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Bringing NMR and IR Spectroscopy to High Schools

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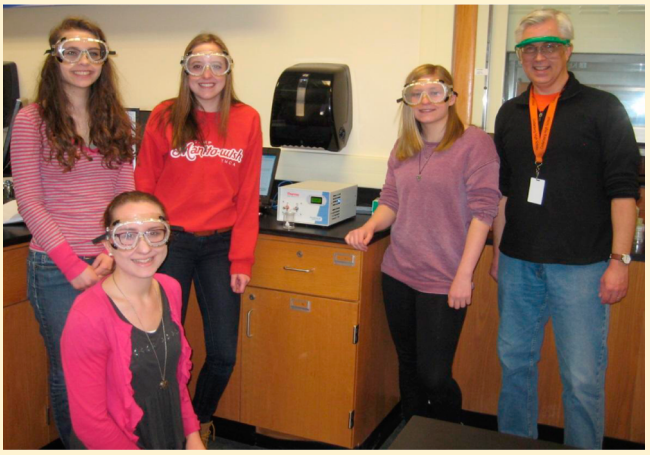
ABSTRACT: Development of benchtop, portable Fourier transform nuclear magnetic resonance (NMR) and infrared (IR) spectrometers has opened up opportunities for creating university−high school partnerships that provide high school students with hands-on experience with NMR and IR instruments. With recent changes to the international baccalaureate chemistry and advanced placement chemistry curricula, NMR and IR spectroscopy can be mapped directly to content that is already built into general, organic, and materials courses offered at the high school level. We developed a program in which the university provides professional development opportunities to high school chemistry teachers and further opportunities for these teachers to bring benchtop NMR and IR instruments directly into their own classrooms. This partnership not only benefits students by providing unique active learning experiences, but also offers opportunities for high school teachers' professional development through on-campus training workshops. These partnerships may also expand university outreach efforts in local secondary schools to increase student awareness of the sciences and bolster recruitment of students.



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INTRODUCTION

Over the past few years, the international baccalaureate (IB)[1] and the advanced placement (AP)[2] chemistry curricula have evolved to include topics related to how molecules interact differently with various portions of the electromagnetic spectrum. While these topics can be covered conceptually in a lecture, students typically do not have much exposure to hands-on experiences in the laboratory with instrumentation beyond visible spectroscopy as it relates to Beer's Law. Additionally, more high schools are expanding their chemistry curricula to include organic and even materials chemistry courses. Students in each of these courses would greatly benefit from hands-on experience with nuclear magnetic resonance (NMR) and/or infrared (IR) spectrometers. In this paper, we describe how the use of these spectrometers can be mapped to various high school chemistry curricula. We also describe a program developed through a partnership between the Chemistry Department and the science outreach program at the University of Wisconsin—Whitewater (UW—Whitewater) and Thermo Fisher Scientific that provides high school teachers an opportunity to bring a benchtop NMR spectrometer and a portable Fourier transform infrared (FTIR) spectrometer into laboratories to give students hands-on experience with these instruments.

CURRICULA MAPPING

International Baccalaureate Chemistry

In the *Diploma Programme Chemistry Guide* published in 2014,[1] there was a significant increase in the coverage of organic chemistry, specifically NMR and IR spectroscopies. Under the old curriculum, topics beyond the core were confined to one of seven options: modern analytical chemistry, human biochemistry, chemistry in industry and technology, medicines and drugs, environmental chemistry, food chemistry, and further organic chemistry. Students were responsible for being proficient in two of the seven options. Additionally, teachers usually only had time to cover two of the options, as they were

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extensive and required a high level of student preparation as the options comprised a large portion (24%) of the students' IB assessment grade.[3] The concepts of NMR and IR spectroscopy, which were found in the analytical chemistry option, were usually passed over because providing students with hands-on experience required equipment that was inaccessible at the high school level.

