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A Community Springs to Action to Enable Virtual Laboratory Instruction

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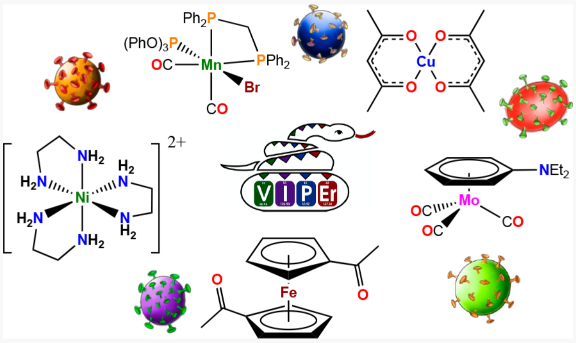
ABSTRACT: The COVID-19 pandemic put an abrupt end to face-to-face teaching at many colleges and universities. While numerous methods for synchronous and asynchronous teaching in lecture courses were quickly deployed, laboratory instruction presented a unique set of challenges. To develop online laboratories quickly would be a daunting task for an individual. However, contributions of many individuals could ease and hasten the transition to using virtual laboratories. The IONiC-VIPEr community of practice is one such community that sprung to action to provide virtual alternatives for inorganic chemistry laboratory instruction.



KEYWORDS: *First-Year Undergraduate, Second-Year Undergraduate, Upper-Division Undergraduate, Inorganic Chemistry, Laboratory Instruction, Analogies/Transfer, Distance Learning/Self Instruction, Coordination Compounds, Electrochemistry, Organometallics*

■ INTRODUCTION

On March 10, 2020, a BITeS (blog post) titled "Asynchronous online teaching in the COVID-19 era" appeared on the Virtual Inorganic Pedagogical Electronic Resource (VIPEr) Web site—ionicviper.org.[1] This was three days before a national emergency was declared in the United States[2] and nine days before California became the first state to issue a statewide stay at home order.[3] In this BITeS the author (A.J.) voiced the concerns shared by thousands of chemistry instructors, "How do I transition to completely remote teaching in a very short period of time?" Not surprisingly, this quickly became one of the most viewed and most commented on BITeS on the site.[4]

The answer to the question was given by the mere fact that there was a venue to express what the author and so many others were wondering. The VIPEr site is the web presence of a community of practice—the Interactive Online Network of Inorganic Chemists (IONiC).[5−7] The crux of this BITeS was that the solution to this sudden transition to remote teaching was not going to come from a single person, it would come from a supportive community of people facing the same challenges. The idea of developing half a semester of online materials for a lecture course in a matter of days is daunting to say the least. To have to do the same thing for the corresponding laboratory seemed nearly impossible. As inorganic courses do not have the large numbers of students that go through the general chemistry and organic chemistry sequence, online laboratory resources for inorganic chemistry are limited. However, the IONiC community was quick to action, and several individuals were able to share online laboratory experiences on the VIPEr website. Those laboratories were made available to the entire inorganic teaching community to help others make this transition.

This article discusses four virtual laboratories and one virtual tutorial that were developed using different methodologies in a rapid response to the COVID-19 pandemic. These laboratory experiences cover topics from coordination chemistry, electrochemistry, and organometallic chemistry. The learning objects (LOs) are freely available on the VIPEr site. In addition, VIPEr already had two laboratory experiment LOs that were updated with additional data to make them more widely usable as virtual experiments. This discussion will focus on the planning and work that went into the rapid deployment of these online experiences which were ultimately combined as a collection on VIPEr.[8] During this time period we were cognizant of a blog post that encouraged us to do our best to deliver online instruction and to not worry about doing "a bad job".[9] This was not a time for nuanced deliberation on the best practices of distance learning. We knew that we were not going to be

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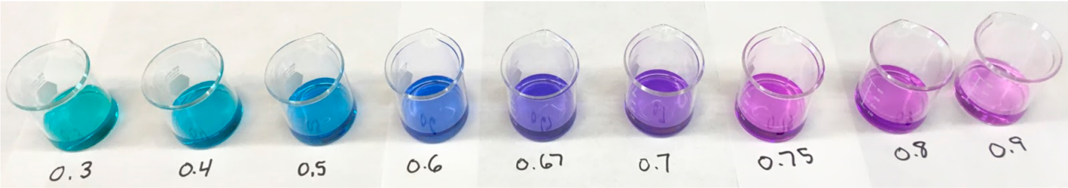

Figure 1. Prepared solutions of Nickel(II) sulfate with different mole fractions of ethylenediamine (photo taken at Lafayette College March 22, 2020 by Chip Nataro).

able to replicate years of online education experience, but we also knew we had to get something deliverable. We will also discuss problems and unforeseen issues with their development, and some thoughts as we look to the future.

