MicroCalvet Differential Scanning Calorimeter (DSC)

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Other Sources of Funds and Existing Infrastructure in the College

The Tier-1 Engineering Initiative is the only available and immediate source of funds that can be allocated to purchase the MicroCalvet Differential Scanning Calorimeter (DSC). Other sources of funds prefer to support highly qualified personnel. There is only one current instrument in the College that could operate similarly to the MicroCalvet DSC; however, it is aging and not functioning properly.

Statement from the Department of Energy and Petroleum Engineering

If the MicroCalvet DSC is purchased with the aid of the Tier-1 Engineering Initiative Fund, it will be placed in Laboratory 4006, which belongs to Principal Investigator (PI), Morteza Dejam, and is located on the 4th floor of the Engineering Building. The required power for installation of the MicroCalvet DSC is already available in Laboratory 4006.

Advantages of MicroCalvet DSC for Capturing Grants from Different Agencies

Since the MicroCalvet DSC has different applications in engineering and science, as indicated later, this instrument will strongly help the users capture external grants from several agencies (including National Science Foundation, Department of Energy, Environmental Protection Agency, National Aeronautics and Space Administration, Gas Processors Association, and American Chemical Society) and companies (ExxonMobil, Chevron, and Saudi Aramco).

Budget for MicroCalvet DSC

MicroCalvet Differential Scanning Calorimeter (DSC)	$79,432.80

Anticipated Contribution

Tier-1 Engineering Initiative Contribution	$79,432.80

Budget Justification for MicroCalvet DSC

The MicroCalvet DSC will be purchased from SETARAM Inc. (Kep Technologies) at the quoted price (79,432.80), which includes the prices of the MicroCalvet Calorimeter plus Calisto data acquisition and data processing software ($88,380.00), technical support (cost of installation, commissioning, and on-site training) ($3,490.00), and travel charge ($2,262.00). The total cost of the instrument was initially quoted as $94,132.00. However, we have successfully negotiated a significant discount of $14,699.20, reducing the final purchase price to $79,432.80. The MicroCalvet DSC incorporates a calorimetric block using PELTIER effects elements, an ultrasensitive “3D” heat flow calorimetric sensor, a water-cooling circuit, one pair closed “batch” vessels in Hastelloy, the CS Evolution controller with 32 bits microprocessor, 24 bits A/D converter, Ethernet connection, and the CALISTO data acquisition and data processing software.

Introduction and Description of Instrument

The study of the phase behavior and phase transition of confined fluids is critical in many different fields and has several applications, such as catalysis, surface and subsurface gas storage, and the recovery of hydrocarbons from unconventional reservoirs. The information collected from experiments focusing on confined fluids has aided in improving our knowledge and understanding of adsorption-desorption processes and fluid-fluid/fluid-pore interactions. This body of knowledge has also been instrumental in the development and formulation of several equations of state for confined fluids, thus contributing to theoretical and practical advancements in the field. In the investigation of the phase behavior of

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fluids confined in nanopores, the condensation of vapor in the pores (capillary condensation) usually occurs at a very low temperature or high pressure. The MicroCalvet DSC can operate under such conditions. More importantly, it can operate at these conditions (high pressure and low temperature) without the difficulty of using cooling agents such as liquid nitrogen or liquid helium during the cooling process as commonly encountered by some of the in-house DSC instruments. This outstanding performance of the MicroCalvet DSC advances science and technology in different fields that demand operations at severe conditions.

The proposed instrument is the MicroCalvet Differential Scanning Calorimeter (DSC) manufactured by SETARAM Inc. (KEP Technologies). The MicroCalvet DSC can operate under either vacuum, atmospheric, or pressurized conditions, with a pressure limit of 1000 bar and a temperature range of -45 to 120°C. The system provides the option to be used at high pressure with a high-pressure gas panel or ISCO pump. The pressure can be measured and recorded in the software with the use of an analog inlet. The temperature scanning rate of the instrument ranges from 0.001 to 2°C/min. It has a high sensitivity with a resolution 0.02µW. The system temperature is controlled by using the advanced Peltier cooling and heating principle with the assistance of an auxiliary cooling circulator. The system also has one-pair of Hastelloy cells each with a volume of 850µL. The Calvet-type design and holding volume of the Hastelloy cells would allow us to conduct experiments using cylindrical rock core samples without the need to crush them.

The MicroCalvet Differential Scanning Calorimetry (DSC) analyzer is equipped with the advanced CALISTO software for both data acquisition and data processing. This software automates the recording of critical experimental parameters such as temperature and heat flow during each experimental run. In addition to its automation capabilities, CALISTO supports an extensive range of data analysis functions. For example, the amount of heat absorbed or released during phase changes and other thermal transitions can be accurately calculated. The system is highly efficient, delivering processed data points within just a few seconds from the time of sampling, which significantly enhances workflow efficiency.

The MicroCalvet DSC can also accommodate a wide variety of cells, each offering specific functions such as mixing, stirring, pressure resistance, and pressure regulation. This flexibility allows the instrument to cater to a broad spectrum of applications, including studies on heat capacity, thermal transitions, and chemical reactions in gas, liquid, and solid samples.