When the curriculum was revised, the options were condensed to four: materials, human biochemistry, energy, and medicines and drugs. Food chemistry and environmental chemistry concepts were incorporated throughout the four new options. Further organic chemistry was largely excluded from the options, and some of these concepts were instead included in the core organic content. Examples of these concepts include a deeper look into Kekule's structure of a benzene ring and NMR and IR spectroscopies. Now that NMR and IR concepts have been moved out of the options and into the core curriculum, all students must have a proficient understanding of basic NMR and IR theory and how to interpret NMR and IR data.

In the 2014 Chemistry Curriculum Guide, the NMR and IR spectroscopy content standards are included in section 11.3: Spectroscopic Identification of Organic Compounds (ref 1, p 75). Essential understandings include the following:

- The degree of unsaturation or index of hydrogen deficiency (IHD) can be used to determine from a molecular formula the number of rings and/or π-bonds in a molecule.
- Mass spectrometry (MS), proton nuclear magnetic resonance spectroscopy (^{1}H NMR), and infrared spectroscopy (IR) are techniques that can be used to help identify compounds and to determine their structure.

These concepts are also included in the Additional Higher Level (AHL) Section 21.1: Spectroscopic Identification of Organic Compounds (ref 1, p 104). Essential understandings include the following:

- Structural identification of compounds involving several different analytical techniques including IR, ^{1}H NMR, and MS.
- In a high resolution ^{1}H NMR spectrum, single peaks present in low resolution can split into further clusters of peaks.

Teaching students how to interpret NMR and IR data is possible with just pen and paper data sets and a Google image of the instrumentation. However, being able to actually touch and see an instrument in real life and collect data from a real sample add an invaluable dimension to the learning process as they increase engagement, offer real-world tie-ins, and foster students' desire for further study.

Advanced Placement Chemistry

The *AP Chemistry Course and Exam Description* also underwent changes in 2014.[2] While these changes were not as explicit about the inclusion of spectroscopy as those seen with the IB Chemistry curriculum, one level of the Essential Knowledge framework (1.D.3) includes (ref 2, p 89) the following: "The interaction of electromagnetic waves or light with matter is a powerful means to probe the structure of atoms and molecules, and to measure their concentration." This appears under the Big Idea 1, which states (ref 2, p 88) "The chemical elements are fundamental building materials of matter, and all matter can be understood in terms of arrangements of atoms. These atoms retain their identity in chemical reactions." The inclusion of an NMR and/or IR spectroscopy experiment could significantly enhance students' understanding of the essential knowledge framework components. The inclusion of IR spectroscopy will specifically cover Learning Objective (1.15) (ref 2, p 98): "The student can justify the selection of a particular type of spectroscopy to measure properties associated with vibrational or electronic motions of molecules." Inclusion of an NMR and/or IR spectroscopy experiment also relates to the idea, "How do we know what we know?" with respect to molecular structure.

In addition to enhancing several of the learning outcomes within the AP curriculum, the Science Practices for AP Chemistry can be significantly augmented by the use of NMR and IR instrumentation. The six Sciences Practices state the following (ref 2, pp 82−85):

1. The student can use representations and models to communicate scientific phenomena and solve scientific problems.
2. The student can use mathematics appropriately.
3. The student can engage in scientific questioning to extend thinking or to guide investigations within the context of the AP course.
4. The student can plan and implement data collection strategies in relation to a particular scientific question.
5. The student can perform data analysis and evaluation of evidence.
6. The student can work with scientific explanations and theories.

