ICSET

Institute for Combustion Science and Environmental Technology

WESTERN KENTUCKY UNIVERSITY

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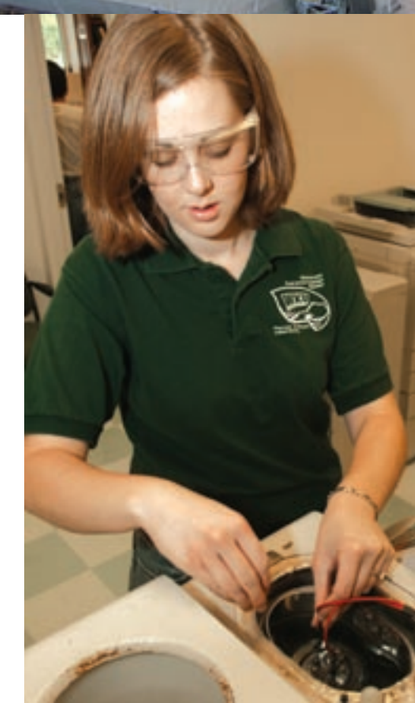
Quentin Lineberry, Ph.D. candidate, uses EPA Method 5 to test for mercury speciation from a stack.

(Cover) Chemistry research assistant Bobby Chen checks the SO_3 sampling system.

Overhead photo of a sampling location after electrostatic precipitator and a wet scrubber.

(Middle) Ginger King, an undergraduate employee, using the bomb calorimeter to determine chlorine content.

(Right) Dr. Li and Matt King test samples with the CVAA Leeman Mercury Analyzer.



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The Institute for Combustion Science and Environmental Technology (ICSET)

Three prominent laboratories at Western Kentucky University (WKU) have been combined into a new entity, the **Institute for Combustion Science and Environmental Technology (ICSET)**. The **mission** for this new institute is:

- To provide the best educational experiences for WKU students, including opportunities to use state-of-the-art instrumentation in course work and research.
- To provide multidisciplinary approaches to solve industrial problems and for research projects.
- To promote professional growth of faculty by providing avenues for collaboration with professors at WKU and other institutions and with professionals in industrial, state, and federal research institutions.
- To provide professional contacts for WKU professors in search of funding for research projects.

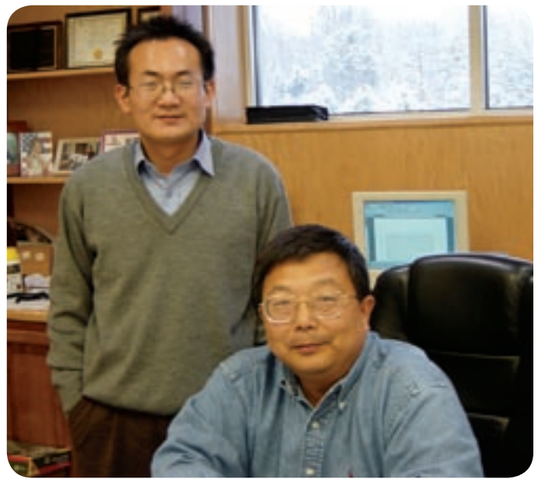
(Top left) Mobile Mercury Monitoring Laboratory

(Top right) Dr. Pan signs a research agreement with Black Double Dragon Boiler in China.

(Bottom left) Sampling personnel collaborationg with plant personnel on-site.

(Bottom right) Dr. Pan poses with one of our visiting scholars.

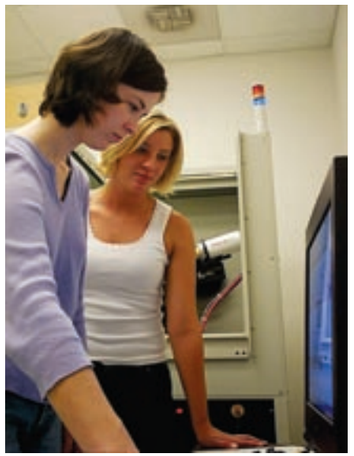
The WKU Thermal Analysis Laboratory was established in 1986 and the Combustion Laboratory was established in 1993, following completion of the construction of a 12-inch fluidized bed combustor. The Mercury Emissions and Control Laboratory was established in 2002 after a Mobile Mercury Monitoring Laboratory was constructed. All three laboratories have been managed through the Materials Characterization Center. Following strong efforts to maintain excellence in WKU programs and create a hands-on learning environment for science students, these prominent laboratories have grown and evolved very rapidly. In light of this growth and the changing needs of both our educational and economic climates, on March 1, 2004, the organization of the Applied Research & Technology Program (ARTP) centers was changed and these three laboratories formed the **Institute for Combustion Science and Environmental Technology (ICSET)**.



