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RPPR Final Report
as of 27-Dec-2018

Agency Code:

Proposal Number: 70407CHREP **Agreement Number: W911NF-17-1-0539**
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Final Report for Period Beginning 25-Sep-2017 and Ending 24-Sep-2018
Title: Clayton State University: Strengthening Science, Technology, Engineering and Mathematics (STEM) through Nuclear Magnetic Resonance (NMR) Research
Begin Performance Period: 25-Sep-2017 **End Performance Period:** 24-Sep-2018
Report Term: 0-Other

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STEM Degrees: 32 **STEM Participants:** 116

Major Goals: The major goal of this proposal was to purchase Nuclear Magnetic Resonance (NMR) instrumentation to be used in undergraduate teaching and research laboratories. The original proposal was for a 400 MHz NMR spectrometer ($309,556.00) and travel for training ($3,000.00). After the grant was awarded, we asked for and received approval to purchase four benchtop NMR instruments (one 80 MHz, two 60 MHz and one 43 MHz) in place of the 400 MHz instrument for the same price. Since training for these instruments could occur on site at no cost, approval was granted to use the allocated travel money for computers for the instruments.

The timeline in our proposal indicated that needed specifications for the instrument would be finalized and competitive bids would be requested by September 1, 2017. The instrument would be ordered and installed by February 28, 2018. Training of key faculty and support staff would be completed by April 1, 2018. This timeline was slightly delayed due to the bid and procurement process taking longer than anticipated. Magritek, Inc. was chosen as the supplier for the instruments and an order was placed in December, 2017. The instruments were delivered and installed in April 2018, and in-depth training occurred in July, 2018.

Specific goals for this project include:
1. To better meet ACS standards in its Chemistry degree program by allowing students to have hands-on experience and skills with NMR.
2. To provide 100-200 STEM majors the opportunity to use an NMR instrument in the following courses: Organic Chemistry I, Organic Chemistry II, Physical Chemistry, Instrumental Chemistry, Advanced Organic Chemistry, Advanced Biochemistry, Inorganic Chemistry, Polymer Chemistry, Medicinal Chemistry, and Chemistry Research Practicum courses

We were able to start implementing use of the NMR instruments in teaching (Inorganic Chemistry) and research (Chemistry Research Practicum) courses in Spring 2018, and have continued to increase the use of the instrument

in additional courses each semester. So far, 116 students have used the NMR instruments, which matches our projection of 100-200 students per year. This number will increase as we continue to expand the use of the instruments in additional courses. A summary of our instrument usage can be found in the Accomplishments section of this report.

Accomplishments: The NMR instrumentation has been used in both research and teaching courses by 116 students since the instruments were delivered and installed in April 2018. The specific objectives were to allow STEM students to have hands-on experience and develop skills with NMR. A summary of the major activities completed as well as anticipated future use follows.

Research activities:
The NMR instruments were used to analyze samples from the research labs of Dr. Agyeman, Dr. Meyers, and Dr. Sheppard. In the future, the 80 MHz instrument will be used for research projects, but the installation of that instrument occurred too late for the student projects described here, so the 60 MHz (installed previously) was used instead.

Dr. Agyeman's project aims to isolate and identify enzymes and other biomolecules that may be present in the leaves of fagus grandifolia, a tree commonly known as American Beech. This particular species of tree has been found to contain vulgarenol, a sesquiterpene that eradicates coronary vasoconstriction, as well as enzymes such as tyramine and monoamine oxidase. Also, the leaves remain green and do not fall off throughout the winter season. The aim is to identify what sustains the leaves during winter. Extracts from the fresh leaves are obtained and analyzed using various types of NMR spectroscopy. Future projects will involve similar extraction and identification of compounds in banana peels and papaya fruits and seeds.

Dr. Meyer's research involved studying the bonding of multiple benzenoid isocyanides with the half-sandwich complex Cp*Ir(III) (Cp* = pentamethylcyclopentadienyl) in order to gauge the feasibility of producing 2D and 3D metal-organic frameworks with the aforementioned components. Multiple NMR experiments including 1H, 13C, COSY, and HSQC were performed on each sample. Future research students will also use this instrumentation to analyze crude and purified samples to understand complex formation and eventually metal-organic frameworks.

