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Incorporating Sustainability and Life Cycle Assessment into First-Year Inorganic Chemistry Major Laboratories

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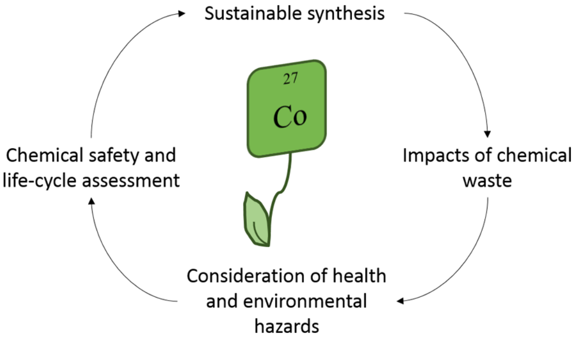
Ⓢ *Supporting Information*

ABSTRACT: Although much of the scientific community concerns itself with ideas of a sustainable future, very little of this interest and motivation has reached the classroom experience of the average chemistry major, and therefore, it is imperative to expose students to these ideas early in their careers. The focus of most undergraduate chemistry curricula rests on the preparation of the next generation of researchers, ensuring that students are capable and effective in the laboratory. A majority of laboratory experiences focus on building basic technical skill sets for chemists. However, little time is spent ensuring that students are aware of the impacts of their research, as it pertains to chemical waste and the sustainability of research, in general. At Villanova, an existing first-year undergraduate inorganic chemistry laboratory course was modified to promote novel ideas in research with an emphasis on life-cycle thinking and analysis in terms of sustainability. Initial results are reported, as well as an outline of the novel aspects of the course.



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"What happens to waste after it leaves a laboratory?" This question is one that many highly qualified researchers would struggle to answer and is an effective starting point for first-year undergraduate chemistry majors to contemplate life-cycle thinking and impact of the field on which they are just embarking. Although students did not explore a chemical's full "life cycle," disregarding difficulties in synthesizing the purchased chemicals, students did engage with what happened to chemicals placed in hazardous waste containers in a teaching laboratory. Students were allowed to focus either on personal health (carcinogens, mutagens, corrosives, etc.) or on the environmental difficulties of disposing of chemicals, such as hazards to aqueous life.

Numerous resources have concluded that there is a real need to emphasize green chemistry ideas in an undergraduate curriculum.[1] Unfortunately, there is a startling list of limitations to doing this. Many educators feel that including ideas of sustainability will take away from core content that must be covered, whereas others feel inadequately supplied with the resources available to encourage such thinking in a research-driven curriculum. Some educational innovations have recently been made in organic chemistry laboratory curricula that have married the need for content learning with a push for research potential, using sustainability as a framework.[2] This type of innovation is one that was recently applied at Villanova University in an introductory first-year inorganic chemistry laboratory course for incoming chemistry majors. The challenges differ from those of the organic chemistry laboratory, largely owing to the experience level of students, as well as the subject matter. A successful application of the interplay of research-minded problem solving with ideas of sustainability in the context of introductory inorganic chemistry is illustrated herein. Preliminary questioning of students showed that sustainability within a laboratory is an area in which students have a great deal of interest but little incoming knowledge, making it an ideal point of discussion.

In the Fall of 2014, a group of 15 first-year undergraduate chemistry majors was introduced to a laboratory experience in Inorganic Chemistry Laboratory I, focused on personal safety and hazard awareness in the context of coordination compound chemistry. In previous years, students were given prescribed instructions for how to proceed through all experiments until a final "mini-research challenge" where students performed an assigned coordination compound preparation without knowing what they were synthesizing.[3] Students synthesized the product, hypothesized the identity of the coordination compound synthesized, and used various characterization methods to support their hypothesis. Because these students lacked laboratory experience coming in, it was the duty of the instructional faculty to ensure that students were fully equipped

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to pose reasonable conclusions to their experiments, whose products were, in actuality, well-characterized Werner complexes.[4] The project culminated in a poster session where students presented and discussed their results with faculty and other students. Although the process was successful in stimulating critical thinking and introducing students to early successes in Nobel Prize history in inorganic chemistry, the curriculum has since been improved to allow students to design their own syntheses based on published ones and support their findings, in the context of sustainable chemistry.