Thanks to the ARTP's financial support over several years, the effectiveness and outreach of these laboratories has increased to a point of self-sufficiency. The new Institute is still an integral part of the ARTP program but is no longer dependent upon ARTP funding for daily operations.

The **distinctive and unique features** of the institute are:

- According to senior officers of the North American Thermal Analysis Society, the Institute for Combustion Science and Environmental Technology (ICSET) has the best-equipped Thermal Analysis Laboratory in the U.S., and possibly the world.
- The ICSET Mobile Mercury Monitoring Laboratory is one of only five such laboratories in the U.S. capable of conducting both on-line and Ontario Hydro methods for mercury monitoring. The ICSET Mobile Monitoring Lab is the only facility that is totally mobile for both methods.
- The ICSET Combustion Laboratory has ten years of research experience with fossil fuel combustion including high sulfur and high chlorine coals, MSW (RDF), and other fuels.
- The ICSET Combustion Laboratory has operated its multifunctional fluidized-bed combustion (FBC) system nearly 8,000 hours, including seven runs with 1,000 hours of continuous operation. This 12-inch combustion system has seen more hours of operation than any university FBC system in the U.S.
- The ICSET Mercury Emissions and Control Laboratory has four years of field-testing experience on mercury species conversion, mercury emission, and metal corrosion testing after firing various coals in 55 different boiler/unit configurations.
- The ICSET Mercury Emission and Control Laboratory has developed an industry-wide reputation for excellence, due to its highly trained personnel, up-to-date instrumentation, operational specifications reflecting EPA requirements, and the drive for efficiency and accuracy, which is a hallmark for ICSET.
- The ICSET is an academic and industrial research and development facility located in an academic institution.
- All ICSET labs are multifunctional facilities used for teaching, research, and service work.
- All ICSET labs are self-supported.
- All ICSET graduates have co-authored one or more papers, made one or more presentations at professional meetings, have instructed lab sessions, and have worked on several industrial research projects. In other words, the ICSET graduates are well-trained and experienced professionals.



(Above) Undergraduate chemistry students work with the X-ray diffractometer.