■ JOB'S METHOD LAB

This is a classic lab used in general chemistry and inorganic courses.[10] The lab exercise requires students to make stock solutions of nickel(II) sulfate and ethylenediamine and then use those solutions to generate new solutions with varying mole ratios of the metal and ligand. Students are always amazed at the colors of the complexes ranging from blue to purple that are generated starting with a green nickel(II) solution and adding the colorless solution of ethylenediamine (Figure 1). While students can always use practice making solutions, the techniques required for this lab are common and somewhat repetitive, so the focus of the virtual experiment was data analysis. We felt that this popular experiment was well-suited for rapid conversion into a virtual laboratory. It was easy to make short videos showing each solution being made and then obtain the UV−vis spectroscopic data for each solution for the students to analyze.[11] This was obtained in the space of about 2 h the evening that Governor Wolf announced the state of Pennsylvania would be shutting down.

■ ACYLATION OF FERROCENE

On March 17th, 2020, a post in the VIPEr forums requested unprocessed NMR data for students to analyze, particularly of organometallic compounds.[12] The acylation of ferrocene is another classic laboratory exercise in which ferrocene is converted to 1-acetylferrocene and 1,1′-diacetylferrocene.[13,14] With limited time it was not possible to perform this laboratory exercise, however samples of all three compounds were readily available in my (CN) research laboratory. After obtaining standard ^{1}H and ^{13}C{^{1}H} spectra, I then took advantage of our instrument's automation features and acquired distortionless enhancement by polarization transfer (DEPT), correlated spectroscopy (COSY), heteronuclear multiple bond correlation (HMBC), and heteronuclear single quantum coherence (HSQC) NMR spectra. The NMR samples were then reverse engineered into a crude reaction mixture by being combined, and GC−MS data were obtained of this mixture. The unprocessed NMR and MS data were posted on the VIPEr site along with links to two excellent, pre-existing videos showing this experiment being performed in the laboratory, including the chromatographic separation of the different compounds.[15,16] Soon after posting this material on VIPEr and announcing it through various social media platforms, I was contacted by the second author of this LO with an offer to provide additional characterization data for these compounds, including IR and UV−vis spectroscopy and cyclic voltammetry.[17] All of this data can be manipulated and analyzed by students. This experiment provides a prime example of how a virtual community can rally to support the needs of each other.

■ ELECTROCHEMISTRY

The experiment was rapidly developed to replace the normal in-person general chemistry experiment at Hope College.[18] It has the same student learning outcomes as the in-person experiment: writing balanced redox reactions, calculating cell voltages under standard and nonstandard conditions, using the Nernst Equation to create a graph for a concentration cell, and using that graph to find the concentration of an unknown given its cell potential. Students use an online electrochemical cell simulator[19] to construct electrochemical cells, measure voltages, and interpret results. Resources for students include a template with instructions for the lab and places to record their answers, a video introduction to electrochemistry, and a video tutorial explaining how to use the simulation software. Resources for faculty include electrode assignments (students work with different cells), an answer key, and instructor notes.

■ ARENE CARBONYL LAB

The synthesis of group-6 metal arene complexes from their corresponding carbonyl complexes is a classic organometallic experiment that introduces students to π-backbonding and the use of carbonyls as reporter ligands.[20−24] For the past several years at Harvey Mudd College, we have collected data using several different arenes to assemble a class data set. Students can examine the carbonyl stretching frequency as a function of different arenes to see that more electron donating ligands caused a larger decrease in the ν_{CO} according to the Dewar-Chatt-Duncanson bonding model. Students submitted their raw spectra as part of their reports in prior years, so I (A.J.) had access to data (^{1}H NMR, IR, APCI-MS, mp) for three $[Cr(\eta^6\text{-arene})(CO)_3]$ complexes (arene = *N,N*-dimethylaniline, *N,N*-diethylaniline and mesitylene) and two $[Mo(\eta^6\text{-arene})(CO)_3]$ complexes (arene = *N,N*-dimethylaniline, *N,N*-diethylaniline). We have had some difficulty replicating the literature procedures for phenols and unsubstituted anilines,[24] so our data set is smaller than might be hoped for when looking for trends. In fact, the spring of 2020 was to see an increase in the number of arenes tested in this experiment to complement the local data set. As IR stretching frequencies are relatively sensitive to phase (Hunter et al. report solution IR in CH_2Cl_2 while we do solid phase using a diamond anvil ATR), our local data set is important for comparative purposes. Differences in the ν_{CO} values of up to ±30 cm^{-1} were noted for spectra of compounds obtained in different phases.