■ LECTURE SUPPORT FOR LABORATORY CONCEPTS

The Inorganic Chemistry I lecture is taught in concert with the Inorganic Chemistry I laboratory; however, the courses are kept separate from a grading standpoint. The goal of the lecture is to provide a foundation for understanding many practical applications in inorganic chemistry. The course is divided into three major topics: atomic structure, covalent molecular substances, and coordination chemistry. A major component of the course is dedicated to spectroscopy, and these topics are reinforced in the laboratory course where students use instrumentation including UV/visible, infrared, and atomic absorption spectrophotometers. Spectroscopy is introduced early in the lecture course including a discussion of light interacting with matter and the wide array of energy units (including how to convert between them) to provide students with a solid foundation moving forward. Specific instrumentation is introduced appropriately in context with the course material. For example, infrared spectroscopy is taught during the covalent molecular substances section of the course, immediately after students have been introduced to bonding and Lewis structures. This topic aligns with the laboratory experiment, where students synthesize alum, $KAl(SO_4)_2 \cdot 12H_2O$, from aluminum cans and then analyze their compound by infrared spectroscopy, observing sulfate and water vibrational modes. In cases where the course material does not directly align with the laboratory experiment, the course offers "Laboratory Digressions" where the topic of the laboratory is discussed in detail before the experiment is carried out.

The final laboratory project (the subject of this manuscript) aligns directly with the third section of the course, coordination chemistry. During this section of the course, students are introduced to Alfred Werner, Lewis acid/base chemistry, and the coordinate bond. Topics within coordination chemistry include: hard/soft acid base theory, electron counting in transition metal complexes, nomenclature, isomerism, crystal field theory, and UV/visible spectroscopy. Students are learning this material as they are carrying out synthesis and characterization of their unknown compounds in the laboratory. They are expected to utilize this new information in context, along with other topics they have learned earlier in the course (infrared spectroscopy, atomic absorption spectroscopy, VSEPR theory, symmetry and point groups, and more) to identify their coordination compound. The final project allows students to put all of the pieces that they have learned throughout the semester into a cohesive project and reinforce these inorganic chemistry topics by putting them in context with a simulated research experience.

■ OVERVIEW OF CURRICULAR CHANGES

In devising changes to the curriculum, three major components of the course were introduced to prepare students to discuss their results meaningfully in the final portion of the mini-research challenge. A syllabus may be found in the Supporting Information.

The first component introduced students to safety in the laboratory and life-cycle thinking on the first day of laboratory. Students learned how to access chemical information through the university's database, ChemWatch, and personal safety vocabulary in the context of safety data sheets (SDSs) for chemicals students would actually be using. In addition to traversing the language of chemical safety through examples, students also learned what happens to waste when it leaves the laboratory, contributing to their knowledge of a chemical's life-cycle. Through this exercise, students knew why it was important to treat chemical waste carefully, as well as what types of chemicals could be mixed with others and why. The carboy in the waste hood was no longer a "black box" where chemicals were simply discarded without further thought. Rather, students could envision the environmental impact of different classes of chemicals. This information was later used to inform their suggestions for improvements to existing syntheses.

After this introductory presentation, five of the eight students in the class who normally wore contact lenses for every-day work made the conscious decision to wear glasses to lab because they became more aware of the hazards of the chemicals. This was in stark contrast to previous years when students listened to the recommendation not to wear contact lenses to lab but chose to wear them, anyway. Although an unintended consequence, the enhanced focus on personal and environmental safety in the introductory class undoubtedly helped to develop a culture of safety in the laboratory.

The second significant component was a change to students' ongoing laboratory report assignments. In the first four experiments, students followed a "recipe" as they might in other introductory laboratory courses, but the techniques and instrumentation utilized were more advanced, to prepare and encourage students to begin research early in their academic careers. Various synthetic methods were introduced and studied using instrumentation such as UV/visible and infrared spectrophotometers. Students wrote their reports in the style of the ACS journal *Inorganic Chemistry* and additionally were required to include a "Sustainability" section to their report. A guide to writing laboratory reports was introduced and is included in the Supporting Information. In the Sustainability section, students identified one reagent or technique that could be improved in order to make the experiment more sustainable. Students used SDSs for reagents and explained why their suggestion was an environmental improvement over the published procedure they used. The most common suggestions were to use more environmentally friendly solvents or to change the metal in a compound formed in an effort to synthesize less toxic products. Time was devoted during prelab for discussions on why it might be reasonable to make the suggested substitutions on a chemical level. For example, a change from one solvent to another solvent might be suggested where a similarity in polarities and physical properties would be singled out as a reason for the chemical compatibility of the change. Students wrote five laboratory reports in this fashion, so they became well versed with writing and thinking scientifically before the mini-research challenge.

