only part of the capacity of IOSI laboratory. Please consult IOSI technical staff regarding the full capacity of equipment tailored for your particular application.
3. Brief introduction of each equipment is listed in the document based on the alphabetical order.

Example Application in Oil Sands Extraction

- Interaction of solvent (e.g. cyclohexane or water) with oil sands components
 - Solvent vapor: Intelligent Gravimetric Analyzer (IGA, Hidden Isochema). Microbalance; sample: 50 mg – 2.0 g; gas vapor: up to 95% partial pressure of solvent with balance inert carrier gas, $T = 5 - 400$ °C; $P = 0.1$ to 2.0 MPa.
 - Liquid solvent: Quartz Crystal Microbalance with Dissipation (Q- Sense Analyzer E4, Biolin Scientific). Measurement of the changes of resonant frequency and energy dissipation; various sensors; $T = 15 - 65$ °C; ambient pressure.
- Determination of residual solvent content in oil sands gangues (IOSI protocol):
 - Gas and vapor analyzers - Dynamic Sampling Mass Spectrometry (Hidden Analytical) coupling with thermal desorption unit.
 - Gas and vapor analyzers - HPR-20 QIC MS (Hidden Analytical) coupling with thermal desorption unit.
 - Mass spectrometry based technique with mass range of 1 -200 amu and detection limit of 10 ppb.
- NAE Bitumen recovery determination (IOSI protocol)
 - Organic Elemental Analyzer – CHNS/O (Thermo Flash 2000). Oil sands gangues: 10-50 mg; measurement time: < 15 min.
- Simulated NAE process at high temperature and pressure (IOSI protocol)
 - Accelerated solvent Extraction (Dionex ASE 300). Cell volume = 100 mL; $T = RT - 200$ °C ; $P =$ ambient – 10MPa; N_2 as purge gas.
- Quantitative mineral analysis of oil sands samples:
 - Powder X-Ray Diffraction with RockJock method.

Example Application in Product Cleaning and Partial Upgrading

- Aggregation behavior of bitumen components in solvent or bitumen phase:
 - Isothermal Titration Calorimetry (ITC) (TAM III, TA Instruments). Direct measurement the heat; Cell volume: 20 mL; T: 15 – 150 °C; P: ambient pressure.
 - Dynamic/Static Light Scattering (ALV/CGS-3 Goniometer). Measurement of light intensity; Cell volume: 2 mL; T: - 6 – +150 °C; P: ambient pressure)
- Interfacial behavior of bitumen components at liquid/liquid and air/liquid interfaces:
 - Brewster Angle Microscope (Nanofilm, EP3) with Langmuir Trough. Visualization of monolayer and films at the air/liquid interface to understand both the structure and flow behavior of materials; field of view = 3600 μm x 4000 μm ; image resolution = 12 μm .
 - Interfacial Shear Rheometer (KSV). Studying the viscoelasticity of films at liquid/liquid interface; dynamic moduli resolution: 0.001 mN/m; frequency range: 0.0016-1.6 Hz.
- Interactions of bitumen components and minerals substrates:
 - Quartz Crystal Microbalance with Dissipation (Q- Sense Analyzer E4, Biolin Scientific). Measurement of the changes of resonant frequency and energy dissipation; various sensors; T =15 – 65 °C; ambient pressure; liquid media.
 - Ellipsometer (Sopra). Measurement of the refractive index and the thickness of semi-transparent thin films by means of the reflected light waves; thickness range: 0.1 nm – 5 μm ; ambient conditions.
 - Atomic Force Microscope (Bruker Innova). High-resolution imaging of topology of materials in air and liquids media; up to atomic resolution; ambient conditions.
 - Confocal scanning microscope (Zeiss Axio CSM 700). Measurement of the 3D topography over large sample areas as well as roughness with high lateral resolution (0.16 μm); ambient conditions.

Example Application in Tailings Process Fundamentals

- Solids/liquid separation:
 - Filter press filtration (SERFILCO 0.02-7PPHM Lab Press). Sample: 1.5 -2.0 kg; thickness of cake: 2.5 cm; P: up to 690 kPa; direct measurement of dewatering rate.
 - Pressure filtration unit (EMD Millipore). Sample: 0.5 kg; P: up to 690 kPa with air; direct measurement of dewatering rate.
- Flocculants optimization:
 - Sedirack (Cetten Instruments). Measurement of the sedimentation rate of particulate solid suspensions in liquids with flocculants; 5 x 2 L columns; on-line monitor of mud line with software.
 - FBRM G400 (Mettler Toledo). Real-time monitoring of changes in floc density and floc size of oil sands tailings (up to 30 wt% solids content); measurement range: 0.5 – 2000 μm ; T: 10 – 90 °C; ambient pressure.
- Evaluation treatment performance:
 - Capillary Suction Timer (Triton Electronics). Measurement of the rate at which water is released from treated oil sands tailings.
 - Rotational Rheometer (Kinexus lab+, Malvern) with vane shear tool. Measurement of yield strength of treated oil sands tailings with standard method.
- Interaction of flocculants with mineral solids at molecular level:
 - Isothermal Titration Calorimetry (ITC) (TAM III, TA Instruments). Direct measurement the heat; Cell volume: 20 mL; T: 15 – 150 °C; P: ambient pressure.
 - Quartz Crystal Microbalance with Dissipation (Q- Sense Analyzer E4, Biolin Scientific). Measurement of the changes of resonant frequency and energy dissipation; various sensors; T =15 – 65 °C; ambient pressure.
- Quantitative mineral analysis of oil sands tailings samples:
 - Powder X-Ray Diffraction with RockJock method.

Accelerated Solvent Extraction (Dionex ASE 300)



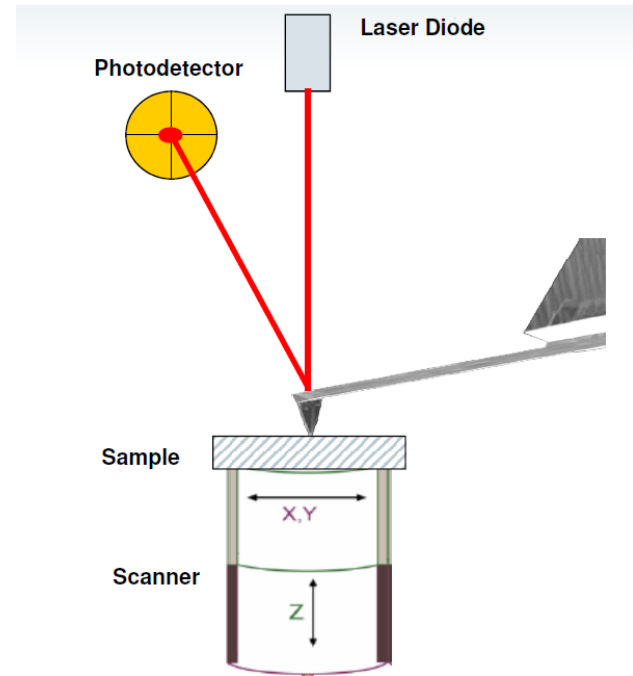
- A technique similar to traditional Soxhlet or sonication but at high pressure and temperature.
- Extraction of compounds from solid and semi-solid samples quickly with small solvent volume.
- Cell size: 100 mL.
- Operation pressure: up to 1500 psi.
- Operation temperature: up to 200 ° C.

Acoustic & Electroacoustic Spectrometer (DT-1200, Dispersion Technology)



- Measure acoustic attenuation spectrum to determine particle size distribution. Size range: 0.005-1000 μm .
- Use electroacoustic technique to determine zeta-potential.
- Applications in coagulation and flocculation of concentrated fluid systems (with particle concentration up to 50 vol%).

