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Oregon NMR Consortium: A Collaboratory for NMR Data Acquisition and Processing

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The Oregon NMR consortium was created to provide access to a modern, high-field 400 MHz NMR spectrometer for students and faculty at several two-year and small four-year institutions. Students use both on-site and remote access to conduct their NMR experiments. Remote access involves connecting to the NMR console over the Internet. Features of the consortium are described, including the details of remote access, sample transport, and data processing. This paper also discusses the impact that the access to the NMR spectrometer has had on student achievement of several learning goals in the organic chemistry courses at the partner institutions.

Introduction

Chemical research and chemical laboratory education have become increasingly dependent upon instrumentation. A quick survey of the *Journal of Chemical Education* reveals a large number of laboratory experiments that involve some type of chemical instrumentation. Chemical educators need to keep pace with this development in order to prepare their students for future success in graduate school and chemical industry careers. However, the high cost of some instruments has limited the access of many students and faculty, primarily at small undergraduate institutions. In particular, high-field nuclear magnetic resonance (NMR) spectroscopy is not available at many colleges and universities due to the high cost (> $200K) of a new instrument as well as the ongoing cost of cryogens for the maintenance of the superconducting magnet. Even used or reconditioned NMR spectrometers are beyond the budget of many small schools.

The lack of access to an NMR spectrometer is unfortunate as NMR spectroscopy has become one of the most important methods for structural analysis and determination of compounds. The central importance of NMR spectroscopy to the undergraduate chemistry curriculum is underscored by the fact that an NMR spectrometer is the only instrument required for program certification by the American Chemical Society Committee on Professional Training (*1*). Many computer and Internet-based simulations and databases have been developed that provide users access to virtual NMR spectroscopy data (*2*). These simulations no doubt help students develop the skills of spectroscopic analysis and data interpretation. However, they do not provide students with the opportunity to execute experiments or to analyze samples that they have prepared themselves.

The increased reliance on chemical instrumentation has been matched by an increase in educational computer-based networks (*3*). Most college campuses have local area networks as well as access to the World Wide Web. As almost all modern chemical instrumentation involves a computer interface and digital data acquisition and storage, instruments have increasingly been placed on networked systems. "Collaboratories" or shared instrumentation facilities have been designed to meet the needs of regional (*4–8*) and international users (*9, 10*). Indeed, a number of collaboratories have been developed over the last decade, such as the Integrated Laboratory Network (ILN) at Western Washington University (*11*). Collaboratories have lowered the barrier to access for many instruments, and students and faculty can now perform experiments in real time through the remote control of instruments at a distant location.

While access to an instrument through a collaboratory does provide some opportunities, there are still several barriers that limit the impact of these networked instruments on chemical education. Remote access is possible and has been achieved in a number of places, but delivery of samples from a remote site is an issue. The distance from a remote site to the host facility may limit the opportunity to perform experiments on samples that students have prepared themselves within a short time frame. In addition, many faculty members may lack the training to operate the equipment, the expertise to guide students through the data interpretation steps, or the confidence to update their chemistry curriculum with new experiments.

To address these and other issues the Oregon NMR Consortium was created with four objectives: 1) increase understanding of chemistry through hands-on access to modern NMR technology; 2) increase competency in using modern chemical instrumentation in teaching laboratories; 3) develop technical expertise among chemistry faculty; and 4) enhance chemical instruction at all consortium institutions by sharing curriculum ideas, teaching pedagogies, and assessment information.

The Oregon NMR Consortium was not designed to serve the NMR spectrometer needs of all chemical education institutions in the region, but rather the membership of the Oregon NMR Consortium was intentionally drawn from institutions that serve primarily undergraduate student populations and that did not have an NMR spectrometer or convenient access to an instrument. The schools in the Oregon NMR consortium are small, private four-year institutions and a two-year public community college. In 2010, private four-year and public two-year institutions enrolled 3.9 million and 7.2 million students, respectively, which represented 63 percent of the total students enrolled in undergraduate programs (*12*). While many of the students at private four-year and public two-year institutions do have access to NMR spectroscopy, it is safe to assume that some percentage of these students have no access to NMR spectroscopy instrumentation at all.