In the third component, students made a brief proposal to alter an existing literature procedure for a Werner complex, whose identity they did not know. Each student group of two was assigned a different synthetic procedure a month before the project began without being given the literature reference. Students proposed a change to one step in the outlined procedure that would decrease the biological or environmental impact of the synthesis. Students were not required to synthesize the same product as the literature procedure but, rather, had to justify why their change was chemically compatible with the literature procedure. Chemicals were obtained for students to attempt their novel syntheses. A report and a poster presentation were given to compare the literature product and the "sustainable" product using methods of characterization discussed throughout the semester. Some examples (Table 1) of student suggestions were to change the halide counterion, attempt to coordinate a different nitrogen-containing ligand, use a different solvent, or try a different metal.

Table 1. Student-Devised Changes to Literature Procedures for Mini-Research Project

Change in Procedure	Original Procedure	Sustainable Procedure
Metal center	Cobalt	Iron Copper
Ligand	Ethylenediamine	Diethylenetriamine 1,10-phenanthroline
Counter ion in starting material	Co(II) chloride hexahydrate	Co(II) bromide hexahydrate
Product wash material	Diethyl ether	Ethyl acetate

Students proposed sustainable changes to the original laboratory procedures based on safety information derived from SDSs[5] (Table 2). For the original procedures, all students used cobalt(II) chloride hexahydrate ($CoCl_2{\cdot}6H_2O$) as their starting material. For the sustainable procedures, two groups of students chose to use iron(II) chloride tetrahydrate ($FeCl_2{\cdot}4H_2O$) as an alternative complex with a different metal center. The iron and cobalt complexes possessed similar health hazards, but $FeCl_2{\cdot}4H_2O$ does not possess toxicity to aquatic life, whereas $CoCl_2{\cdot}6H_2O$ remains very toxic to aquatic life with long-lasting effects. A second change involved a modification of the ligand coordinated to the metal center. One of the original preparations used ethylenediamine (en), which is moderately flammable, highly hazardous to human health, and harmful to aquatic life with long-lasting effects. In an attempt to make the procedure more sustainable, one group chose to use diethylenetriamine as the ligand. As amines, diethylenetriamine possesses similar health hazards to ethylenediamine, but has much less effect on aquatic life; it remains harmful to aquatic life with mild toxicity. A third change involved the counterions associated with the starting material, $CoCl_2{\cdot}6H_2O$. One group chose to use cobalt(II) bromide ($CoBr_2$) with bromide counterions. Overall, $CoCl_2{\cdot}6H_2O$ is highly hazardous to human health, indicating extreme danger, whereas $CoBr_2$ possesses more moderate health hazards. In addition, $CoCl_2{\cdot}6H_2O$ is very toxic to aquatic life while $CoBr_2$ remains harmful to aquatic life with no long-lasting effects. A final change involved the replacement of diethyl ether with ethyl acetate used to wash the synthesized product. Ethyl acetate remains the more sustainable option because it poses no hazards to aquatic life, whereas diethyl ether remains more environmentally hazardous. Overall, students based their sustainable changes on health and environmental hazards associated with materials used in the original procedures. By changing one of the materials in the original procedures to a less hazardous chemical with similar properties, students performed a more sustainable experiment under similar reaction conditions. All chemicals were recycled appropriately according to standard protocol in place at the University.

■ SAMPLE PROJECT—A GLIMPSE INTO THE PROCESS

A group of two students was assigned the literature preparation for *trans*-dichlorobisethylenediamine cobalt(III) chloride (*trans*-$[Co(en)_2(Cl)_2][Cl]$).[3g] These students were unaware this was their target at the outset. This group opted to replace en with 1,10-phenanthroline (phen). According to the SDS information, phen is toxic if swallowed and an irritant, whereas en is acutely toxic and is considered hazardous by OSHA, carrying many more warnings than phen. It is important to note that students were asked to choose one characteristic to be less hazardous, even if other effects of the chemical could be perceived as more hazardous. The idea behind this distinction was for first-year students to think about ideas of hazards and safety in a qualitative way before doing chemistry, so as not to overwhelm their understanding so early in their college careers.