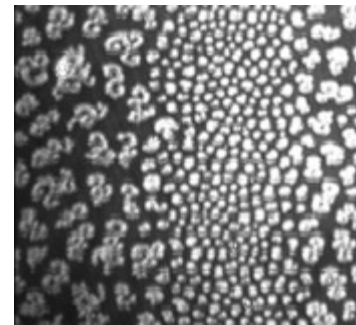
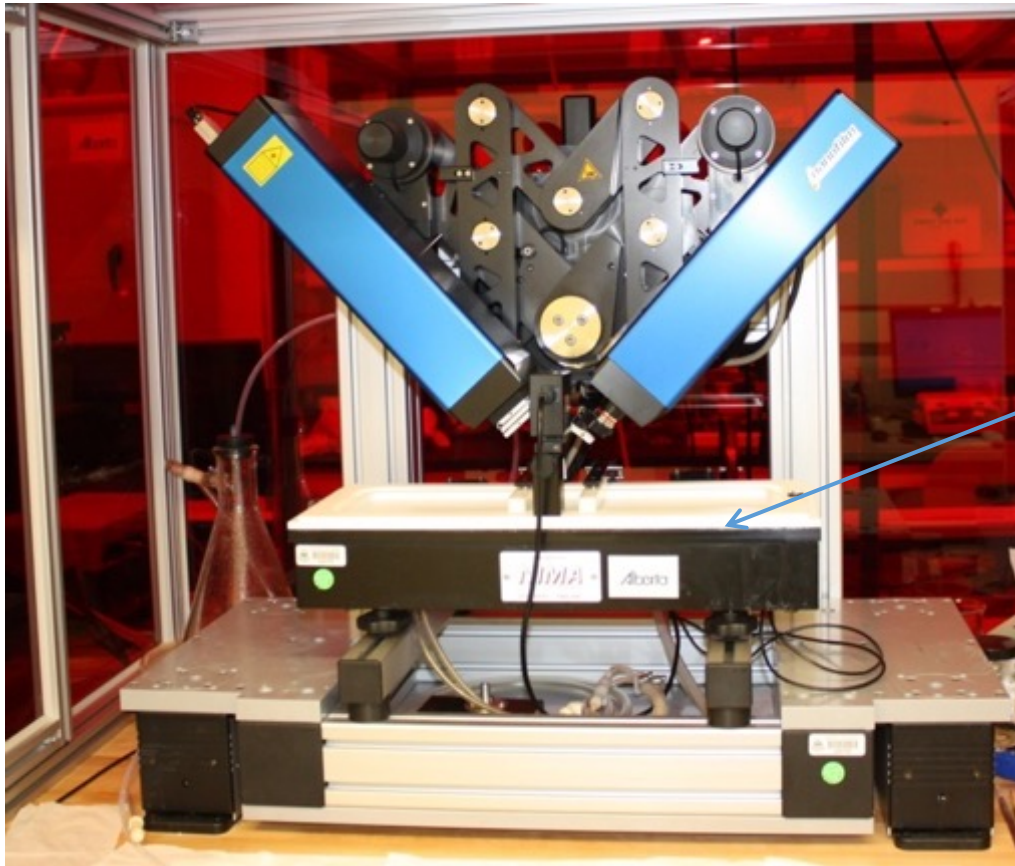
Atomic Force Microscope (AFM) (Bruker Innova)



- Routine high-resolution imaging in air and liquids (up to nanometer resolution).
- Modules: contact and tapping.

Brewster Angle Microscope -BAM (Nanofilm, EP³)

- Use microscope to record the p-polarized reflectance of the sample film at the Brewster angle of the substrate
- Light source: 532 nm green laser
- Measure the film thickness and topography of thin films on a dielectric substrate

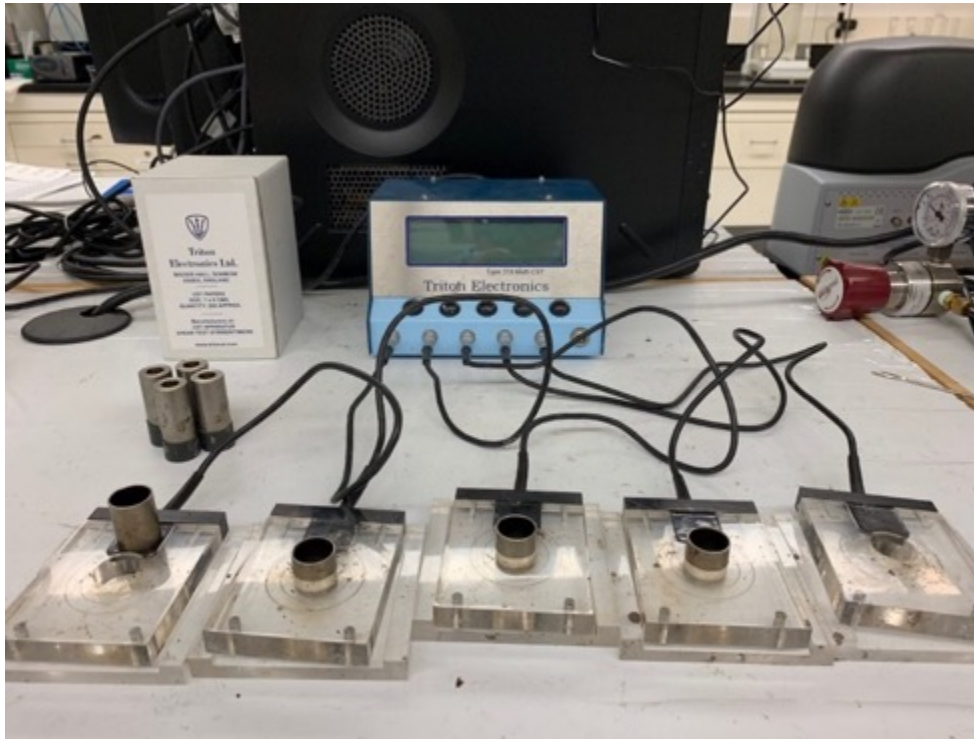


BAM image of DPPC thin film at air/water interface when surface pressure was at 10mN .

Langmuir Trough (Nima)

- Measure surface pressure of thin film at air/water or oil/water interface.

Capillary Suction timer (Triton Electronics)



- A simple and precise measure of the rate at which water is released from a sludge matrix.
- Capillary suction time (CST) of sludge with 5 multi-radii heads.
- Studying filterability of sludge and evaluating the effects of chemicals and process conditions of sludge treatment.
- Example application: oil sands tailings treatment; wastewater treatment.

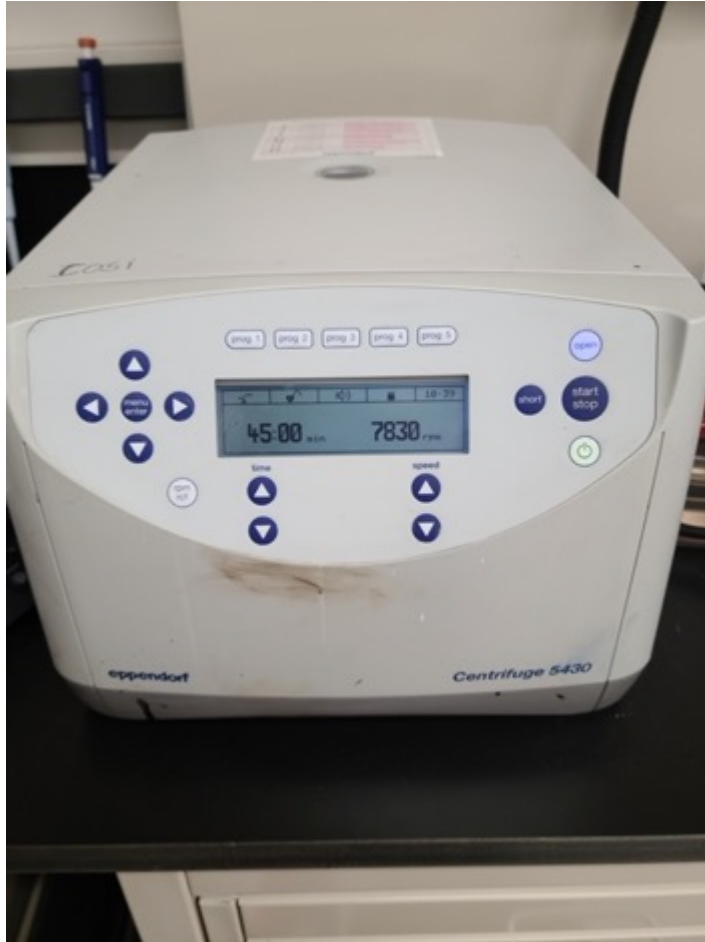
Centrifuge (high speed, Avanti J-30I, Beckman Coulter)



- Swinging-bucket and fixed-angle rotors provide maximum separation forces in excess of 100,000 x g at speeds up to 30,000 rpm.
- 4.0 L max capacity.
- Temperature range: -20 ° C to +40 ° C.