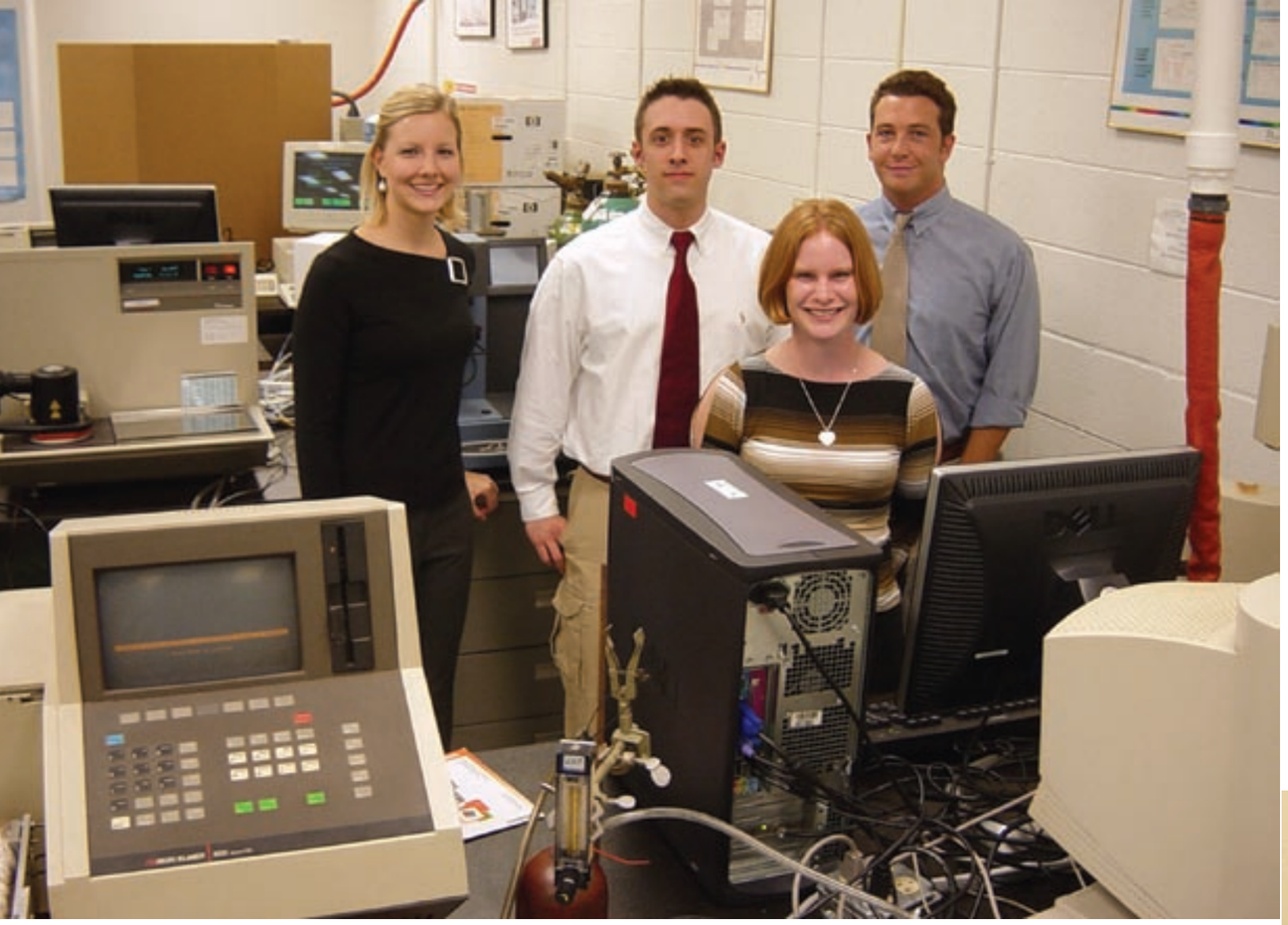
(Right) Dr. Jiang Wu works with the dry Hg SCEM system.

- ICSET has had over 200 industrial clients in the 2000-2004 period. Many of these clients have visited the labs to discuss projects.
- There have been over 70 graduates (Ph. D., M.S. and B.S.) who were employed as students in the labs of ICSET. After graduation, 45% of these students entered graduate and professional degree programs. The other graduates were all professionally employed within two months of graduation.

Students, staff, faculty and visiting scholars from many countries will continue to staff the advanced laboratories of this institute utilizing sophisticated and cutting-edge instrumentation that in turn provides the most relevant training experience.

The Institute for Combustion Science and Environmental Technology is pleased to announce the renovation and construction of two new facilities, totaling over 38,000 square feet, located in the former Bowling Green Mall on Nashville Road. The Combustion Laboratory and the Mercury Emission Control Laboratory will be housed in a 13,500 square foot facility that includes fuel preparation and analysis equipment, as well as the newest circulating fluidized bed combustion system (CFBC). The expected completion date for the renovation is June of 2005 and the expected completion date for CFBC construction is October of 2005.

Hand-in-hand with the construction of the Combustion Laboratory will be the construction of a substantially expanded Thermal Analysis Laboratory (ICSET) and Materials Characterization Center (Coal and Fuel Laboratory). Currently these laboratories, housed at Western's South Campus, have outgrown their space. Consisting of 24,500 square feet, the new facility will increase the capability of services offered to many industries, both regional and national, that require accurate and fast analysis for manufacturing, quality control, and public health purposes.