Students were provided with an experimental section (previously written by students but slightly edited) and lists of data (mostly unassigned) in addition to the raw spectra (as PDF files).[25] Students assigned the peaks in the data and wrote up an evaluative summary explaining the effect of arene ligand on ν_{CO} values. In addition, there was a "discovery" component to the laboratory. The initial complexes are formed according to the following reaction (eq 1) which is given in the lab manual:

$$[M(CO)_6] + \text{arene} \rightarrow [M(\eta^6\text{-arene})(CO_3)] + 3CO \quad (1)$$

The students were then asked to predict the outcome of the following reaction (eq 2):

$$[M(\eta^6\text{-arene})(CO_3)] + P(OR)_3 \rightarrow ? \quad (2)$$

The manual was written to suggest that the phosphite ligand could replace either the arene or the three remaining carbonyls. They were then given data (^{1}H NMR, ^{31}P{^{1}H} NMR, IR, APCI-MS) and the students had to determine the product and provide their reasoning.

HANDLING AIR-SENSITIVE REAGENTS

A major experimental technique in the inorganic laboratory is the introduction to handling air-sensitive materials using specialized techniques, such as Schlenk techniques, glove bags and glove boxes, and the use of solvent stills or solvent purification systems. To provide students with at least some exposure to the concepts behind these techniques, a guided inquiry worksheet was uploaded to VIPEr.[26] While going through the worksheet, which contains links to several short web-based resources, students answer questions to show they understand the principles of an air-free technique (drying and degassing solvents, transferring solids and liquids). This learning object could stand alone or be used in coordination with the other virtual laboratories. In addition, a video was made available as a comment on this LO that shows students the proper setup for the manganese carbonyl experiment (*vide infra*).[27] This video was originally recorded for local use during the pandemic but was added to the LO as a comment for broader use in the community.

UPDATED LABORATORIES

In addition to the new LOs that were prepared, two existing experiments were quickly updated with additional data or details making them appropriate for implementation as a virtual experiment.

Manganese Carbonyl and DFT Addition

This laboratory is an extension of a laboratory published in this journal in 1988 in which students initially synthesize *fac*-$[MnBr(CO)_3(dppm)]$ (eq 3) and then prepare two different isomers of $[MnBr(CO)_2(dppm)(P(OPh)_3)]$ (**1**) from it (eqs 4 and 5).[28,29] I (A.J.) had been collecting student data from a modified version of this lab where both the bidentate phosphine and the monodentate phosphite were changed.[30,31]

$$[MnBr(CO)_5] + \text{dppm} \xrightarrow{\text{toluene}} [MnBr(CO)_3(\text{dppm})] \quad (3)$$

$$[MnBr(CO)_3] + (\text{dppm}) \xrightarrow{\text{toluene}} \textit{cis-mer-}\mathbf{1} \quad (4)$$

$$\textit{cis-mer-}\mathbf{1} \xrightarrow[\mathbf{2}.H_2NNH_2]{\mathbf{1}.[NO][BF_4],CH_2Cl_2} \textit{trans-}\mathbf{1} \quad (5)$$

This extension of the original lab adds depth and allows students to see a trend in ν_{CO} values versus electron donor properties of the phosphines, similar to the arene tricarbonyl experiment discussed previously. In response to the pandemic, modifications to the previously deployed extension included the removal of stereochemical descriptors. This required the students to not only determine how the reaction unfolded, but also the stereochemistry of each product. DFT calculations for one series of complexes were also provided.[32] This allowed students to interpret the results of modern calculations instead of relying on the simpler EHMO calculations provided in the original experiment.[28]