Each of these practices can be addressed through the use of NMR and/or IR instrumentation. When using the instrumentation, students must draw chemical structures (practice #1). When working with integration within NMR spectroscopy, mathematics can be a significant contributor (practice #2). Practices 3−6 can all be directly linked to the use of any instrumentation in the laboratory. The specific addition of NMR and IR instrumentation will further enhance these practices. The focus of the NMR and/or IR spectroscopy lab content could center on organic molecules, structure analysis, unknown determination, and so forth. The lab could also reinforce mathematical concepts such as percent composition as described in an earlier paper in this *Journal*.[4]

Most AP Chemistry teachers would agree that it is already difficult to get through the required AP Chemistry content in time for the AP exam in early May. The following question arises, however: "How are teachers spending the remainder of the school year—up to a month or more for some school districts?" Many try to fill the time with meaningful projects that expand upon the content learned in the course. As part of their course plans, AP teachers must include activities to incorporate seven Curricular Requirements (CRs). The fourth CR (CR4) states (ref 5, p 11), "The course provides students with the opportunity to connect their knowledge of chemistry and science to major societal or technological components (e.g., concerns, technological advances, innovations) to help them become scientifically literate citizens."

Inclusion of deeper lessons on NMR and IR spectroscopy with laboratories and/or projects that include the use of these instruments would be a valuable use of the additional end of school year time and help cover CR4 in an exciting and relevant way.

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General Chemistry

In schools that do not offer IB or AP Chemistry, the chemistry curriculum is likely a bit more flexible. In this case, NMR and/or IR spectroscopy could be introduced at varying levels. As mentioned above, it could be used in connection with Lewis structures of organic molecules, for determination of unknowns, or with more mathematical content as with the percent composition lab. Along these lines, a low field NMR instrument would allow for analysis of aqueous solutions, so other concentration type laboratories could be developed. Laboratories could focus on identifying an unknown substance with a forensics theme as described by Wilson and Myers.[6] In their experiment, a sample of unknown soda was matched to a known sample through qualitative comparison of NMR spectra.

Organic or Materials Chemistry

For high schools with sufficient staff and support to offer Organic and/or Materials Chemistry courses, NMR and IR instrumentation fit perfectly. An introductory level analysis of NMR and IR spectra, either to identify unknown samples and/or verify results of a synthesis lab, is within the realm of a high school organic course. For Materials Chemistry, the use of IR with an attenuated total reflectance (ATR) accessory could greatly aid in the analysis of many materials including plastics.

Benefits for All Students

Students are typically first exposed to NMR and IR spectroscopy, including the skill of interpreting spectral data, in organic chemistry courses taken in their second or third year of college. This course is viewed by many students as an intimidating one. A study designed to determine why premedical students change majors found that among students who indicated that low grades were the reason, 78% named organic chemistry as the single course that caused them to change their career plans.[7]

As discussed above, there is an ever-expanding opportunity to introduce these concepts to students in high school. The idea of covering such complicated topics in high school might sound imposing, but introduction of the basic topics to students in a less intimidating high school setting would significantly help students when they take organic chemistry under the pressure of college work and entrance requirements for medical school, graduate school, and other professional disciplines.[8]

Hands-on activities in the laboratory are necessary to enhance empirical information taught in the high school classroom. Laboratory experiments should help students develop a deeper understanding of the content while increasing their skills in scientific reasoning. The National Research Council has defined a set of seven science learning goals that should be directly enhanced by laboratories:

1. Enhancing mastery of subject matter
2. Developing scientific reasoning
3. Understanding the complexity and ambiguity of empirical work
4. Developing practical skills
5. Understanding the nature of science
6. Cultivating interest in science and in learning science
7. Developing teamwork abilities[9]

Access to these NMR and IR instruments provides students with the opportunity to gain a deeper understanding of spectroscopy as a technique that extends beyond Beer's Law.

WORKSHOP DESCRIPTION

Advances in benchtop NMR and IR/ATR instrumentation have made robust, low cost, and portable NMR and IR spectrometers commercially available, increasing access to a broader range of institutions, including high schools. While these instruments are considered "low cost", this is relative to the cost of standard instruments. These portable, benchtop instruments are likely still out of the budget range of high schools unless they are able to obtain an unconventionally large grant. One way to circumvent this issue is through partnerships with nearby universities and businesses. Initially, this project was conceived by a faculty member of the chemistry department and the science outreach coordinator at UW—Whitewater and was funded by an internal Strategic Initiatives Grant. This grant allowed for the purchase of a low field, benchtop NMR spectrometer (Thermo Fisher Scientific picoSpin 45). The addition of an IR/ATR spectrometer (iS5) to the program did not occur until several years later, through additional collaboration with Thermo Fisher Scientific. The focus of the remainder of the discussion will be on NMR spectroscopy, but addition of IR spectroscopy is a straightforward expansion of what is discussed.