The Thermal Analysis Laboratory

The **Thermal Analysis Laboratory** at Western Kentucky University was established in 1987 and has evolved into one of the best, if not the best, equipped thermal analysis laboratories in the United States. As a result, the lab has developed into an internationally recognized facility. The facility is used as an instructional lab for physical and materials chemistry courses, as a research facility for 10-15 students each year, and has provided analytical services to over 230 companies, universities and agencies in 30 different states. Analytical results for over 2,500 samples have been provided to these agencies and to WKU researchers.

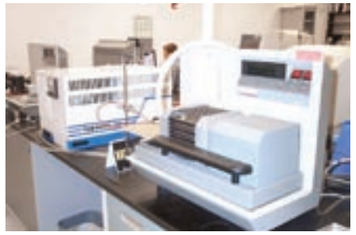
Samples analyzed include everything from meat to explosive materials, but the majority of samples have been polymers. Revenue brought into the laboratory by this service work is used for students' salaries and maintenance expenses for the equipment. The Thermal Lab has provided part-time employment to thirty undergraduate students and twenty-five M.S. and Ph.D. students.

The transition from an academic laboratory setting to an industrial research setting can be difficult. A large number of students enter industry after obtaining degrees, and project-based curricula involving the partnership of universities and industry provided at the lab, ease the transition and increase the probability of our young professionals' success. By partnering with local industries, we are able to participate in challenging research projects that are associated with cutting edge technology. Likewise, through partnerships, industries gain fresh ideas while educating a new employee pool**. The mission of the Thermal Analysis Laboratory at Western Kentucky University is to work alongside governmental, industrial research, and production laboratories.**

Current Research Projects:

- Thermal Degradation of Modified Carbon Nanotubes
- A Study of the Thermal Stability, Degradation Mechanisms and Properties of Polymer/Organically Modified Layered Silicate Nanocomposites
- Development Novel Sensors for Detecting Biologically Active and Chemical Warfare Agents
- Analytical Services

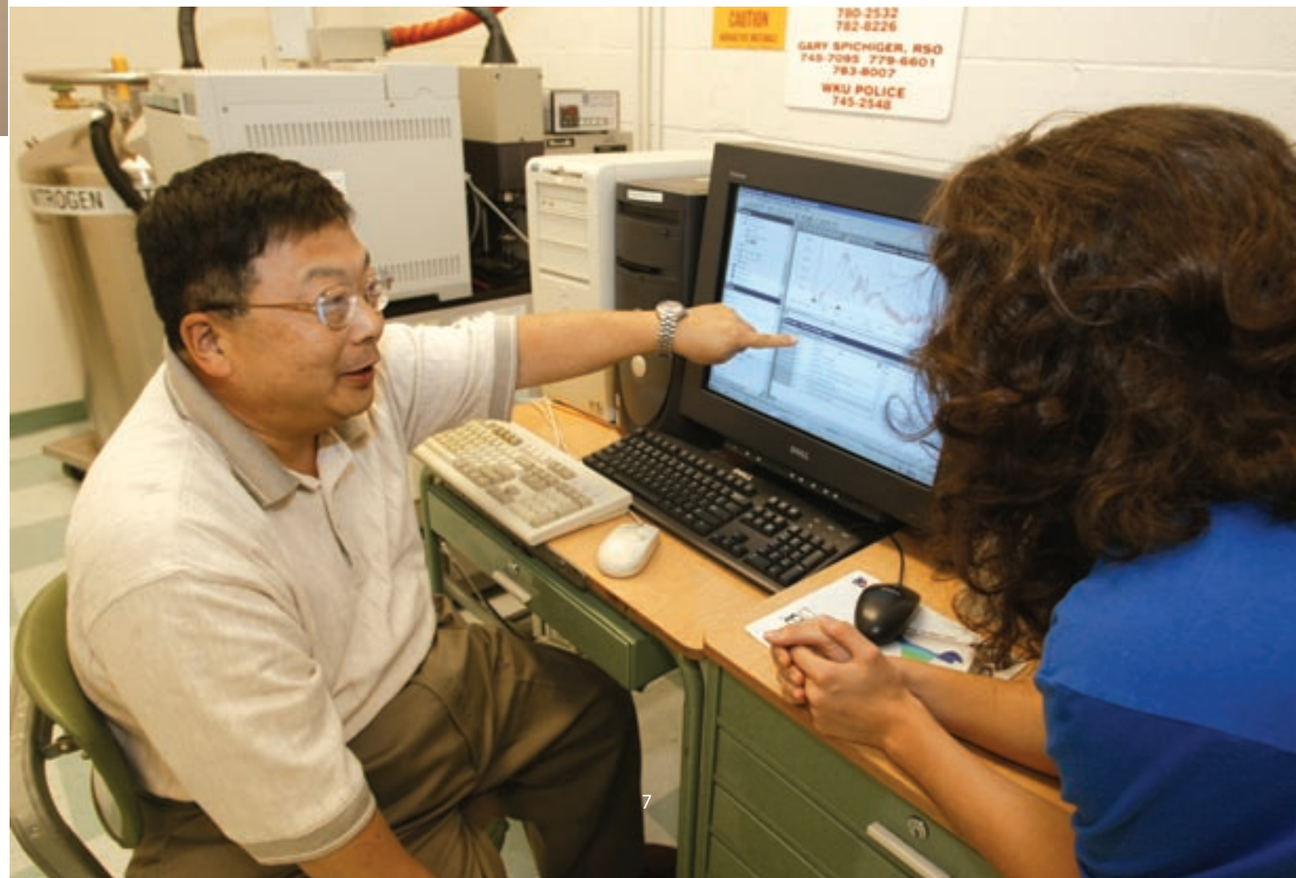
Cooperation with governmental organizations includes collaborations with the United States Air Force Research Laboratory, the United States Navy—Naval Undersea Warfare Center, and NASA. The research with the United States Air Force involved the determination of the thermal stability of organically modified layered silicates (OLS) and also a mapping of the thermal decomposition kinetics of these materials. The Thermal Lab used evolved gas analysis to identify combustion products of materials that were to be used on submarines to determine what potential hazards these materials may pose. The work with NASA is focused on the modification of single-wall carbon nanotubes to enhance solubility and degradation studies of the modified nanotubes.