Table 1. Overview of Oregon NMR Consortium member institutions

	GFU	*PCC*	*CU*	*WPC*
Location	Newberg	Portland	Salem	Portland
Type	Private, 4-yr	Public, 2-yr	Private, 4-yr	Private, 4-yr
Total undergraduate student population	1500	27,000	750	500
Offers BS/BA in chemistry	Yes	No	No	No
Highest CHEM offering	Physical	Organic	Organic	Organic
Typical Organic Chem. enrollment	45	35	12	5, alt yrs
Distance from GFU, Newberg		17 miles	38 miles	30 miles
Distance from GFU, branch		2 miles	6 miles	10 miles

Through the support of the Course Curriculum and Laboratory Improvement (CCLI) program of the National Science Foundation (NSF), the Chemistry Department at George Fox University in Newberg, Ore. purchased a new NMR spectrometer. George Fox is responsible for the maintenance and upkeep of the spectrometer. The first section of the proposal to the NSF-CCLI program dealt with curricular changes that took place at George Fox. As a result of the CCLI support a number of laboratory experiences were upgraded or introduced to take advantage of the new instrument. The second section of the CCLI proposal, which is the focus of this report, concerns the development of the Oregon NMR Consortium which has had a significant impact on the regional chemistry educational community.

Program Design

Overview

The Oregon NMR Consortium initially involved three institutions: George Fox University (GFU), Warner Pacific College (WPC) and Corban University (CU), with Portland Community College (PCC) joining shortly after the consortium was launched. The four institutions of the consortium represent a mix of large and small, public and private, urban and rural institutions with diverse chemistry curricula and student populations (Table 1). Of the consortium members, GFU, PCC and Corban University have made significant use of the instrument during the project. Access to the NMR spectrometer is free for all consortium members, with each user group being responsible for purchasing their own sample tubes and deuterated solvents, as well as preparing and labeling their samples.

Equipment

The equipment selected and purchased for this project was intentionally chosen to meet the program objectives. The NMR spectrometer is a JEOL ECS-400 which has the standard features of most modern NMR spectrometers, including a 9.39 T superconducting magnet, a two channel broadband probe, gradient shimming, an autotune unit and a low-temperature/variable-temperature accessory. While these items are certainly important with respect to project success, other features of the JEOL instrument proved just as crucial. The instrument also was equipped with a 24-sample autosample carousel, JEOL data processing software (Delta) that can be loaded and used separately from the acquisition software, unlimited acquisition and processing software licenses for Mac, Windows or Linux operating systems, and a hardware/software interface that uses an Ethernet connection to the NMR console from any workstation equipped with the JEOL software. Any workstation can be used to access and control the JEOL NMR spectrometer so long as it is connected to the Internet and hosts the JEOL Delta acquisition software (Figure 1). In fact, all users connect to the NMR spectrometer through the network, including those who initiate experiments and collect data locally at the NMR facility. In this sense, everyone is a remote or client user of the instrument. The appearance and features of the JEOL acquisition software are the same for all users at all locations. Consequently, students who operate the instrument from both the local workstation and from a remote location will experience the same software interface. To maintain network security, the George Fox institutional technology department has configured the institutional firewall to permit access to the NMR spectrometer by known clients who attempt to connect to the instrument from a remote site.

The NMR spectrometer console can be owned only by one workstation at a time. Moreover, experiments can be initiated only when the spectrometer is owned by a workstation. At the conclusion of an NMR experiment, the final package of spectroscopic data is transmitted to whichever workstation initiated the experiment. This approach to data storage eliminates the need for any subsequent data transfer or retrieval from a primary server. We have restricted the acquisition software to one workstation at each consortium site to avoid too many users trying to initiate experiments at the same time. However, the processing software is available to anyone in the consortium who wishes to have it, and the unlimited number of software processing licenses means that all faculty and students in the consortium are able to load the JEOL software on their personal computers. Spectroscopic data can also be sent by email from the workstation that initiated the experiment, which allows students to view and process spectra without waiting for access to a single, central workstation.