Once the change was approved, the group had 1 week to incorporate the change into a modified procedure. During the first of four, 4-h laboratory sessions, this group executed the published procedure to make *trans*-$[Co(en)_2(Cl)_2][Cl]$. During

Table 2. Known Hazards of Reagents Used in Syntheses for Mini-Research Projects

Original Procedure	Health and Environmental Hazards		Sustainable Procedure
$CoCl_2{\cdot}6H_2O$	Harmful if swallowed Causes severe skin burns/eye damage May cause cancer May cause respiratory irritation Very toxic to aquatic life with long-lasting effects	Corrosive Causes burns Harmful in contact with skin Harmful if inhaled or swallowed Causes skin burns/eye damage	$FeCl_2{\cdot}4H_2O$
Ethylenediamine	Harmful if swallowed/in contact with skin Causes severe skin burns/eye damage Flammable Harmful to aquatic life with long-lasting effects	Harmful if swallowed/in contact with skin Causes severe skin burns/eye damage Flammable Harmful to aquatic life with low toxicity	Diethylenetriamine
$CoCl_2{\cdot}6H_2O$	Harmful if swallowed Causes severe skin burns/eye damage May cause cancer May cause respiratory irritation Very toxic to aquatic life with long-lasting effects	Causes skin/eye irritation May cause respiratory irritation May cause cancer Harmful to aquatic life	$CoBr_2$
Diethyl ether	Extremely flammable liquid/vapor Harmful if swallowed May cause drowsiness/dizziness Harmful to aquatic life	Highly flammable Causes serious eye irritation May cause drowsiness/dizziness	Ethyl acetate

the second session, the group synthesized their "less hazardous" product, *trans*-dichlorobisphenanthroline cobalt(III) chloride (*trans*-$[Co(phen)_2(Cl)_2][Cl]$) according to their modified procedure. In the remaining two laboratory sessions, the group characterized and interpreted UV/vis and IR spectra of their compounds with the assistance of a laboratory instructor and teaching assistant. The identities of their products were determined using atomic absorption and gravimetric analysis to determine percent cobalt and percent chlorine, respectively; magnetic susceptibility to find the number of unpaired electrons in the complex; and solubility and conductivity measurements to assess the chemical properties of their compounds. This group was successful in growing crystals of *trans*-$[Co(en)_2(Cl)_2][Cl]$ that were characterized by X-ray crystal diffraction.

After interpreting the data on their own, the group met with their instructors to strengthen their understanding of the results. The students came to the discussion with their perceptions of the interpretations of the results. The instructors coached the students through any results that were inconsistent with their conclusions on the identity of the compounds made and also assisted the students in seeing the positive correlations between characterizations and the proposed structures of the products made. The group found that their percent cobalt for the ethylenediamine complex (16.5%) was within a percentage of a theoretical value (17.4%). For their modified phen procedure, however, the percent cobalt was 11.2% against a theoretical value of 17.1%. Because the % Co obtained was different from a calculated theoretical percent based on complete reaction, the result may indicate that there may be impurities (side products), or the product contained a different counterion than was postulated. For example, using $CoCl_4^-$, a counterion observed in previous years, instead of chloride, the theoretical % Co value was closer to the actual value.

This process of critically thinking about each individual result and finding out how each piece fit into the big picture was effectively done by encouraging students not to dismiss a result simply because it did not agree with what they thought it should be. The data opened new doors to possibilities that students may not have considered if all the results had been ostensibly in agreement. The process of guiding students through so much data and interpretation demands a small class size, as it requires a great deal of resources from instructors and ready access to laboratory equipment for each group.

Finally, students reported on the published procedure, as well as the modified procedure. To evaluate student writing, each student in a group wrote a report. Partners worked together to generate posters to present at a poster presentation in the final laboratory session as a final exam. Students interpreted their data and answered questions about the procedures and the results, much as they would be required to do in the context of chemical research.