(Left) Physical chemistry students pose in the Thermal Analysis Laboratory.

(Above) Evolved gas analysis equipment in the TA Lab

(Below) Dr. Pan discusses GC/TOF-MS results with Pauline Hack (laboratory coordinator).



Recently, the Thermal Lab teamed up with a local rubber manufacturer to characterize automotive belts. A particular lot of belts operated at approximately ten percent of the expected performance levels. Differential scanning calorimetry (DSC) was used to show that the glass transition temperature of the two polymeric portions was equivalent, indicating that the poor performance was due to a filler material. Thermogravimetric Analysis (TGA) experiments under pyrolysis conditions showed no apparent differences in the two belts; however, in oxidative atmospheres, the poor performance belt had decreased oxidative stability of carbon black. Upon examining the lots of raw carbon black, the faulty lot was identified.

Gas Chromatography/ Mass Spectrometery (GC/MS) has been used to perform an analysis for salicin—a mild analgesic—for a local willow farm. Salicin is a naturally occurring compound found in willow. Rabbit farmers use willow as a mastication agent to prevent the overgrowth of the rabbits' teeth; however, too much salicin can have adverse effects on the rabbits' health. Salicin was extracted from the bark of the willow and GC/MS used to quantitatively determine the amount of salicin within two hybrids of willow in order for the customer to choose the best plant for the rabbits' health.

Instrumentation:

- Thermogravimetric Analyzer (TGA)
- Differential Scanning Calorimeter (DSC)
- Thermomechanical Analyzer (TMA)
- Dynamic Mechanical Analyzer (DMA):
- Simultaneous Thermogravimetric/Differential Thermal Analyzer (SDT)
- Dielectric Analyzer (DETA)
- Micro- Thermal Analyzer (µTA)
- Rheology
- TG/FTIR, TG/MS
- GC/TOF-MS/ThermEx, LC/MS
- SEM/EDX and Optical Microscope/Image Analysis System
- X-Ray Differaction Spectrometer
- Thermo-IR