Moreover, the MicroCalvet DSC offers external coupling capabilities, making it an ideal tool for expanding research possibilities. By integrating the system with complementary equipment such as manometry devices, Brunauer-Emmett-Teller (BET) instrumentation, gas analyzers, and humidity controllers, the scope of available experimental investigations would enable us to increase our research options. The MicroCalvet DSC is suitable for the isochoric cooling procedure developed by our research group.

The existing high-pressure low-temperature SETARAM BT215 DSC, which was acquired using Tier-1 Engineering Initiative Fund, has been operating well in our lab and producing data that have been published. These data have also been used recently in our proposals to obtain external grants. Note, however, that the high-pressure low-temperature SETARAM BT215 DSC is not designed for cooling procedures without the use of liquid nitrogen as the cooling agent. Our goal is to be able to *experimentally* measure the phase transition conditions of different pure fluids and fluid mixtures in Mowry shale outcrops and representative samples. To the best of our knowledge, this would be the first time phase transition experiments have been extended from synthetic nanoporous media to rock core samples.

In summary, the proposed MicroCalvet DSC will tremendously advance our research capability and expand the scope of applications we can investigate. The instrument will help advance our studies across diverse fields including the phase behavior of confined fluids, separation engineering, material science and engineering, carbon capture and storage (CCS), hydrogen storage, to membrane characterization. The MicroCalvet DSC will serve as a valuable educational tool, providing our students with hands-on training in cutting-edge calorimetric techniques. By enabling the exploration of a broader range of scientific and engineering challenges, the system will foster skill development and innovative thinking, equipping students with the expertise necessary to address complex, real-world problems. This instrument represents a transformative addition to our laboratory, offering both immediate research advancements and long-term educational benefits.

Impact on the CEPS

1. *Excellence in Undergraduate Education*

The MicroCalvet DSC can be used as one of the main instruments to help students learn about gas recovery from ultra-tight shale reservoirs. This topic needs an understanding of the fundamentals of Reservoir Engineering (which is a core course of the Petroleum Engineering Undergraduate Curriculum).

2. *World-Class Research and Graduate Education*

The topics of research that will be impacted by the proposed MicroCalvet DSC are listed in the following table, where the major users of the proposed instrument, along with the topic, and the project title can be found.

Topic: Underground hydrogen storage, petroleum fluids, and enhanced oil recovery	
Morteza Dejam	Towards a fundamental understanding of underground hydrogen storage from reservoir engineering perspectives Experimental investigations of phase behavior of confined fluids in nanopores of ultra-tight shale reservoirs
Topic: Environmental geophysics	
Andy Parsekian	Below-zero saline fluid phase behavior in porous materials
Topic: Confined fluid thermodynamics	
Hertanto Adidharma	Systematic study on the phase transition of confined fluids
Topic: Biophysics	
Utkarsh Kapoor	Fundamental understanding of self-assembly in biological systems
Topic: Carbon capture	
Maohong Fan	Novel carbon capture technologies for sustainable engineering and environment
Topic: Materials	
Jing Zhou	Nanomaterials used in catalytic processes related to energy production and atmospheric pollutant control
Topic: Sensitizers	
Jinke Tang	A new type of sensitizers – magnetically doped quantum dots – for improved quantum dot sensitized solar cells (QDSSCs)
Topic: Geophysical/Seismic inversion	
Subhashis Mallick	Constrain baseline and time-lapse seismic data for characterizing underground reservoirs for carbon dioxide/hydrogen storage Characterization of geothermal reservoirs

The proposed MicroCalvet DSC will be part of the Confined Fluid Phase Behavior Laboratory, where the investigation of the most unknown behavior of fluid confined in nanopores is performed. This world-class facility houses state-of-the-art instrumentation for such studies, including the Two-Stream Dynamic Gas and Vapor Sorption Intelligent Gravimetric Analyzer (IGA-003) with Integrated Interchangeable Inlet-Pressure Dynamic Sampling Mass Spectrometer (DSMS) and the high-pressure low-temperature SETARAM BT215 DSC. The addition of the proposed MicroCalvet DSC will make the lab become one of the most sophisticated facilities for confined fluid study in the world.

The quality of research and the extent of collaboration among researchers at CEPS will be tremendously enhanced with the existence of the proposed instrument. More importantly, through purchasing the suggested equipment, UW can align with R1 institutions in terms of laboratory capability while it tries to achieve R1 status in the next few years.

3. *Productive Economic Development through Partnerships*

The addition to the MicroCalvet DSC, which can be used to address research on underground hydrogen storage and other energy storage challenges, will assist the State of Wyoming to be an advocate and champion of regional industry during the energy transition.

4. *K-14 STEM Education*

Since UW is the only provider of baccalaureate and graduate education and research in Wyoming, the acquisition of the proposed MicroCalvet DSC is expected to have a great impact on the training of future scientists at the K-14, undergraduate, graduate, and postdoctoral level, for which the participation of underrepresented groups will also be encouraged. UW actively promotes college preparedness, access, and success among students traditionally underrepresented in STEM fields by focusing special programs on first generation, female, low-income, and ethnic minority students. The Summer Research Apprentice Program (SRAP), Engineering Tier-1 Initiative on K-14 STEM Education, Engineering Undergraduate Research Scholars Program, McNair Scholars Program, Women in Math, Science and Engineering (WiMSE), and Wyoming EPSCoR are existing programs at UW that encourage students to take STEM as their career and encourage underrepresented group.

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Vapor Phase Transition

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