Centrifuge (Eppendorf 5430)



- Can handle 50ml/15ml/5ml sample sizes
- Max RCF : 7197 for F-35-6-60 Rotor
- Max RPM : 7830 for F-35-6-60 rotor
- Max RCF : 20817 for FA-45-30-11 Rotor
- Max RPM : 14000 for FA-45-30-11 Rotor

Centrifuge (Eppendorf 5424)



- Small sample sizes only, 5ml Max
- Max RCF : 21130
- Max RPM : 15000

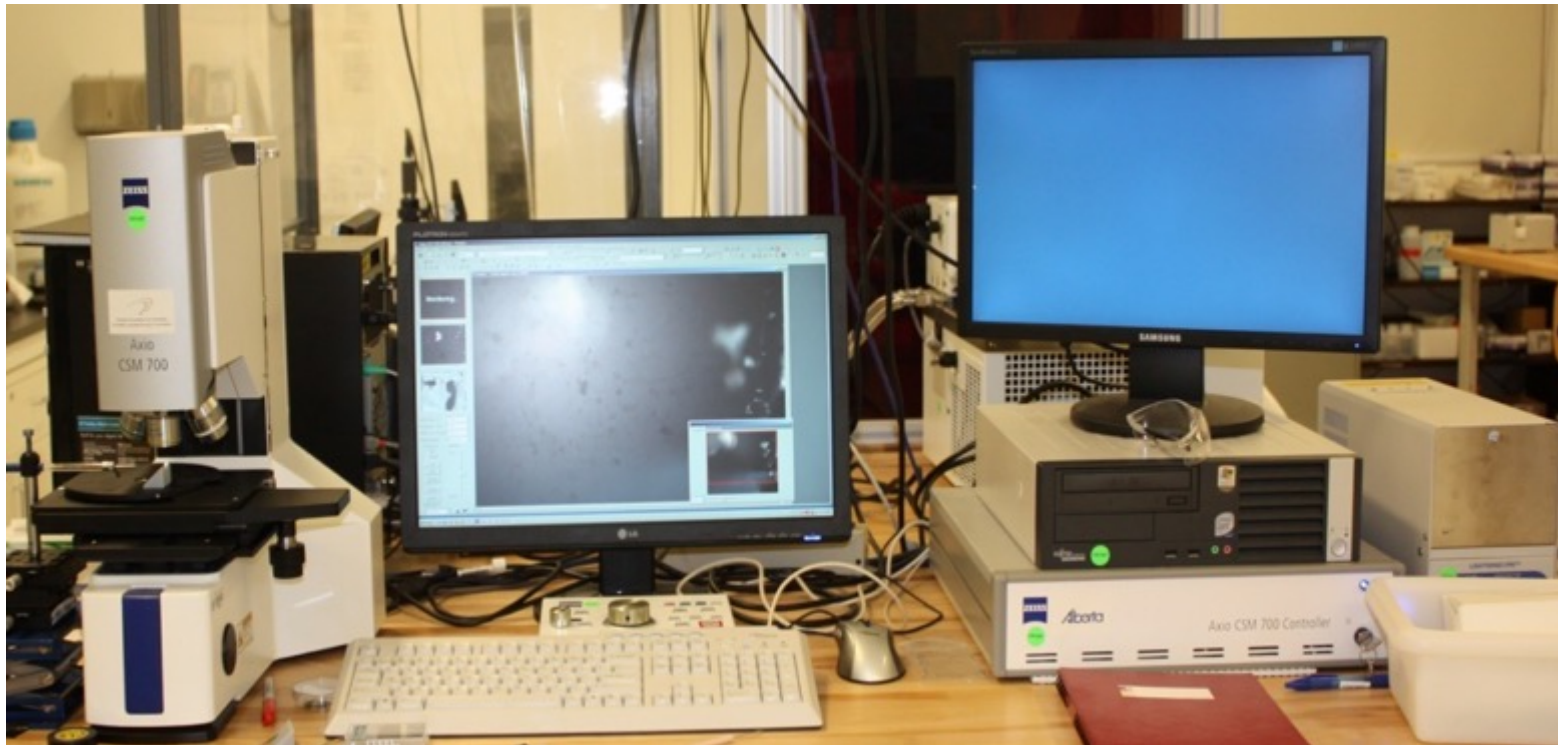
Confocal Fluorescence Microscope and Optical Tweezer (Quorum Technologies and MMI)



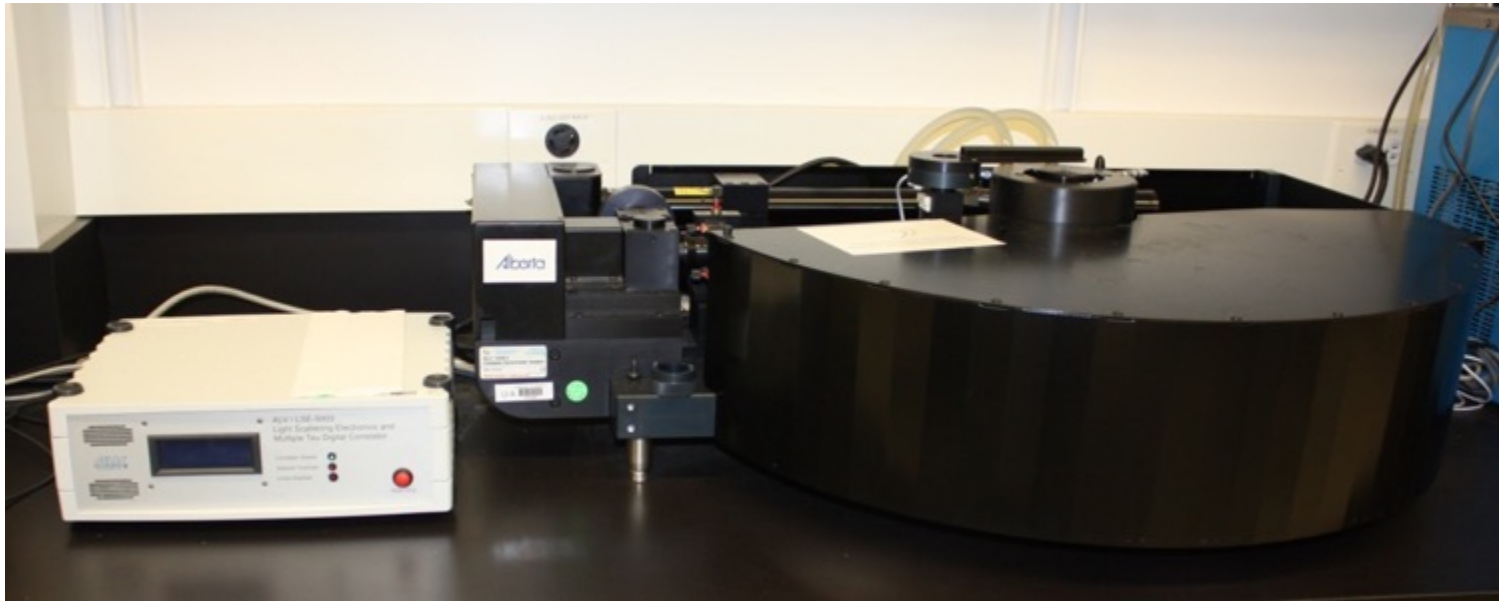
- Visualization, explorations, and analysis of multi-channel high-performance 3D fluorescence imaging with three excitation lasers and four spectral emission bands.
- Attached to the confocal is the MMI Optical Tweezer which combines the capabilities of laser technology and microscopy for contact-free microscopic particles manipulations.

Confocal scanning microscope (Zeiss Axio CSM 700)

- High acquisition speed (7.5 fps, high-speed color mode).
- True colour confocal microscopy.
- High resolution (Lateral resolution 0.16 μm).
- Optical 3D profilometer.



Dynamic/Static Light Scattering (ALV/CGS-3 Goniometer)



Dynamic light scattering (DLS)