(Below) An overview of the Thermal Analysis Laboratory

Over the past 17 years, the thermal analysis group has presented over 50 papers at the North American Thermal Analysis Society (NATAS) annual conferences. Students from the thermal analysis group at WKU also attend the NATAS Conference each year to serve as aides. The Thermal Lab also organizes at least two thermal analysis short courses every year--one with a national/international focus at the NATAS Meeting while the other one is of a more regional nature at Western. Over 50 participants per year come from all over North America to attend these two-day short courses. The Thermal Lab has organized other thermal analysis symposia, including the 2nd International Symposium on Micro-Thermal Analysis at WKU. The Symposium featured talks by invited speakers and poster presentations by WKU students and attracted participants from all around the world. The Thermal Analysis Laboratory also will host the 2006 NATAS Conference in Bowling Green, Kentucky. More than 200 thermal analysts are anticipated to attend this conference.

Capabilities:

For Research and Development

- Formulation optimization
- Materials selection
- Application development
- End-use performance prediction
- Competitive product evaluation
- Theoretical research on new materials and processes

For Quality Control/ Quality Assurance

- Vendor certification
- Incoming/outgoing materials consistency
- Process optimization
- Finished product performance
- Troubleshooting

(Left) An undergraduate student works with the calorimeter.

(Above) An undergraduate student works with the Q-series TGA.

(Right) Undergraduate chemistry students host a poster competition at the Thermal Analysis Lab.

Bianca Casenas (an undergraduate employee) works on the TGA 701 for moisture and ash content determination.

The Combustion Laboratory

The **Combustion Laboratory**, which was established in 1993, focuses on the behavior of chlorine, sulfur, and mercury during combustion. Since the construction of the 0.1MWth laboratory scale fluidized-bed combustor (FBC system) in 1995, over a half million dollars in research funding has been received from the U.S. Department of Energy, EPRI, the Illinois Clean Coal Institute, and the Tennessee Valley Authority. The FBC system has been involved in over 8000 hours of testing. This amount of testing time is the longest that has been conducted by any university FBC system in the United States.

Current Research Projects:

- Establishment of Environmental Control Technology Laboratory with a Circulating Fluidized Bed Combustion System
- Development for Clean Coal Technology: Horizontal Swirling Fluidized Bed Boiler
- Development of a Method for Measuring Carbon Balance in the Chemical Sequestration of Carbon Dioxide
- Study on Carbon Dioxide Removal from Flue Gas by Reaction with Aqueous Ammonia
- Application of a Circulating Fluidized Bed Process for the Chemical Looping Combustion of Solid Fuels
- Maximizing Co-Benefits from Multi-Utilization of Chicken Waste

2" drop-tube reactor at the Combustion Laboratory

Instrumentation:

- 2" I.D. tube flow reactor (up to 12000C)
- 1" Fixed Bed reactor (12000C)
- Sulfur trioxide generator
- 3" I.D. simulated fluidized bed combustor (up to 14000C)
- 0.1MWth pilot-scale Fluidized Bed Combustion (FBC) system.
- 0.6MWth bench-scale multi-functional Circulation Fluidized Bed Combustion (CFBC) system
- Simulated flue gas generation system
- Simulated Selective Catalytical Reactor (SCR) system
- Horizontal Circulating Fluidized Bed (HCFB) cold model
- 1312 Photoacoustic Multi-Gas Monitor (NH_3, CO_2, N_2O, SO_2, HCl, and H_2O)
- IMR 7000 Combustion-Gas Analyzer (O_2, NO, NO_2, CO, SO_2, and HC)
- TELEGAN gas monitoring -- TEMPEST 100 (O_2, NO, NO_2, CO, SO_2, and HCl)

The Combustion Laboratory at Western Kentucky University (WKU) has been awarded a two million dollar grant from the U.S. Department of Energy for their project "Establishment of an Environmental Control Technology Laboratory with a Circulating Fluidized Bed Combustion System." The grant was awarded in open competition throughout the U.S. under the Broad-based Agency Solicitation Program (Solicitation No. DE-PS26-02NT41613). This is the largest competitive grant ever received by Ogden College. The grant award was made through the Combustion Technology University Alliance (CTUA), a program at the National Energy Technology Laboratory in Pittsburgh. Approximately 20 universities (almost all PhD granting institutions) and 15 companies make up the CTUA. This applied research program was started to help coal-fired power plants deal with current problems of operation and help develop strategies to meet environmental requirements. Senator Mitch McConnell was instrumental in getting Congress to appropriate the funds to establish the CTUA.