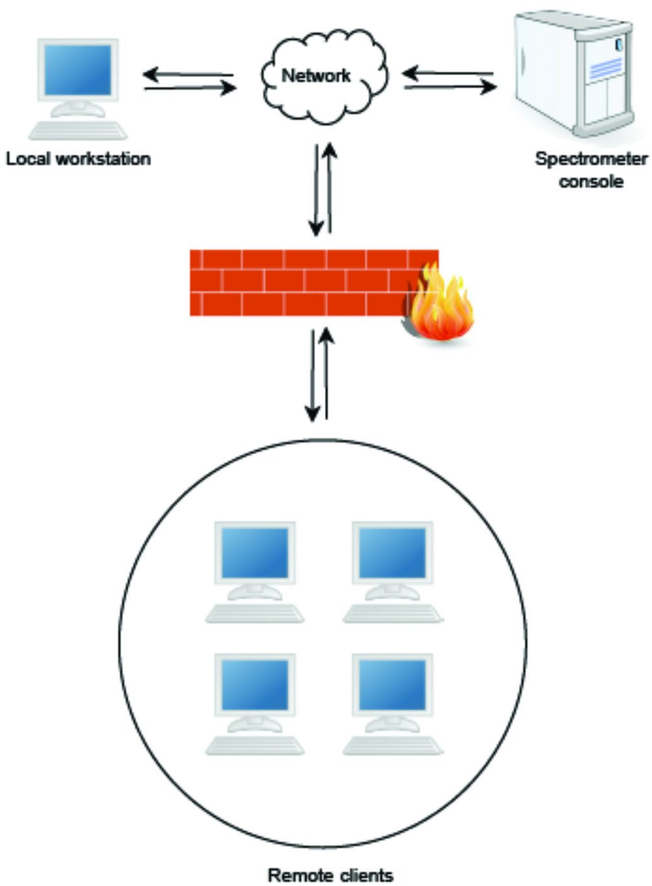

Figure 1. Consortium network configuration for users.

Activities

A planning session was held at George Fox University before the NMR spectrometer was installed. At that meeting the consortium members discussed the features of the instrument, the logistics of on-site and remote access, the types of experiments that could be shared among the member institutions, and plans for common assessment instruments. After the NMR spectrometer had been delivered and installed, an initial training session was held at which representatives from each of the consortium members were trained on basic procedures, such as loading and unloading samples, locking and shimming samples, and selecting and executing a simple ^{1}H NMR experiment while operating the instrument on-site. On the basis of the initial training session, several stand-alone modules were developed by GFU faculty to provide more detailed training in sample preparation, remote access of the instrument, obtaining an FID, generating a processed spectrum and obtaining integration and peak information from a ^{1}H spectrum. The training modules were initially developed for faculty training on the NMR spectrometer and have since been modified for introducing students to the basic principles of NMR data acquisition and interpretation. All of the training materials and tutorials are available on the consortium website that is hosted by George Fox University.

In subsequent meetings, the consortium has discussed specific experiments that can be added to the chemistry curriculum, along with the development of assessment instruments to evaluate the NMR project. Both curricular changes and assessment data will be discussed below. Member institutions have continued to meet at the conclusion of each academic year, with evaluation of assessment data, discussion of best teaching practices in the undergraduate laboratory, and faculty development in NMR spectral data interpretation being topics at these annual consortium workshops.

Curriculum

The theory and practice of NMR spectroscopy has been integrated into the chemistry curricula at the various consortium institutions. The faculty at each college determines the specific curricular changes, such as experiment choice and scheduling. In general, each institution introduces compound identification using a variety of techniques, including NMR spectroscopy, during the first semester or term of the organic chemistry sequence. Specific experiments or laboratory activities are then incorporated into the curricula throughout the remainder of the academic year according to the schedules prepared by the individual instructors. There are several common experiments that are performed by all members of the consortium (Table 2). One experiment (Table 2, Experiment 1) that has been shared, is an active learning experiment developed at GFU for general chemistry students that relates the chemical shifts of a homologous set of small organic molecules to electronegativity trends (*13*). In addition, a set of NMR samples, containing simple organic molecules that are used to introduce the principles of ^{1}H and ^{13}C NMR spectral interpretation, were prepared and are available for use by the various consortium members (Table 2, Experiment 2).