The grant had several teacher- and student-focused goals:

- Provide free educational and professional development opportunities to area teachers in an effort to help them learn more about NMR spectroscopy and to help the teachers integrate NMR spectroscopy into their classes. This includes the development of curricula they can use in their classrooms.
- Provide high school students with direct hands-on experience in, and knowledge of, technologies such as NMR spectroscopy, to be better prepared for college chemistry classes and have an academic edge over their fellow students.
- Enhance university recruitment by providing a glimpse into college level studies for high school students who may be considering attending UW—Whitewater for a science-related major.

Teacher Professional Development

The first aspect of placing the instrumentation into the high school laboratories was to train the teachers. Spectroscopy is an area in which most teachers do not feel knowledgeable enough to teach their students.[10] To remedy this, a one-day (Saturday) workshop was held during the fall semester (2013 and 2014). The exact timing of a workshop can be chosen in a way to accommodate those involved. A fall workshop worked well for our purposes in that teachers were not scattered during their off summer months, and it allowed for ample planning to work the use of the instrument in during the school year. Teachers were contacted by email through a list compiled by the Science Outreach program. Initial notification went out in the prior spring with details provided to those who responded with interest in attending. While a large number (>30) of teachers were initially contacted, no more than six teachers attended a single workshop. Keeping the number of teachers at each workshop low allowed for more one-on-one support throughout the day as well as more hands-on experience with the instrument.

To aid in the hands-on aspect, Thermo Fisher Scientific loaned our program additional instruments for the workshop. Additionally, they provided a representative who specialized in

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Table 1. Summary of Teacher Responses to Workshop Survey

Survey Question	Teacher Responses
Do you expect the material presented will be useful for implementing the picoSpin in your classroom? What information do you feel will be the most useful?	"It was extremely helpful having a refresher of NMR analysis."
	"Hands on lab experience!"
	"Visuals for NMR theory, hands-on NMR work."
	"Happy to have access to PowerPoints."
What did you like best about the picoSpin NMR Teacher Workshop?	"Being better prepared to integrate actual NMR work in the HS classroom."
	"Actual use of machine and problem solving."
	"IR would also be of use."
	"Loved that we had time to practice."
What did you like least about the picoSpin NMR Teacher Workshop?	"Computer glitches..."
	"Sometime[s] it went a little fast and I am not sure I can repeat."
	"It was all great."
	"Early start time."
	"I wish I had done this earlier."

NMR spectroscopy. This was an invaluable addition to the workshop. While this was extremely helpful, having a company representative and additional equipment is not a requirement for a successful workshop. There are opportunities for borrowing additional instruments from Thermo Fisher Scientific, but if the number of teachers participating is kept small, having a single instrument would not greatly reduce the quality of a workshop.

The workshop was designed with two sections. The first half of the day was a focus on introductory NMR spectroscopy theory and a basic lesson on NMR spectral interpretation. Many of the teachers had only a small recollection of NMR spectroscopy from their undergraduate courses. The materials were presented at a basic level, as they would not be going into great detail for their students. Topics included spin states, chemical shift and nuclear shielding, integration, splitting patterns, and a step-by-step demonstration of determining a basic chemical structure from an NMR spectrum.

Teachers were then given an NMR spectra problem set to work on with the support of the workshop leader. This allowed them to take the position of the student and practice their NMR spectral interpretation skills. Working through the problems brought up additional questions and discussion with the full group. By the end, many were relatively confident in being able to solve the spectra and being able to teach the process to their students at a basic level (see Workshop Feedback section below). The afternoon portion of the workshop was dedicated to hands-on time with the instrumentation. Teachers were shown how to set-up the instruments to enable them to do so in their own laboratories independently. The Thermo Fisher Scientific representative also provided the teachers with additional information about the instrument, how it is designed, and how it relates to the more familiar, standard, high field NMR spectrometers.