The primary objective of this project is to establish an Environmental Control Technology Laboratory (ECTL) using a multifunctional circulating fluidized bed combustion (CFBC) system. The system can be easily configured to make combustion runs with various fuels under varying conditions to analyze and monitor air pollutant emissions, as requested by the lab's industrial partners. The ECTL will help develop technologies that can be used to control emissions under multi-pollutant control legislation that is under consideration by Congress. The successful development of these technologies will provide scientific data on atmospheric pollutants resulting

(Left) Pauline Hack works on the CHN analyzer.

(Right) Larry Meng (M.S. employee) works with a multi-gas analyzer for a combustion project.

from combustion systems, and the methodologies required to reduce the emission of these pollutants across the United States. This grant award comes as a result of cooperative efforts between the Department of Chemistry and the Department of Architecture & Manufacturing Science through the Applied Research and Technology Program.

Other research projects, such as "Development of a Method for Measuring Carbon Balance in the Chemical Sequestration of CO_2," "Development for Clean Coal Technology-Horizontal Swirling Fluidized Bed Boiler," "Application of a Circulating Fluidized Bed Process for the Chemical Looping Combustion of Solid Fuels" and "Maximizing Co-Benefits from Multi-Utilization of Chicken Waste" are also conducted in this laboratory.

Capabilities:

- Study pyrolysis, combustion and gasification for different fuels, such as coal and biomass.
- Investigate incineration of different waste materials including RDF, biomass, MSW, and waste liquids.
- Conduct experiments of cracking reaction for different chemicals.
- Develop new technologies to control pollutant emission.
- Carbon dioxide reduction and sequestration.
- Evaluate different catalysts for chemical and combustion processes.
- Develop the novel gas-solid reactors.
- Mathematical modeling for various industrial processes.
- Investigate hydrodynamics, heat and mass transfer in multi-phase flow systems.
- Active carbon preparation and evaluation.
- Pollutant emission monitoring.
- Develop new instruments.
- Erosion and corrosion of heat exchange tubes in combustion systems.

The 53-foot mobile Mercury Emission Monitoring Laboratory

The Mercury Emission & Control Laboratory

Mercury is a toxic, persistent pollutant that accumulates in the food chain. Fossil fuel-fired utilities are the largest source of human-generated mercury emissions in the United States. The U.S. Environmental Protection Agency (EPA) announced its regulations on mercury emission standards in accordance with the Clean Air Act on December 15, 2003, and required compliance from the power industry. The regulations required the power industry to implement the maximum achievable control technology to reduce mercury

Current Research Projects:

- Field Testing and Data Collection
- Partitioning and Mechanism Studies of Mercury
- Demonstration of Amended Silicate Sorbents for Mercury Control in Coal-fired Power Plants
- Field Testing on Advanced Utility Mercury/Sorbent Demonstration Program
- Modeling of Mercury Speciation Transformation: Kinetic & Dynamic Mercury Chemistry

Bobby Chen prepares the SO_3 sampling system.

Members of the sampling team take Ontario Hydro Method readings.

emission 30% by 2010, and 70% by 2018. The **Mercury Emission & Control Laboratory** at Western Kentucky University (WKU) is one of five laboratories in the nation capable of performing mercury sampling and testing. Specifically, the Mercury Emission & Control Laboratory operates a unique facility, the Mobile Mercury Monitoring Laboratory that contains numerous analytical instruments, to provide real-time, on-site, and validated information quantifying mercury emission that is correlated to power plants' operating parameters.