Table 2. Common NMR Spectroscopy Experiments

Experiment	*Course*
The effect of atom structure on electron clouds	General
Introduction to organic structure determination using ^{1}H and ^{13}C NMR data	Organic
^{13}C NMR spectroscopy identification of alcohols	Organic
^{1}H and ^{13}C NMR analysis in banana oil synthesis	Organic

Sample Transport and Analysis at Remote Locations

The transport of samples to the NMR spectrometer facility at George Fox from the other consortium members is one of the key issues in the success of the consortium. Typically, students and faculty from the consortium partners travel to the GFU-Newberg campus for their first NMR experiment in the Organic Chemistry course. They have the opportunity to see the instrument, directly experience inserting samples into the magnet bore, execute an NMR experiment, and process the spectral data. The Chemistry Department at GFU has a computer workroom with several workstations that contain the JEOL Delta processing software. However, while there is value in hands-on use of the NMR spectrometer, the lack of a credible sample transport system could lessen some of the benefits of real-time access and control of the instrument from a remote location since the consortium members are too far from the GFU-Newberg campus to travel to the NMR facility for sample analysis on a regular basis. Other collaboratory arrangements have used mail delivery to transport samples to the host NMR facility (*5*, *9*). However, in addition to the cost associated with mail delivery, this system would likely involve several days between sample preparation and arrival at the NMR facility.

The Oregon NMR Consortium has developed a system in which a one-day turnaround time is possible for samples sent to the NMR facility at George Fox University. In addition to the main campus in Newberg, George Fox University also has branch sites at other locations throughout Oregon, including Portland and Salem. These branch locations are very close to the home institutions of the consortium partners (Table 1). George Fox University employs a courier system through its mail services department for transporting internal mail, supplies and other items to the branch locations. The GFU courier makes daily trips to the branch campuses from Newberg, and we have used this system to transport samples between the remote partners and the NMR facility.

The U.S. Department of Transportation allows for the transport of small amounts (< 30 mL) of hazardous materials by courier under a limited quantity regulation (*14*). Under this arrangement, NMR samples are placed inside a metal canister that is lined with adsorbent material. The canister is then placed inside a sturdy five-gallon bucket that is packed with additional adsorbent material. When a consortium member is ready to send samples to the NMR facility they contact the project director who sends the empty transport container through the GFU courier system to the appropriate branch location. The samples are then placed in the container and returned to the Newberg campus via the GFU courier system. When the samples are received in Newberg they are loaded into the 24-sample carousel for the remote partners to access. The faculty and students at the remote location then connect to the NMR and conduct the experiments on their samples using the Delta data acquisition software running on a remote client workstation. For large laboratory sections, the gradient shimming routine and software macros available with the JEOL Delta system streamline the acquisition process. Again, the spectroscopy data is owned by the workstation that initiates the experiment and can be subsequently sent to a student email account for later processing. Upon completion of the NMR analysis the GFU courier system is used to return the samples from the GFU-Newberg campus to the appropriate branch location for pick up by a representative of the cohort institution.

Project Assessment

Overview

The assessment plan involved an evaluation of active consortium members over the length of the project. Three general areas were evaluated in the project: (i) student success in achieving the NMR learning goals; (ii) student learning through remote access to the NMR spectrometer; (iii) student confidence in using modern chemical technology. The assessment plan involved administering questionnaires to students before and after installation of the NMR spectrometer. Baseline responses (Year 0) were collected from George Fox students before the installation of the NMR spectrometer. The populations evaluated in this project were the student cohorts in the organic chemistry courses at all of the participating consortium institutions. Evaluation of the students in organic chemistry occurred at the end of the last semester or term of the course, and the baseline results were likewise collected at an analogous point in the spring semester of Year 0. The assessment plan was not designed as a rigorous evaluation of an academic experiment, but rather as an examination of the extent to which the project goals were achieved.

Assessment of NMR Learning Goals

Several NMR spectroscopy learning goals were surveyed in the various consortium Organic Chemistry courses (Table 3). The assessment instrument used a 5-pt Lickert scale (1-strongly agree, 5-strongly disagree) for evaluating the extent to which students achieved the learning goals.

