

Analytical Research Group

General Description

The Analytical Research Group (ARG) is a service unit within the EMS Energy Institute committed to:

- the upkeep the off-line instrumentation;
- training faculty, staff, and students on each instrument's correct operation;
- coordinating the scheduling and usage of equipment;
- informing and educating investigators on appropriate instrumental techniques and their availability;
- maintaining The Energy Institute Sample Archive; and
- preparing ASTM-certified grindability standards.

Service Commitments

Utilizing and understanding instruments to the highest degree ensures good data and good research. Having the ability to use multiple instruments to determine physical and chemical properties of samples leads to more accurate data. ARG ensures that all researchers at the EMS Energy Institute are able to take complete advantage of the analytical capabilities of the facility, by working with principle investigators to acquaint them with the full range of analytical capabilities. ARG also promotes and provides analytical support, and is responsible for all the off-line instrumentation within the EMS Energy Institute. ARG coordinates equipment scheduling and usage, works to inform and educate principle investigators on appropriate instrumental techniques and their availability, trains users, and maintains the upkeep of the instruments.

ARG is responsible for preparing ASTM-certified Hardgrove Grindability Index Standard Reference Samples for commercial coal users, research organizations, and laboratories. ARG also maintains the EMS Energy Institute Sample Archive and Penn State Coal Sample Bank.



GC-MS and Py-GC-MS Systems

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Key Equipment

The EMS Energy Institute instrumentation is divided into four categories: Chromatography and Spectroscopy, Physical Properties Analysis, Proximate and Ultimate Analysis, and Thermal Analysis.

Chromatography and Spectroscopy

- Simulated Distillation Gas Chromatography
- S, N and P Selective Detection (Gas Chromatography)
- Separation of Chiral Components by Gas Chromatography
- Headspace Gas Chromatography
- High Performance Liquid Chromatography
- Gas Chromatography/ Mass Spectrometry
- Pyrolysis-Gas Chromatography/Mass Spectrometry
- Solid-State Carbon-13 NMR Spectroscopy
- Liquid-State NMR Spectroscopy with Imaging Capabilities
- Fourier Transform Infrared Spectroscopy/ Infrared Microscopy/ Raman Scattering Spectroscopy

Physical Properties

- Surface Area, Pore Size, and Pore Volume Analysis
- Fluidity Determination
- Particle Size Distribution Analysis
- Rotational Viscometry

Proximate and Ultimate Analysis

- CHN Elemental Analysis
- Total Sulfur Determination
- Proximate Analysis
- Microwave Moisture Analysis
- Multiphase Carbon Analyzer

Thermal Analysis

- High Temperature Atmospheric Thermogravimetric Analysis
- High Pressure Thermogravimetric Analysis
- Atmospheric Thermogravimetric Analysis
- High Pressure Differential Scanning Calorimeter
- Adiabatic Calorimetry

Equipment Capabilities

Chromatography and Spectroscopy.

Chromatography is a class of analytical chemistry that enables the detection and identification of individual species in a complex mixture by selective separation. Several chromatography techniques have been developed due to the need to identify different compounds within complex mixtures. The EMS Energy Institute has a diverse selection of chromatography techniques at its disposal. The majority of these technologies are focused on the detection and speciation of complex hydrocarbon mixtures (i.e. determining the concentration of specific sulfur compounds in diesel fuels, or measuring the concentration of specific toxic chemicals in the emissions from flue gas).

Nuclear Magnetic Resonance Spectroscopy (NMR) is a technique that may be employed to determine the average structures of complex hydrocarbon liquid mixtures or solids. Magnetic Resonance Imaging (MRI) enables the analyst to determine the three-dimensional structures within samples by imaging the void spaces between the structural elements with spectroscopically active species. For example, NMR has enabled researchers to elucidate typical structures within coal (in combination with other techniques), and MRI is being employed in food science to follow the migration of water through candy and chocolate as they cure.

Fourier Transform Infrared Spectroscopy (FTIR) is a useful analytical technique for both qualitative and quantitative analysis of organic and inorganic materials. FTIR may be employed to characterize the chemical structure and the composition of various materials and to study the changes occurring upon chemical reactions.

Fourier Transform Infrared Microscopy is a hybrid of infrared spectroscopy and optical microscopy. Infrared microsampling has become a popular technique for the analysis of a variety of materials such as composites, fibers, laminate films, polymers, paints and coating, processed surfaces, semiconductor products, and the detection of structure anomalies to name just a few applications.

Fourier Transform Raman Scattering Spectroscopy is another branch of vibrational spectroscopy. The technique is very useful for the characterization of biological materials.

Physical Properties

Measuring the physical properties of samples is important in determining the effects of processing. For example, changing the modes and conditions of the activation of carbon materials severely affects the surface area and



Shimadzu QP-5000 Gas Chromatography/ Mass Spectrometry featuring Direct Insertion Probe

surface properties. Surface area analysis and pore volume are used to give an indication of these changes. Likewise, the measurement of particle size distribution enables the analyst to determine the effectiveness of grinding.

Proximate and Ultimate Analysis

Ultimate analysis is the measurement of the elemental composition of a sample. The instruments available allow for the determination of carbon, hydrogen, nitrogen and sulfur concentrations, which is adequate for most hydrocarbon samples, including coal and crude oil. Proximate analysis is a measure of moisture, mineral matter and volatile matter in hydrocarbon samples such as coal. By determining these values, the analyst calculates how much organic material is in a sample. The multiphase carbon analyzer can be used to determine the presence of different forms of carbon by measuring carbon dioxide evolution over a specific temperature range. This is very useful when trying to determine the mechanism by which the carbon was formed, as different forms of carbon burn off at different temperatures.

Thermal Analysis

Thermogravimetric analysis (TGA) is used to monitor weight change over a given temperature profile in select gaseous atmospheres. The instruments have select capabilities that include maximum temperatures up to 1500°C, gas switching, gas mixing and high pressure TGA (up to 1500 psig). These capabilities allow for the simulating of such effects as coal in flue gas or the behavior of SO_x absorbents in pressurized fluidized bed reactors.

Differential Scanning Calorimetry is used to detect thermal transitions in a sample relative to a reference, e.g., glass transitions and melting point measurements. The Parr Adiabatic Calorimeter is used to measure the gross calorific value of solid and liquid samples in a high-pressure oxygen chamber.

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