After learning about the setup process, teachers worked with injecting samples, collecting data, and working through the data-workup process. The NMR analysis software from Advanced Chemistry Development, Inc. (ACD/Laboratories) was used.[11] At the time of our workshops, this program had free access for educators allowing teachers to download the program on any computer they wish, not just the computer supplied with the instrument. ACD/Laboratories now charges for this program. Having extra instruments provided by Thermo Fisher Scientific allowed for smaller groups of teachers to work together, giving them more hands-on time with the instruments. As mentioned above, this can still be successfully accomplished with a single instrument, provided the teacher count is kept low. To help focus the time, teachers were provided with a lab to determine the percent composition.[4] This is an experiment the teachers can do with general chemistry students without having to go into great detail about spectral interpretation. The lab focuses on calculations such as mole to gram conversions, density, and percent composition.

At the end of the day, teachers were given time to discuss ways they thought they could incorporate the use of the instrument into their various courses. This provided ideas for other teachers and allowed the coordinators to provide advice on what would work for them. Workshop materials (PowerPoint slides, NMR spectroscopy problem set, etc.), new lab ideas from teachers, training videos, and other items were shared by archiving them on an open-access website developed and maintained by Dr. Bonjour.[12]

The professional development (PD) that is produced by such a workshop can be designed in several ways to best appeal to area teachers. The workshop can provide teachers with

- PD hours required for certification
- Graduate credits required for certification or salary schedule advancement
- Ways to bring modern techniques to their classroom
- A networking opportunity
- An opportunity for PD in their content area, which can be rare and difficult to find

Workshop Feedback

After the completion of the workshops, teacher participants were asked to complete an IRB approved survey. Many of the questions asked were designed to improve future workshops. Some of the widely useful responses are summarized in Table 1.

USE IN THE HIGH SCHOOL CLASSROOM

After teachers complete the workshop, they are allowed to schedule use of the NMR spectrometer at their school. Scheduling is organized through an online calendar found on the UWW picoSpin Outreach Web site.[12] The NMR spectrometer and now the IR spectrometer are both quite robust and travel well. They are delivered to the high school and set up by Dr. Bonjour or an outreach worker for the initial use. After teachers are comfortable enough to set up the

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instruments themselves, distribution is sometimes made by meeting at a convenient location for both parties, but could easily be shipped. Schools usually keep the instrument for a week or two depending on demand. We have found the end of the spring semester to be a busy time as that is when it can be used in AP and IB courses.

Use by area high schools since late 2013 is shown in Table 2. The equipment has been used in general chemistry, AP chemistry, IB chemistry, and organic chemistry courses by nearly 170 students. A number of different types of experiments have been completed. Some teachers have chosen to use the percent composition lab provided to them at the workshop, whether it is as described or with modifications.[4] Others have chosen to do laboratories that involve known and unknown samples. Examples of some of the known and unknown experiments have been shared on the UWW picoSpin Outreach Web site and include some student work samples.[12] Edgerton High School has their AP students do a project at the end of the year. Two of their students made biofuels and chose to qualitatively analyze them using the picoSpin NMR spectrometer. Additional resources for educational laboratories have been published by Thermo Fisher Scientific: *Educational Experiments Vol. 1 and 2* and *NMR Lesson Plans*.[13,14] The addition of the FT-IR instrument is allowed for more detailed known and unknown analysis.