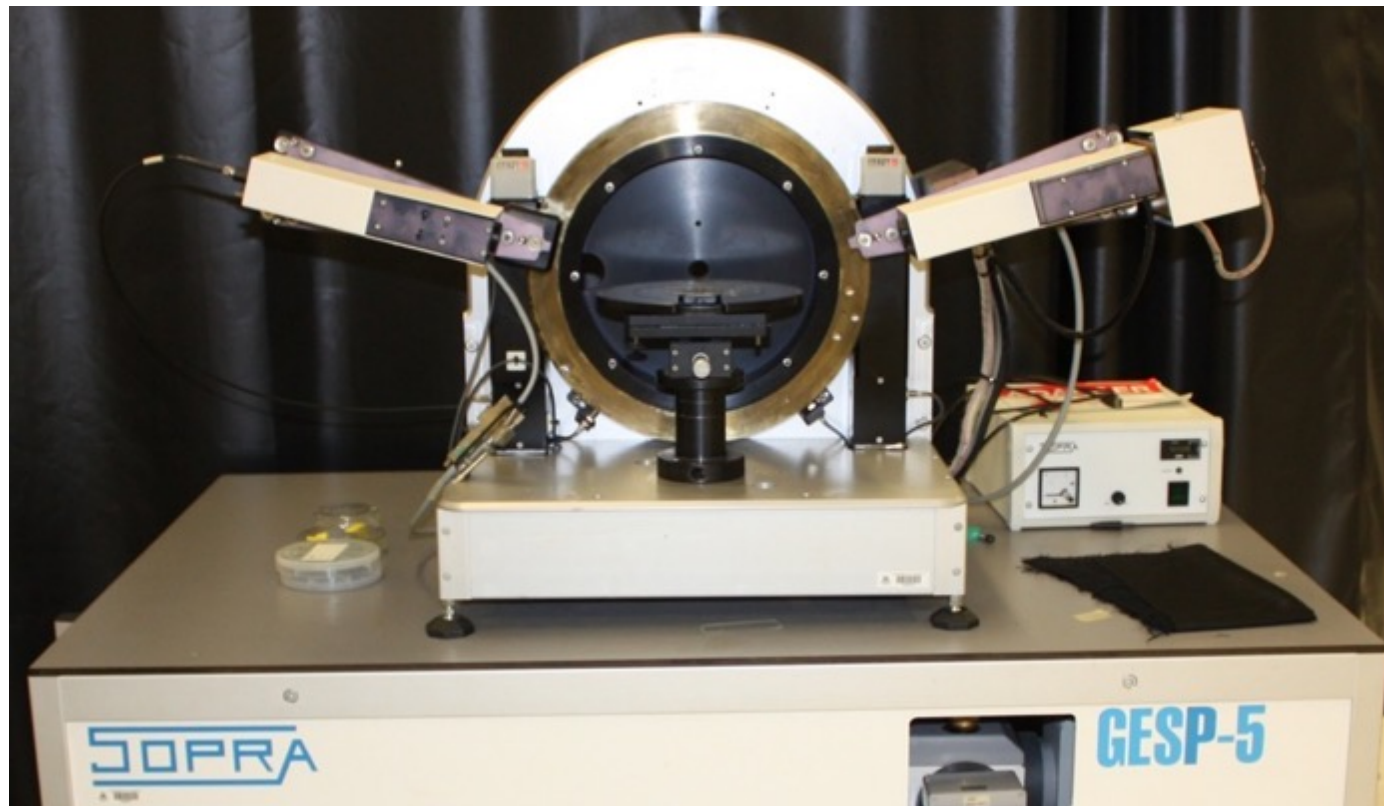
- Measure intensity fluctuation of scattered light
- Particle size distribution
- Hydrodynamic radius (R_H)
- Diffusion coefficient (D)

Static light scattering (SLS)

- Measure light intensity at variable angles
- Polymer molecular weight (M_w)
- Radius of gyration (R_g)
- Second virial coefficient (A^2)

Ellipsometer (Sopra)

- A non-contact , non-destructive optical technique
- Spectroscopic ellipsometry (wave length range: 210 nm to 900 nm)
- Measure the change in polarization of light beam upon reflection from a sample.
- Determine: film thickness and refractive index.



Environmental Chamber (Associated Environmental Systems)



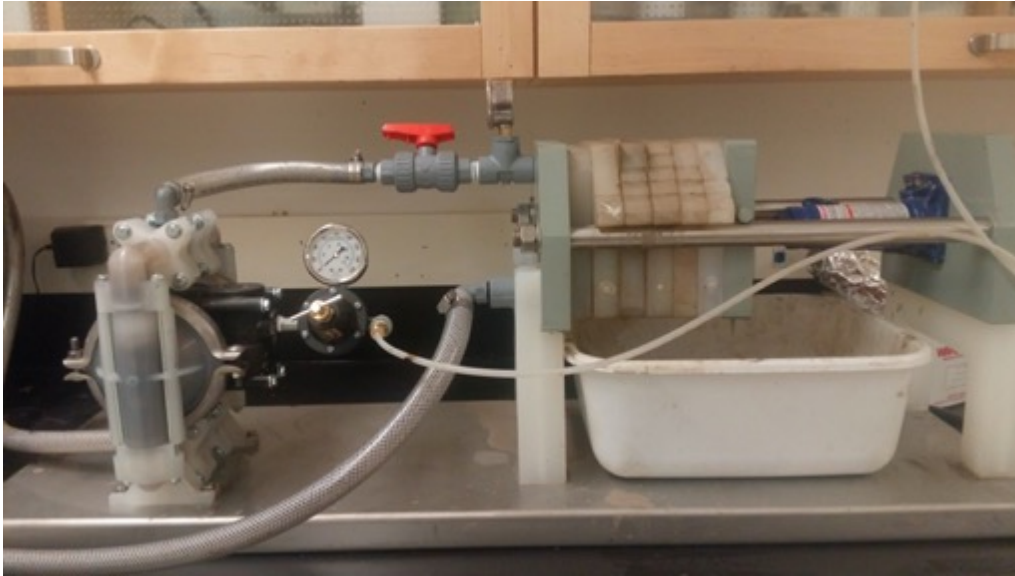
- Benchtop test chamber provides steady-state temperature and humidity environmental testing on samples.
- Working volume: 1.3 cu ft.
- Temperature range: 5 ° C to 94 ° C.
- Humidity range: 10% to 98% relative humidity.
- Example application: Sample treatment.

FBRM G400 (Mettler Toledo)

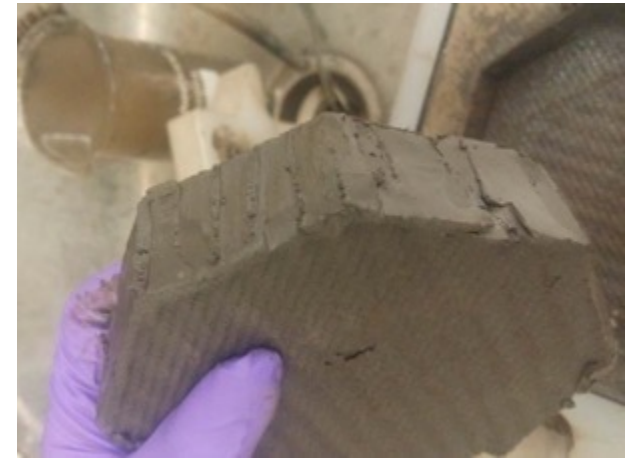


- Track real-time changes in floc density, floc size and morphology directly in the process.
- Characterize flocs and their constituents from 0.5 μm to 1mm.
- Maximum sample concentration: up to 30 wt% solids in aqueous and non-aqueous media.

Filter Press Filtration (SERFILCO 0.02-7PPHM Lab Press)



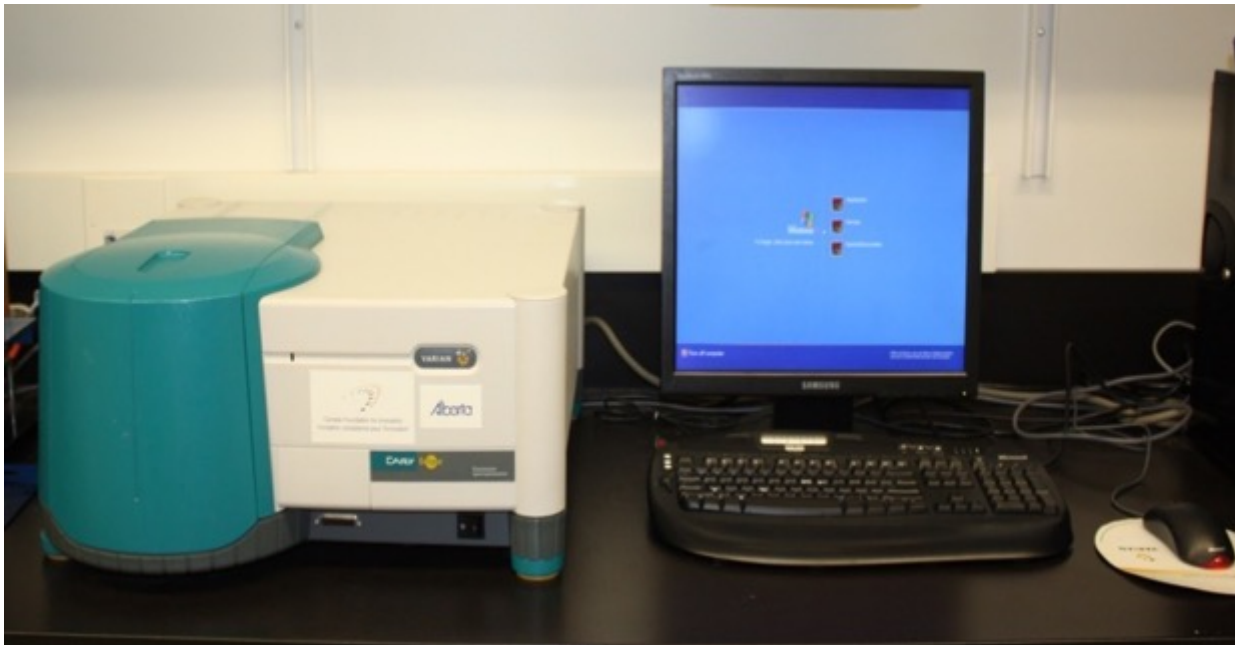
Solid cake formed from treated MFT



- A piece of batch operation, fixed volume equipment that separates liquids and solids using pressure filtration.
- A slurry is pumped into the filter press and dewatered under pressure.
- Air diaphragm pump: 620 kPa; chamber volume: 280 cm³ with cake thickness of 2.5 cm; total filter area is 220 cm²; filter cloth: 15 µm mesh polypropylene; feed flow rate adjustable up to 15.7 L/min.
- Example application: oil sands tailings dewatering.