Evans Method

This LO is an in-class activity highlighting the use of NMR spectroscopy to measure magnetic behavior (magnetic susceptibility, μ_{eff}, and number of unpaired electrons) in the classic laboratory experiment for which students prepare a series of metal acetylacetonate (acac) complexes.[33,34] In this experiment, Cr, Mn, Fe, Co, and Cu starting materials are converted into the corresponding $[M(acac)_n]$ ($n = 2$ or 3) complexes. For the in-class activity, Evans method NMR data is provided so that students can carry out the calculations to determine the number of unpaired electrons for each of the complexes.[35]

UNFORSEEN PROBLEMS

The authors of the LOs described in this article had to deal with several issues while attempting to present potential solutions to a sudden requirement of conducting laboratory exercise online. One issue was caused by the rushed preparation of materials. We did not have the ability, in most cases, to double check data, reprepare samples, or recollect data. For the Job's method experiment, the initial data were collected in a rush to try to beat the shutdown of the state of Pennsylvania. In subsequent days, the community reports of problems with the data from two beta testers arrived. Fortunately, the governor extended the time period before the state shutdown began, so an entirely new data set was collected. The new data were obtained using a solution containing fresh ethylenediamine and gave results that matched what was expected.

For the manganese carbonyl experiment, a major learning goal was for students to determine the stereochemistry of the reaction and assign geometrical prefixes (*cis*-, *trans*-, *fac*-, *mer*-) to their complexes. While the descriptors were removed from the experimental section provided to the students, some of the student data had the descriptors in the title or comments fields.

In both the Cr/Mo arene and the Mn virtual laboratories, the authors considered providing a table of data and having the students interpret that. But in addition to the problems with this as already discussed in terms of sample preparation (solid vs solution IR data), part of the difficulty in interpreting data is determining what data are important. Thus, the IR spectra were not labeled, and the students had to select which peaks were the CO stretches. Similarly, in most cases the students had to assign NMR chemical shifts from the raw data, removing solvent or impurity peaks.

The DFT calculations for the manganese experiment reported two CO stretches for the *trans*- isomers that under idealized symmetry should result in only one peak in the IR (eq 5). After several students had completed the laboratory, explaining that their predicted IR stretches did not match what was provided, the calculations were rerun. The symmetric stretch was found to have approximately 2% the intensity of the asymmetric stretch. Interestingly, a peak in the student data was then found that matched this prediction. This was also true for the *trans*- cation synthesized during the experiment (eq

5). Several typos in the provided materials were found by careful students and the LOs had to be updated to correct them.

For all experiments, real data, especially real student data, are noisy. Often the materials are not purified or dried well leaving solvent peaks in the NMR spectra. Finally, the relative lack of student access to literature sources caused a reduction in the importance of referencing literature data in comparison to the provided data.

CLOSING THOUGHTS

Our goal in developing these laboratories was quick delivery of the materials for use by the community. At that moment in time there was little consideration of the best practices for successfully developing materials for distance education.[36−38] Many instructors faced the need to transition to virtual laboratories in very short periods of time, and thus our motivation was rapid deployment rather than considering best practices.[9]

We have little data beyond anecdotal evidence to support the positive impact of these efforts on the community. This may be due to the fact that most users of the VIPEr site were frantically developing their own virtual courses during this time. However, at the time of writing of this manuscript, the collection[8] alone has 360 views which is well above average for an LO within the first few months of being posted on the site.

Other groups that have an online community of practice, such as the OrganicERs,[39] and the Analytical Sciences Digital Library,[40] or even broader communities such as the POGIL group,[41] or the "Strategies for teaching chemistry online" Facebook group,[42] should be able to leverage their presence to become a platform for delivering virtual content. Having a disciplinary community already extant made the rapid transition to online laboratories a lot easier than it could have been. For Inorganic Chemistry, IONiC VIPEr continues to be a vibrant community of LOs and discussion, though most forum related activities have recently moved to a Discord server.[43] Having the community was essential for this rapid rollout. Many of us already knew about the site and trusted each other to provide quality content. New people actively engaged the community as evidenced by daily increases in the number of participants on the Discord server. We hope that they were able to receive the help that they needed and appreciated their insights into the challenges we all faced. As we look ahead to the Fall semester, we are already in conversation about adapting some of the experiments on VIPEr to first-year laboratory experiences, including more adapted laboratories for virtual instruction should that become necessary. We are also considering best practices for remote instruction.

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Notes

The authors declare no competing financial interest.

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