Table 2. Summary of NMR Use in Southeast Wisconsin Area High Schools

High School	Number of Students Using Instrument	Number of Semesters Used	Types of Experiments
Elkhorn, WI	16	1 (2013)	Unknowns and percent composition
Edgerton, WI	2	1 (2014)	AP project, biofuels
Cedarburg, WI	122	4 (2014–2016)	Knowns and unknowns
East Troy, WI	Teacher ran samples	1 (2015)	Knowns
Westosha, WI	5	1 (2015)	Percent composition
Oconomowoc, WI	24	2 (2015–2016)	Knowns and unknowns

By providing the teachers with the opportunity to have the instruments at their school, they have the ability to use it over several days. Many high schools have classes as short at 50 min. This does not allow adequate time to complete an experiment in a single day. It also provides the opportunity for the teachers to expand beyond the experiment ideas they are given during the workshop. Mr. Pollock at Cedarburg High School has developed an experiment to monitor a substitution reaction in his organic class. He has also considered having students that participate in SMART (Students Modeling A Research Topic) Teams use the NMR spectrometer to further their project.[15] This more in-depth use would not be possible if instead the high school students came to the campus for a visit. In addition to the restrictions with travel (money for buses, permissions from parents, scheduling), limited time would be allowed for students to run samples, likely only on a previously used experiment. Beyond the UW—Whitewater project, Themo Fisher Scientific and The Royal Society of Chemistry in London have a total of 15 traveling picoSpin units providing high schools with access to the instrumentation. In the development of the percent composition lab, and since, over 225 students at Lakeland Union High School in Minocqua, WI, have been using a picoSpin NMR spectrometer to run the experiment.

UNIVERSITIES AND RECRUITMENT

Glimpse into College

Through the partnerships developed between high school chemistry teachers and university professors during outreach projects such as the one described here, high school students can be provided with a glimpse into college. Not only will they be exposed to instrumentation they may see at the college level, but also accompanying opportunities, such as having a university faculty member or upper level undergraduate chemistry major talk to the class or even run a lab with the instruments at the high school, exist and allow students to interact closely with those they might encounter at the university. Also, the teachers can have discussions with their students about the outreach project and how the university is providing the opportunity to work with the instruments. This is not only beneficial to the students, but is also a great recruitment tool for the university as students are able to experience the resources they would have access to at the university as well as understand the opportunities for undergraduate research that may exist for them. A student from Cedarburg High School will be starting as a chemistry major at UW—Whitewater this fall. After her use of the instrumentation in her Organic Chemistry course with Mr. Pollock, she stated:

> *I enjoyed getting to work with borrowed instruments and the fact that it was more modern technology. It was really a different kind of chemistry. I enjoyed getting to use equipment that pro scientists get to work with.*

University Benefits

While the biggest gains for this type of outreach project are for the high school teachers and students, there are also benefits for the university. Additional goals of this project were more university focused:

- Expand the number of high school students that UW—Whitewater Science Outreach can reach and the opportunities and experiences that Science Outreach can provide.
- Provide high impact practices to UW—Whitewater students taking organic chemistry lab (including chemistry majors, biology majors, and preprofessional students) by exposing them to a novel version of a frequently used instrument.
- Provide students with disabilities the opportunity to use an NMR spectrometer. Some of these students may otherwise be unable to use this type of equipment due to the high magnetic fields and the physical size of traditional NMR instruments.

In addition to being a potential recruiting tool for future students, the university can use the instruments in their own teaching and research laboratories. Students gain experience running experiments on a novel instrument while allowing them to apply their theoretical knowledge of spectroscopy. Additionally, the use of a benchtop NMR instrument will drive their skills in NMR spectroscopy data analysis, which is a key requirement in the course as described by the American

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Chemical Society Certification.[16] With more than 500 picoSpin instruments in academic institutions, this type of project could be developed by using existing instrumentation.[17]

CONCLUSION

UW—Whitewater's Chemistry Department and Science Outreach have successfully developed a teacher professional development workshop to allow high school teachers the opportunity to provide their students with hands-on experience using NMR and IR portable, benchtop spectrometers in their high school classrooms. This exposure to advanced instrumentation can be directly mapped to IB and AP Chemistry curricula, as well as fit directly into general, organic, or materials courses at the high school level.

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Notes

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