Fluorescence Spectrophotometer (Varian Cary Eclipse)

- Wavelength ranges 190-1100 nm.
- Liquid sample measurement (FEEM and Synchronous Fluorescence).
- Solid sample measurement.
- Optical Fiber Accessory for in-situ measurement.



Solid sample holder



Fibre optic coupler

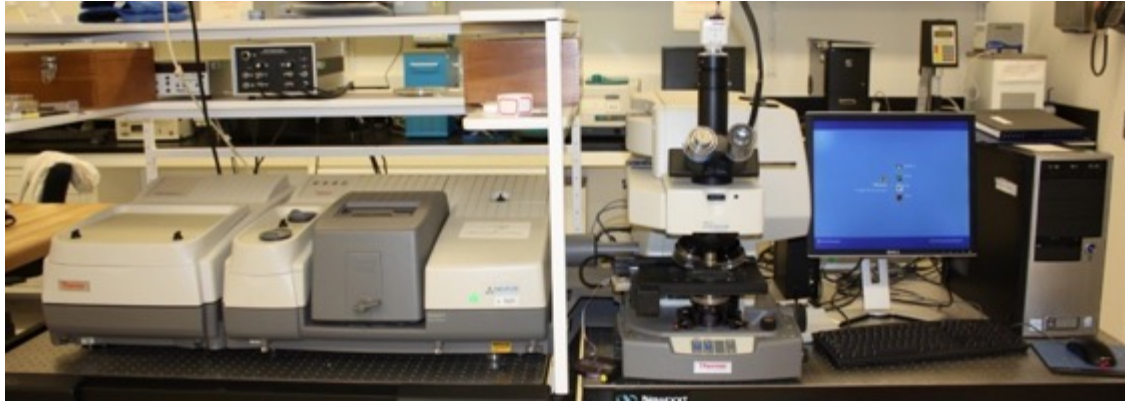
Force Tensiometer (Krüss K100)



- Based on the precise measurement of the force which occurs when wetting a measuring probe or a solid sample.
- Surface tension and interfacial tension using the ring, plate and rod method.
- Contact angle and surface free energy of solids, powders or fiber bundles.
- Sedimentation behavior of dispersions in aqueous and non-aqueous media.
- Density of liquids and solids.

FTIR Spectrometers (Thermo)

FTIR Microscope



FT-IR (Nicolet 6700)



- FTIR Microscope: Transmission, Reflection, ATR , FTIR Mapping.
- Spectrometer: Transmission, Variable angle reflection, Single and Multi – Bounce ATR, Variable angle reflection, Diffuse reflection (DRIFTs).
- Photoelastic Modulator (PEM) : Polarization modulation-infrared reflection-adsorption spectroscopy (PM-IRRAS).

Gas and vapor analyzers (Hiden Analytical)

Dynamic Sampling Mass Spectrometry (DSMS)



Benchtop HPR-20 QIC gas analyzer with thermal desorption unit



- Multi-component, multi-stream off-gas analysis by mass spectrometry.
- Measure the concentration of gases and organic vapors in real time quantitatively.
- Triple filter quadrupole mass spectrometer.
- Mass range: 1- 200 amu.
- Detection limit: > 10 ppb.

Gas Chromatograph (Agilent 7890A GC)



- Identification of components in mixture for both qualitative and quantitative analysis.
- Breakthrough Capillary Flow Technology enables leak-free in-oven connections, enhances productivity and data integrity via backflushing and offers versatile, robust solutions for complex GC analyses.
- Low Thermal Mass (LTM) technology provides direct rapid heating and cooling of capillary columns for extremely fast analytical cycle times and higher productivity
- Temperature range: 50 to 450 ° C.
- Detectors: FID, NCD.

GC-MS (Trace GC Ultra coupling DSQ II Single Quadrupole MS, Thermo)



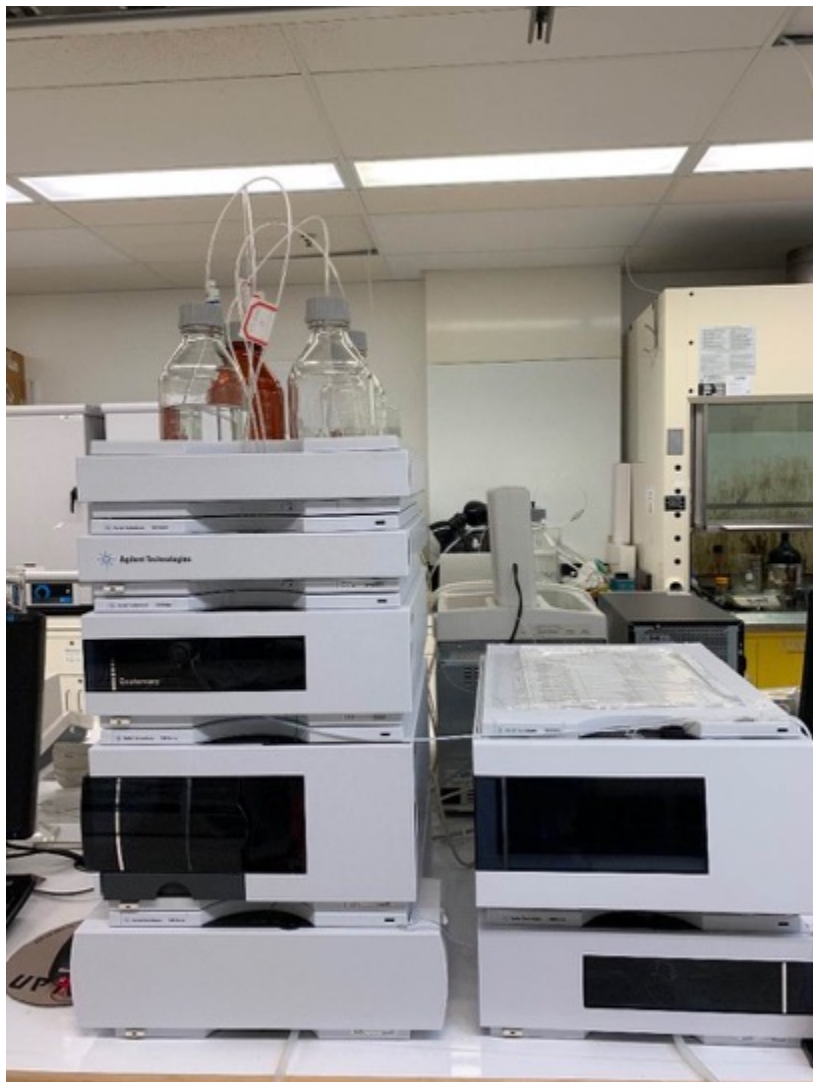
- An analytical instrument combines the features of gas-chromatography and mass spectrometry to identify different substances within a test sample.
- GC: Detector: FID. Oven temperature: up to 450°C with ramp rate of $0.1 - 120^{\circ}\text{C/min}$.
- MS: Single Quadrupole MS. Source: DuraBrite. Mass range: 1 to 1050 amu.
- Example application: petroleum, pharma materials.

Glovebox workstation (UNIlab Plus, MBRAUN)



- Storage and handling air/water sensitive materials and experiment.
- Argon atmosphere.
- Attainable purity level:
 $\text{H}_2\text{O} < 1 \text{ ppm}$, $\text{O}_2 < 1 \text{ ppm}$.

HPLC 1200 (Agilent)



- High performance liquid chromatography (HPLC) is a technique in analytical chemistry used to separate, identify, and quantify each component in a mixture.
- Detector: UV-Vis (190-950 nm).
- Quaternary pump.
- Operating pressure: up to 40 MPa.
- Injection volume ranges from 0.01 μL to 2000 μL with a precision levels of $< 0.5 \%$ RSD.