Table 3. Assessment scores of NMR learning goals by various student cohorts in Organic Chemistry[a]

		GFU		*PCC*		*CU*	
Learning Goal	*Year 0*	*Year 1*	*Year 2*	*Year 1*	*Year 2*	*Year 1*	*Year 2*
I have a good understanding of the meaning of chemical shift in an NMR spectrum.	1.7	1.8	1.4	1.6	1.5	2.5	2.2
I have a good understanding of the meaning of peak integration in an NMR spectrum.	1.7	2.0	1.5	1.6	1.4	2.3	2.0
I am able to interpret correctly a one-dimensional ^{1}H NMR spectrum using chemical shift and peak integration information.	1.7	1.5	1.5	1.5	1.4	3.1	1.4
I am able to interpret correctly a one-dimensional ^{13}C NMR spectrum using chemical shift information.	3.1	1.6	1.5	2.3	2.2	3.1	2.4

[a] Students were asked to respond to the Learning Goal statements with the following scale: 1-strongly agree; 2-agree; 3-neither agree nor disagree; 4-disagree; 5-strongly disagree.

The learning goals (1 – 3) that involve the analysis of ^{1}H NMR spectroscopy data are essentially the same for the study groups as for the baseline cohort (Year 0). As the analysis of ^{1}H NMR data was part of the curricula before the installation of the high field spectrometer it is not surprising that the scores are similar. A significant increase in the ability to analyze ^{13}C NMR data (Goal 4) was observed in all years of the project. The theory of ^{13}C NMR spectroscopy was discussed in lecture settings prior to the installation of the spectrometer. Students in Year 0 and preceding years received instruction in the analysis of ^{13}C NMR data by using spectra from textbooks or databases and online simulations. However, the practice of ^{13}C NMR spectroscopy was not covered at any of the consortium institutions before the new NMR spectrometer was installed due to the lack of an instrument that was capable of this type of analysis. The gains in learning related to ^{13}C NMR spectroscopy have been realized over several years. Moreover, the first cohort of students at Corban University (CU, Year 1) did not take advantage of the ^{13}C capabilities of the new instrument. However, in Year 2, experiments that involved ^{13}C NMR data analysis were added to the Organic Chemistry curriculum at CU and the score for this learning goal showed a corresponding increase. The results in Table 3 indicate that the actual practice of NMR analysis is important in achieving learning goals associated with NMR data interpretation.

Assessment of Student Attitudes on Using Remote Access To Operate the NMR Spectrometer

We were also interested in the attitudes of consortium students who primarily used remote control from their home institution to operate the NMR spectrometer (Table 4). The assessment instrument for evaluating this project goal again used a 5-pt Lickert scale (1-strongly agree, 5-strongly disagree.) Thus far, only the students at PCC have made extensive use of the remote access capabilities of the NMR spectrometer. These students typically come to the GFU-Newberg campus for their first experience with the NMR technology, and in this initial meeting they have the opportunity to load samples, shim the instrument, execute experiments, and process data. All of their subsequent experiments are conducted by remote operation of the instrument from their home campus. It's worth noting that the experiments conducted by remote control involve samples the PCC students have prepared themselves and sent to GFU by courier. At the end of the organic chemistry course, and after they had conducted several other NMR experiments, they were asked if the remote access had hampered their achieving the learning objectives (Table 4). According to the student responses the remote access is a viable method for achieving the desired NMR outcomes. They reported that remote execution of experiments was very similar to direct operation of the NMR spectrometer, and that the remote access did not hamper their understanding of NMR technology or their interpretation of NMR spectroscopic data.

Table 4. Assessment scores of student attitudes on using remote access by student cohorts in Organic Chemistry at PCC[a]

Question	*Year 1*	*Year 2*
Operating the NMR by remote control from PCC was as easy as operating the instrument by direct control at GFU.	1.7	2.0
Using the instrument by remote control did not hamper my understanding of the NMR technology.	1.7	2.0
Using the instrument by remote control did not hamper my understanding of the NMR data.	1.6	1.6

[a] Students were asked to respond to the statements with the following scale: 1-strongly agree; 2-agree; 3-neither agree nor disagree; 4-disagree; 5-strongly disagree.