In order to comply with the EPA mercury emission standard, it is critical for the power industry to determine the amount of mercury emitted from the existing control devices; however, this is not a trivial task. To get an understanding of the difficulties associated with mercury sampling, imagine a baseball field like the Houston Astrodome filled with 30 billion ping-pong balls. This represents the emission environment, but the mercury content is only 30 ping-pong balls. Since 1999, several major utility companies have requested the WKU Mercury Emission Analysis & Control Laboratory to perform field tests in an effort to help these companies determine the amount of mercury emitted and to identify the mercury speciation within the control processes. There have been 21 power companies with 55 different generating units. The Mercury & Control Laboratory always invests in the latest technology for mercury analysis, such as the Mobile Mercury Monitoring Laboratory, mercury analyzers, and the continuous emission monitoring system (CEMS). Furthermore, the laboratory staff has developed operational and QA/QC procedures consistent with the EPA specification and conducted field tests in accordance with them. Specifically, the data collected from the CEMS has been validated with the American Society of Testing and Materials (ASTM) Method D6784-02, commonly known as the Ontario Hydro Method (OHM). All the effort devoted is to provide customers with the most valid data possible. This laboratory also has the capability of sampling and analyzing for arsenic, selenium, cadmium, and nickel in the solid and gas phases.

The Mercury Emission & Control Laboratory at WKU has built up an industry-wide reputation due to its highly trained personnel, up-to-date instrumentation, operational specifications reflecting the EPA requirements, and the desire for efficiency, accuracy, and maintained reputation. In 2005, the WKU Mercury Emissions & Control Laboratory is anticipating substantially increased funding, For example; the "Large Scale Mercury Control Technology Field Testing Program" has been awarded by the Department of Energy, and the "Partitioning and Mechanism Studies for Mercury in SCR Control System" has been funded by EPRI. Based on increased awareness of environmental quality issues, as well as advanced measurement and control technologies implemented by the WKU Mercury Emission Analysis & Control Laboratory, it is expected to be another successful year for the Laboratory.

Instrumentation:

- Isokinetic Source Sampling Equipment - 6 sets
- Semi-continuous Mercury Emission Monitors (SCEM) - 3 Analyzers, 5 Conditioning Modules (4 Dry Based Systems)
- Leeman Hydro Autodigestor & Analyzer (AA), Millennium Excalibur System (AF)
- LECO AMA-254 Advanced Mercury Analyzer
- Photoacoustic Multi-gas Analyzer

Capabilities:

- Wet Chemical Method sampling at 6 locations simultaneously for: Hg, HCl/HF, SO3, Trace Metals, or NH3
- SCEM Hg sampling at 6 locations simultaneously
- Coal, Ash & FGD sludge analysis
- SCR Catalyst Slip Stream Testing Module
- The 53-foot mobile Hg Emission Monitoring Laboratory allows us to analyze samples and process data on-site, drastically reducing the delay between testing and data presentation

Dr. Boshu He checks the sampling train of EPA Method 29, total selected metal determination.

Institute for Combustion Science and Environmental Technology

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Mercury Field Testing Capabilities

Instrumentation:

- Isokinetic Source Sampling Equipment – 6 sets
- Semi-continuous Mercury Emission Monitors (SCEM)- 6 Analyzers, 9 Conditioning Modules
- Leeman Hydro Autodigestor & Analyzer (AA), Millennium Excalibur System (AF)
- LECO AMA-254 Advanced Mercury Analyzer
- Photoacoustic Multi-gas Analyzer (INNOV 1312)

Capabilities:

- Wet Chemical Method sampling at 6 locations simultaneously for: Hg, HCl / HF, SO3, Trace Metals, or NH3
- SCEM Hg sampling at 6 locations simultaneously
- Coal, Ash & FGD sludge analysis
- SCR Catalyst Slip Stream Testing Module
- The 53-foot mobile Hg Emission Monitoring Laboratory allows us to analyze samples and process data on-site, drastically reducing the delay between testing and data presentation

Experience:

- Ten years research experience with fossil fuel combustion processes
- Eight years research experience involving emission controls
- Five years experience with combustion modeling
- Three years of field testing experience on Hg speciation, emission and control with various coal types and over 51different boiler/unit and APCD configurations
- 15 months of activated carbon/sorbent injection projects coordination and evaluation experiences