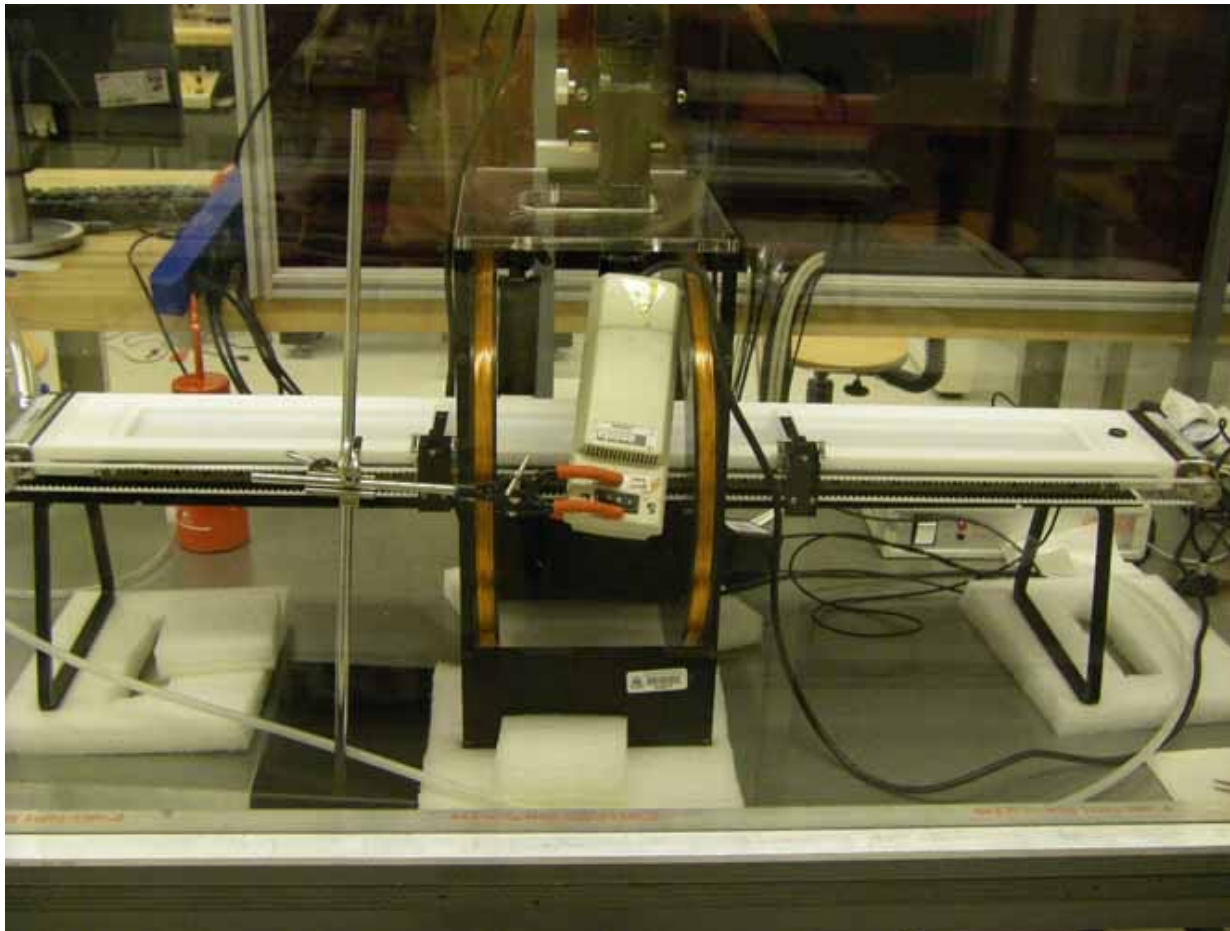
Intelligent Gravimetric Analyzer (IGA, Hiden Isochema)



- A microbalance coupled to two vapour generators to provide controlled solvent atmospheres.
- Measurement of isotherms and the corresponding kinetics of sorption and desorption for set pressure or partial pressure steps at different temperature.
- Sample loading range: 50 mg – 2.0 g.
- Temperature range: 5 – 400 ° C.
- Pressure range: 0.1 – 2.0 MPa.

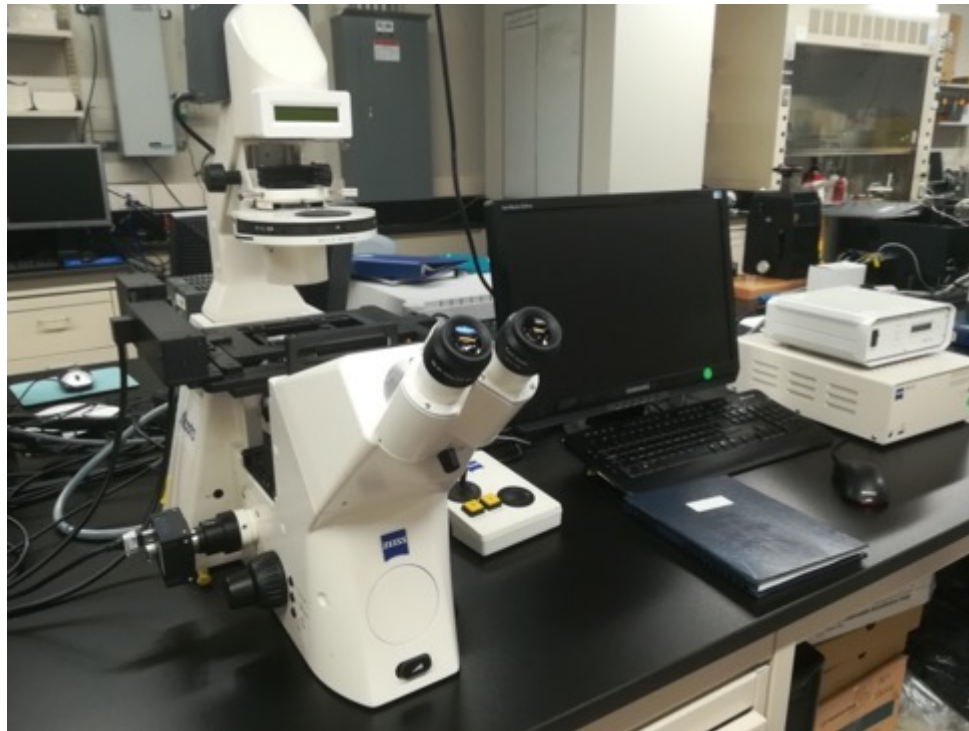
Interfacial Shear Rheometer (KSV)

- Measurement of the viscoelastic properties of film at fluid interfaces (air/liquid and liquid/liquid).
- Viscoelastic properties and surface pressure can be measured simultaneously.



Inverted Microscope Zeiss Axiovert200M

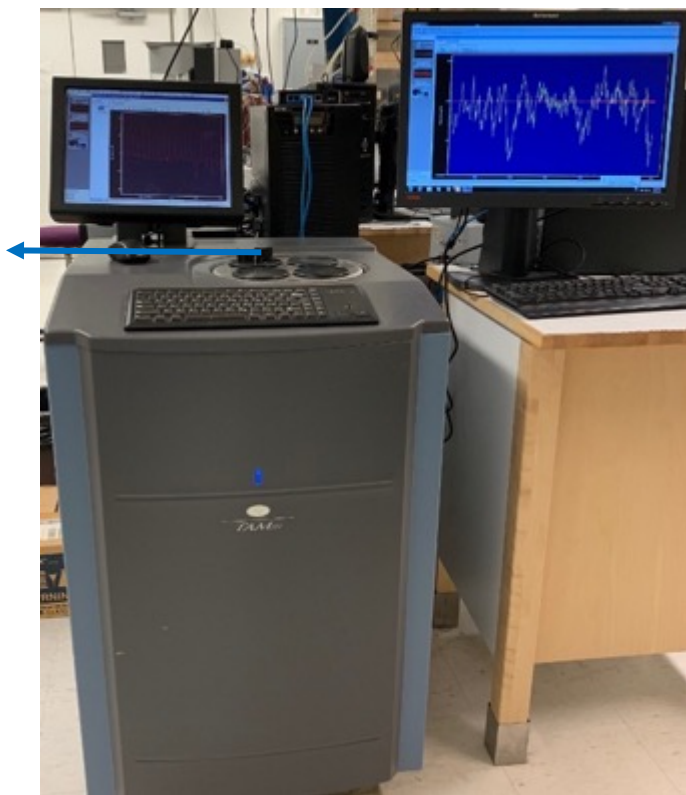
Can be used for the examination of samples in culture flasks, Petri dishes, microtiter plates, etc. in transmitted and reflected light. The microscope can also be used for fluorescence microscopy with a range of filters available. The installed objectives are 10x, 20x, and 40x. Capture still images or monochrome 15fps / color 7.5fps video using the integrated Basler A102FC color imaging camera



Isothermal Titration Calorimetry (ITC)

(TAM III, TA Instruments)

20 mL micro
reaction system



- Direct measurement the heat (precision : ± 100 nW) that is either released or absorbed during an interaction process.
- Characterization of a wide range of molecular interactions, chemical kinetics, and thermodynamic parameters such as binding constants, reaction stoichiometry, enthalpy and entropy in aqueous and non-aqueous media.
- Temperature: 15 – 150 ° C.
- Example application: asphaltenes aggregation in solution; interaction of polymers and mineral solids.