Assessment of Confidence in Using Modern Chemical Technology

The extent to which students gained confidence in using modern chemical technology as a consequence of their hands-on experience with the 400 MHz NMR was also assessed. (Table 5) The assessment instrument once again used a 5-pt Lickert scale (1-very confident, 5-not confident) for evaluating this project goal. Prior to the installation of the high-field spectrometer at George Fox, all NMR experiments were performed by the course instructor or laboratory teaching assistant rather than the students in organic chemistry. The baseline score of 4.0 expressed by students in Year 0 for confidence in their ability to correctly operate the NMR indicates the low confidence that students had in this skill. We also asked the students to express their confidence in using several other instruments, FT-IR, fluorescence spectroscopy, and electrochemistry. The students in Organic Chemistry at George Fox routinely use infrared spectroscopy to analyze laboratory products and the Year 0 baseline score indicates a high level of confidence with this instrument. Experiments that use fluorescence spectroscopy or electrochemistry techniques are not part of the GFU organic chemistry curriculum, nor are these techniques used in the organic chemistry courses at the other consortium institutions. Not surprisingly, the students expressed a low confidence in their ability to correctly operate these instruments in Year 0.

The increased hands-on experience in using the 400 MHz NMR spectrometer corresponded to a higher level of expressed confidence by students at all of the consortium schools. Prior to their participation in the Oregon NMR consortium, the students at PCC and Corban did not have any direct experience in operating a modern NMR spectrometer. Their scores in both years 1 and 2 closely track the values reported by students at GFU.

Table 5. Assessment scores of student cohorts in organic chemistry when asked "For the following instrumental techniques I am confident in my ability to operate the instrument correctly."[a]

	GFU			*PCC*		*CU*	
	Year 0	*Year 1*	*Year 2*	*Year 1*	*Year 2*	*Year 1*	*Year 2*
NMR	4.0	1.3	1.4	1.9	1.7	1.4	1.3
FT-IR	1.5	1.4	1.6	1.4	1.5	4.5	- -
Fluorescence	4.4	3.1	3.2	5.0	4.0	4.5	5.0
Electrochemistry	4.4	3.0	3.2	5.0	4.8	4.0	5.0

[a] Students were asked to respond to the confidence statement with the following scale: 1-very confident; 2-confident; 3-somewhat confident; 4-low confidence; 5-not confident.

An interesting result from this assessment data involves the changes in student confidence in operating other chemical instruments. As noted earlier, none of the students at the consortium schools used fluorescence or electrochemistry techniques in the organic chemistry curriculum prior to the beginning of the NMR project. The NMR project did not change the Organic Chemistry curriculum with respect to fluorescence and electrochemistry techniques, however, GFU students in Organic Chemistry Year 1 and 2 cohorts expressed a higher level of confidence in their ability to conduct experiments using these techniques. We don't know if these differences are statistically significant, but the experience of using chemical instrumentation in the form of a modern NMR spectrometer may have had a positive impact on students' confidence in using unfamiliar instrumentation. A possible explanation is that student experience with the NMR spectrometer has made modern chemical instrumentation more accessible and less of a black box.

Conclusions

The Oregon NMR Consortium has provided access to a modern, high-field NMR spectrometer for a large number of students in our region who previously could not easily use this type of instrumentation. Many of the barriers that limit the impact of remote access to instrumentation have been overcome. We have developed a convenient strategy for transporting samples between institutions. The instrumentation and software used in this project permit real-time access for students at remote institutions. The combination of sample transport and computer access has made it possible for the students at remote sites to analyze samples they have prepared with one-day wait times. The assessment information strongly indicates that the consortium has had a positive impact on the laboratory experiences of the students at the partner institutions, with students having an increased understanding of NMR interpretation and, we hope, the underlying, fundamental chemical principles. We have also found that actual experience of operating the NMR spectrometer appears to have led to an increased student confidence in using any modern chemical instrument. Anecdotal information from interviews with faculty members at consortium institutions confirms that real-time access to a modern NMR spectrometer has also positively impacted faculty development. In ways that parallel the student responses, faculty note a better understanding of chemical and NMR concepts, as well as a higher level of confidence in using modern chemical instrumentation. The NMR consortium has helped develop and strengthen relationships between the participating institutions, and we are continuing to explore other ways in which these relationships could be used to enhance chemical education in our region.

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