Dr. Sheppard's research involved the synthesis of novel sulfa drugs (sulfanilamide derivatives) for use as possible antibacterial agents. Two sulfa drugs were synthesized using a multi-step synthetic route. Products from each step of the synthesis were analyzed and characterized using 1H- and 13C-NMR. Future projects will continue to optimize the synthesis of these sulfa drugs, as well as explore other synthetic routes.

Teaching activities:
The 60 MHz instruments are located in the organic chemistry classroom and are used frequently in the Organic Chemistry Laboratory courses. In the Summer and Fall semesters of 2018, three organic chemistry experiments were conducted using the NMR instruments. The first experiment was an introduction to NMR spectroscopy. Students used the instrument's 1H and 13C functions to identify an unknown compound with a known molecular formula. Later in the semester, students used the instrument to determine unknown functional groups and structure (Ketones and Aldehydes experiment) and to confirm identity of an unknown compound with an unknown molecular formula (Qualitative Analysis experiment). So far, only 1D NMR is being used in this course. Plans are underway to introduce 2D NMR to these students. A new experiment involving the synthesis of aspirin from wintergreen oil and characterization of the products using NMR.is being implemented in Spring 2019.

The 43 MHz benchtop NMR instrument is positioned on a utility cart to allow for transport to different laboratories. In the summer of 2018, the instrument was used in Principles of Chemistry Laboratory I in tandem with a worksheet on Lewis structures and molecular geometries. As students were practicing how to draw Lewis structures and shapes of molecules, NMR spectroscopy was used to explain how the environment of an atom makes a difference and to highlight the importance of studying molecular structure. Students then identified three different neat liquids by collecting data with the NMR, interpreting the data, and then matching to one of the possibilities.

As an internship project, an upper-level chemistry major was tasked with designing an experiment for the inorganic chemistry teaching laboratory. This internship student chose a project that encompasses the synthesis and analysis of metal-organic frameworks (MOFs). After the synthesis of a selected MOF, 1H and 13C spectra will be obtained to determine the success of the reaction and the purity of the product. Additionally, 1H NMR spectroscopy will be used to determine the ability of a MOF to absorb volatile organic compounds. The internship student will present her work, including the NMR data, on this experiment at the ACS National Meeting in Orlando in Spring

2019.

Experiment procedures for courses that have not been offered since the instruments were obtained are being updated to expand the use of NMR. These courses, as well as the semester they will be offered and the titles of the experiments using NMR, are listed below.

• Medicinal Chemistry: offered Fall 2019. Drug-target interactions.
• Advanced Biochemistry Lab: offered Fall 2019. Determination of protein structure.
• Instrumental Analytic Chemistry Lab: offered Spring 2020. Analysis of commercial analgesics; Degree of unsaturation in commercial oils; Analysis of essential oils from spices.
• Advanced Organic Chemistry Lab: offered Fall 2020. Aldol condensation product; Diels-Alder reaction.
• Inorganic Chemistry Lab: offered Spring 2022. Preparation and properties of ferrocene and derivatives; Synthesis and analysis of metal-organic frameworks.

Training Opportunities: On-site training for use and maintenance of the instrument was conducted in July 2018. Training was conducted by a Magritek, Inc. specialist, and lasted for 4 hours. This was a professional development opportunity for the faculty who are the primary users of the instrument (Dr. Sheppard, Dr. Agyeman, and Dr. Meyers) as well as the laboratory staff who maintain the instrument (Ms. Holmes and Ms. Sands).

Results Dissemination: All undergraduate research students who worked on a project involving the NMR presented their data in either a poster or oral presentation format at the Clayton State University Academic Conference, a university-wide conference that incorporates faculty, staff, and students from multiple disciplines. Future research students will also present at Department and/or University conferences. Faculty mentors of these projects plan to publish NMR data in the chemical literature as appropriate.

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

PARTICIPANTS:

Participant Type: PD/PI
Participant: Caroline Sheppard
Person Months Worked: 12.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Co-Investigator
Participant: Augustine Agyeman
Person Months Worked: 12.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Faculty
Participant: John Meyers
Person Months Worked: 12.00 **Funding Support:**
Project Contribution:
International Collaboration:

International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Other Professional
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Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
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Participant: Sheryl Sands
Person Months Worked: 12.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Nothing to report in the uploaded pdf (see accomplishments).