Karl Fischer Coulometric Titrator (C10S Mettler Toledo)



- Uses coulometric titration to determine trace amounts of water in a sample.
- Detection range: 10 ppm to 5.0 wt%.
- Example application: water content in crude oil, bitumen or emulsions.

Mastersizer 3000 (Malvern)



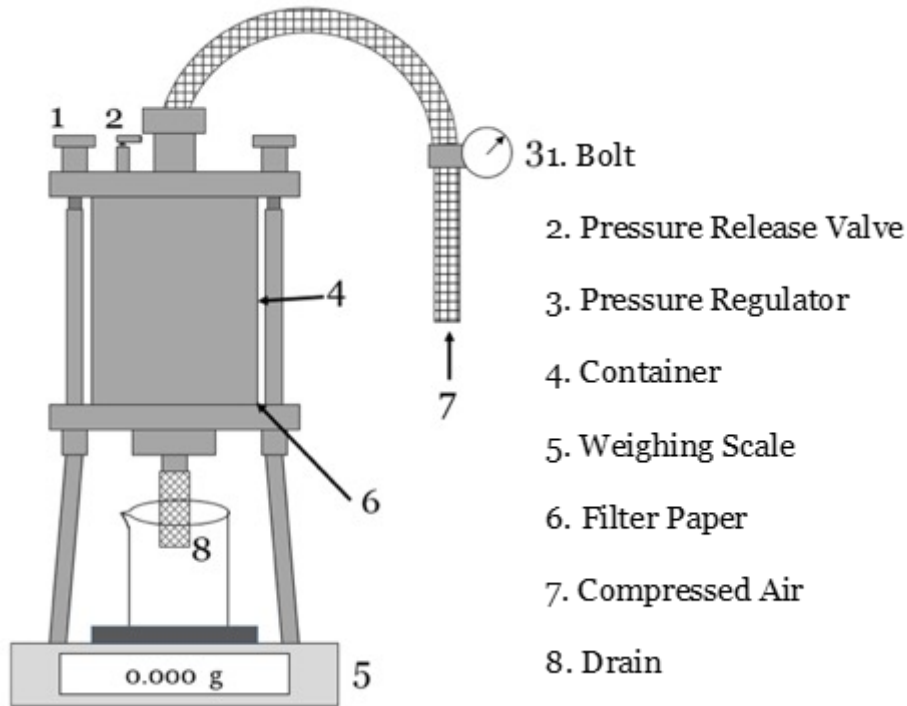
- Principle: Laser light scattering.
- Particle size distribution of suspensions and emulsions in aqueous and non-aqueous media.
- Size range: 10 nm - 3.5 mm.
- Volume or Number distribution.

Organic Elemental Analyzer – CHNS/O (Flash 2000, Thermo)



- Determining the percentages of carbon, hydrogen, nitrogen, sulphur and oxygen of organic compounds based on combustion.
- Detector: TCD.
- Detection range (absolute mass):
C: 0-7 mg (or 100%); O: 0-2 mg;
N: 0-2 mg (or 100%) H: 0-1 mg
(or 100%); S: 0-2 mg (or 100%).
- Detection limit: < 100 ppm.
Standard deviation: < 0.1% abs.
- Example application: organic content in oil sands solids.

Pressure Filtration Unit (EMD Millipore)



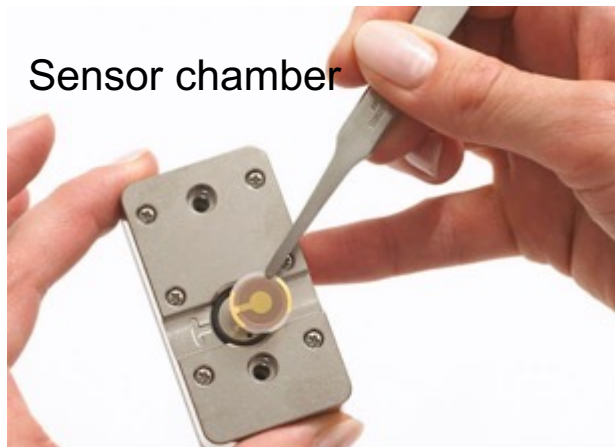
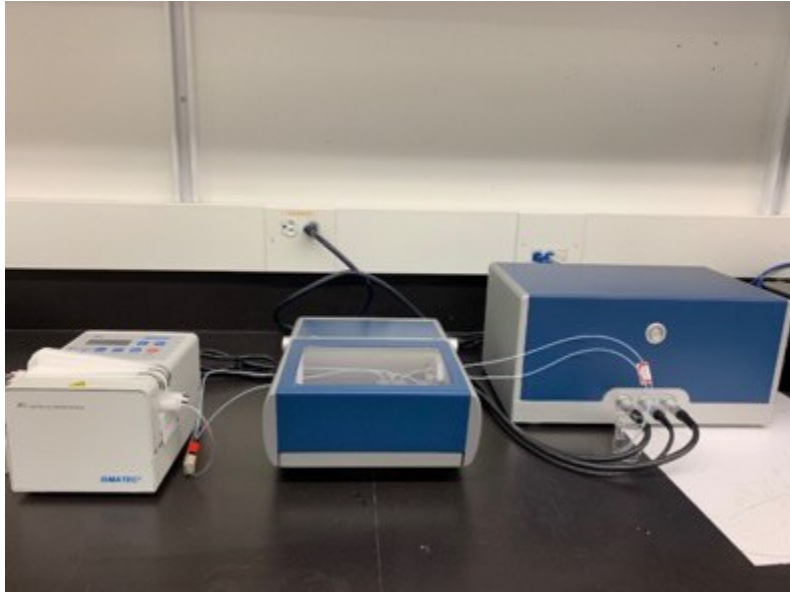
- Pressure range: 1- 6 bar
- Compressed air
- Millipore filter diameter 142 mm
- Feed: ~ 500 g treated MFT.



Filter cake

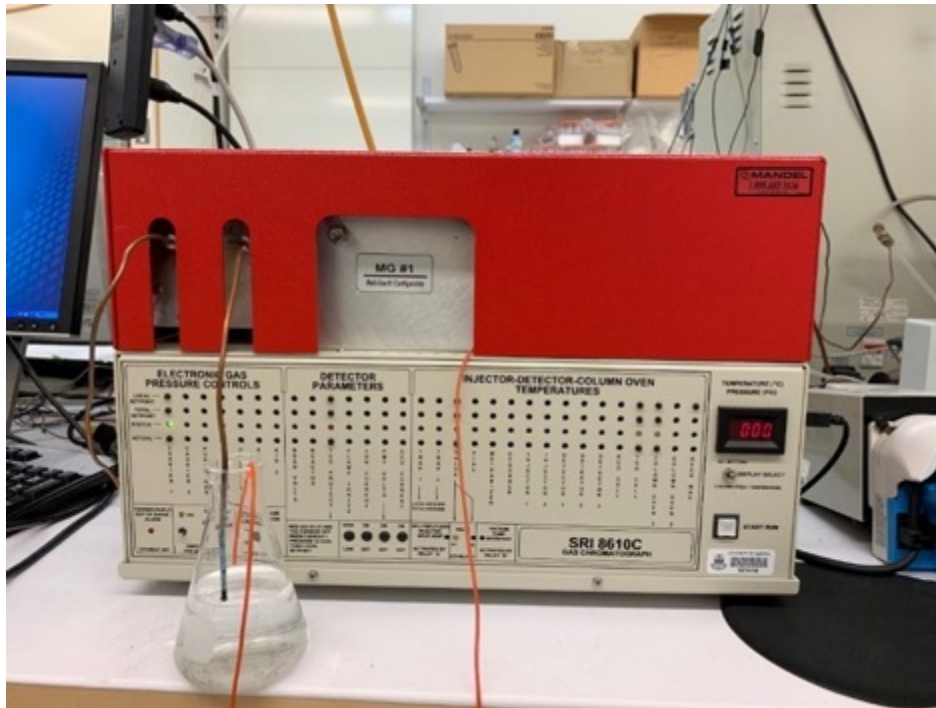


Quartz Crystal Microbalance with Dissipation (Q- Sense Analyzer E4, Biolin Scientific)



- A real time technique with simultaneously measuring the changes in the resonant frequency related to the mass/thickness of the film in contact with the surface and energy dissipation associated with the rheological properties of the adsorbed film, induced by adsorption/desorption processes or by structural changes produced within the thin film.
- 4- sensor chambers system.
- Test volume: $\sim 40 \mu\text{L}$; Normal mass sensitivity: 1.8 ng/cm^2 ; Normal dissipation sensitive: 0.1×10^{-6} .
- Example application: interactions of biomolecules, polymers, surfactants, and nanoparticles at the liquid-solid interface.

Refinery Gas Analyzers (SRI 8610C, Mandel)



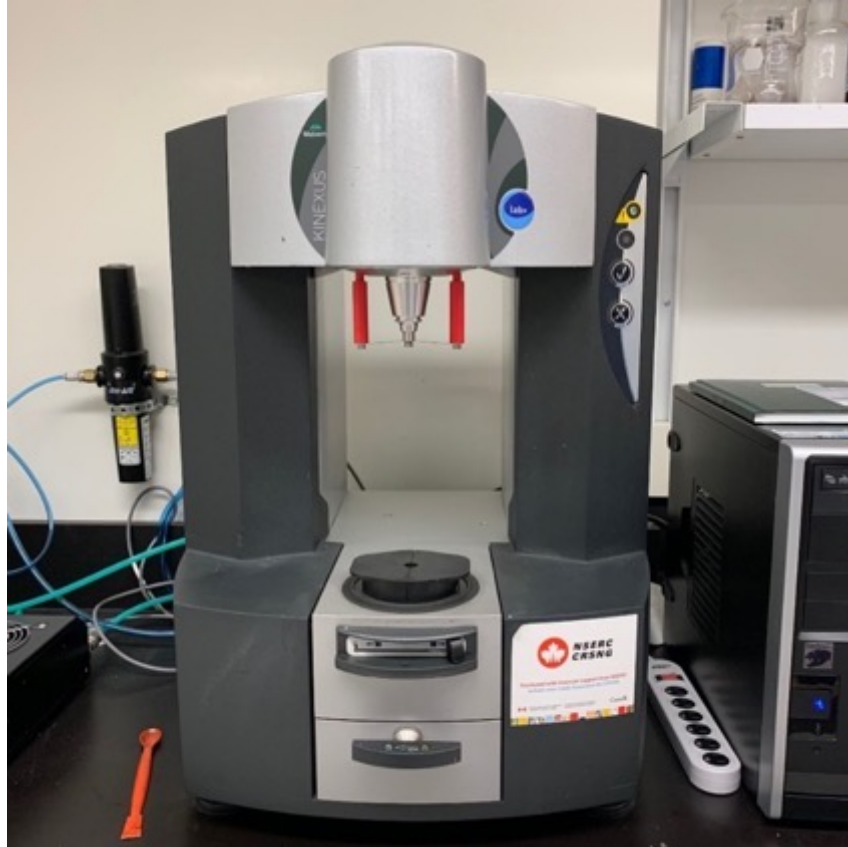
- SRI 8610C is a GC based method to separate and identify gas components (e.g. CO, CO₂, CH₄, C₂H₆ etc. with low boiling points) in mixture for both qualitative and quantitative analysis.
- TCD detector with temperature programmer from ambient to 200° C.
- Example application: gas fractions after thermal cracking of vacuum residues.

Rheometer (Brookfield)



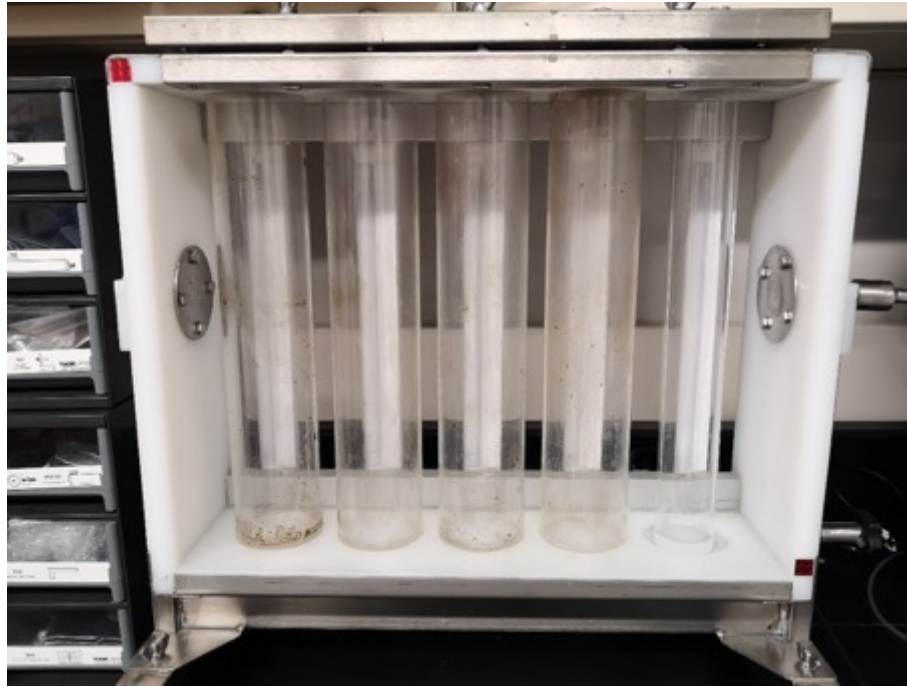
- The Brookfield RVDV-III Ultra Rheometer measures fluid parameters of shear stress and viscosity at given shear rates.
- This rheometer features the Small Sample Adapter and two spindles (bob in fixed cup with samples volumes of 3 and 8mL) capable of measurements from 0.1-12,500 Pa-s.
- Temperature is adjustable between -10C and 80C.

Rotational Rheometer (Kinexus lab+, Netzsch)



- Samples are loaded between two plates, or other similar geometry such as cone and plate or alternatively a cup and bob system. Applying a torque to the top plate exerts a rotational shear stress on the material and the resulting strain or strain rate (shear rate) is measured.
- Torque range: 10 nNm to 200 mNm. (Viscometry); 5.0nNm to 200mNm. (Oscillation).
- Temperature range: -30 to 200 °C.
- Wide variety of measurement for rheological characterization of complex fluids and soft solids, including dispersions, emulsions, polymer and surfactant solutions, pastes and gels.

Sedirack (Cetten Instruments)



- An instrument that allows determining, independent of the operator, the sedimentation rate of particulate solid suspensions in liquids with flocculants.
- Five transparent tubes with rubber stoppers fixed in a rotated frame for homogenization.
- Measurement of the settlement rate of the water-suspension interface is automatically made in all the columns by means of a video camera attached to a laptop.
- Example application: evaluation of performance of flocculants for oil sands tailings treatment.

Simulated Distillation (Simdist) (GC Varian 450, Agilent)



- Simdist is a GC based method to determine the boiling range distribution of petroleum products, fractions, lube oil and, crude oil.
- Boiling point range: 175 – 750° C (with ASTM D 7169).
- Example application: boiling points distribution of bitumen and bitumen-derived products.

TGA (Thermo Cahn 400)



- Thermogravimetric Analysis (TGA) measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere.
- Temperature: RT to 1100° C.
- Mass of samples: 5 – 50 mg.
- Resolution of microbalance: 0.1 μg .
- Example application: characterization of materials that exhibit weight loss or gain due to decomposition, oxidation, or dehydration.

Total Organic Carbon Analyzer (SHIMADZU)



- Use the 680 ° C combustion catalytic oxidation method to determine the amount of carbon in aqueous solutions.
- Measurement: Total Carbon; Inorganic Carbon; Total Organic carbon; Non-Purgeable Organic Carbon; Purgeable Organic Carbon.
- Detection limit: 0.5 ppbw to 3000 ppmw.

UV-Vis Spectrometer (Varian Cary 50)

- Wavelength range: 190-1100nm
- Transmission
- Optical Fiber for in-situ UV-Vis measurement
- Liquid/film



- Numerical bench-scale separation and preparation equipment (not listed as small & non-specific equipment) for solvent extraction of oil sands, minerals separation, tailings treatment, asphaltenes separation, bitumen quality, chemical reactions, etc.