■ ASSESSMENT OF SUCCESS IN ACHIEVING GREENER CHEMISTRY—STUDENT FEEDBACK

At the beginning of the semester before exposure to chemical safety and life-cycle thinking, students anonymously identified their level of knowledge of, as well as their attitudes toward, several aspects of sustainability ideas and chemistry (Table 3). During the final evaluation of the course, students rated the same criteria.

In terms of attitudes, it was evident that attitudes of the students coming in were high, indicating a thirst for an understanding of concepts of sustainability, demonstrating the need for incorporating these topics into laboratory courses. The scores for attitudes either stayed the same or rose slightly over the course of the semester, which indicated the learning students obtained was successful in keeping students engaged in topics of sustainability even throughout the demanding coursework.

Table 3. Comparative Survey Results of First-Year Chemistry Majors Exposed to Sustainability-Based, Problem-Solving Curriculum[a]

	Beginning of Semester Average Score and SD (*N* = 17 students)	End of Semester Average Score and SD (*N* = 15 students)
	Knowledge[b]	
Personal safety in the lab	4.24 ± 0.15	4.73 ± 0.12
Effects of human-generated chemical waste on the earth	3.18 ± 0.22	4.27 ± 0.19
Chemical waste designations	3.18 ± 0.20	4.00 ± 0.20
What happens to chemicals when leave lab	2.11 ± 0.19	3.53 ± 0.22
Ways to make chemical syntheses greener	1.64 ± 0.18	4.47 ± 0.21
	Attitudes[c]	
Personal safety in the lab	4.59 ± 0.18	4.60 ± 0.28
Effects of human-generated chemical waste on the earth	4.53 ± 0.13	4.67 ± 0.13
Chemical waste designations	4.24 ± 0.19	4.27 ± 0.21
What happens to chemicals when leave lab	4.12 ± 0.23	4.33 ± 0.22
Ways to make chemical syntheses greener	4.47 ± 0.22	4.60 ± 0.17

[a] Survey result obtained with approval with exempt status by the IRB. More detailed results may be found in Supporting Information [b] Based on: 1, no knowledge; 2, a little knowledge; 3, some knowledge; 4, a good amount of knowledge; 5, expert knowledge. [c] Based on: 1, no concern; 2, a little concern; 3, some concern; 4, a good amount of concern; 5, greatly concerned.

In terms of knowledge, the survey results are highly indicative of pedagogical success in that students feel they have more knowledge of sustainability concepts in the chemical laboratory while simultaneously showing an intense desire for learning this type of information, as evidenced by the high scores for level of concern and interest in the topics. Because a majority of students at Villanova University, who are declared chemistry majors in their first year, have had advanced-level chemistry in high school, it was not surprising that students had already had some training and awareness of personal safety in the laboratory. The choice of a high percentage (62%) of the students who opted not to wear contact lenses in the laboratory indicated that these attitudes were affected possibly more than students were aware.

The knowledge category had large increases universally, with all areas except personal safety showing a marked increase over the course of the semester, well beyond one standard deviation in some cases. This result demonstrated the success of emphasizing ideas of green chemistry and sustainability into the curriculum. Much of this was achieved by the inclusion of a sustainability section in student laboratory reports, in addition to discussing issues of waste. The greatest point increase in the knowledge category was a 273% increase in the category of "ways to make chemical syntheses greener." The impact of this was astounding, as the average first-year chemistry student has likely never thought about this and has likely not had an opportunity to explore this topic. Students effectively performed novel undergraduate research in their mini-research challenge using only the parameters of a previously existing literature procedure, as well as a need to make the synthesis greener. Students independently proposed their own changes to the syntheses and then implemented the changes and observed how their modifications affected the products. If the job of a chemistry major's undergraduate experience is to train the next generation of researchers, then this was an ideal example of students engaging in research during their first laboratory experience in college with low stakes and ample guidance, but with enough freedom to explore—a tough balance to strike.

LIMITATIONS

Two clear limitations are present in this curricular adaptation, and the analysis of the results and impact. First, the sample size of students was quite small, 15 students at the conclusion of the course. A relatively small population of students at Villanova University are chemistry majors, and thus, the impact of these changes cannot be felt by many students. The benefit to this was that these students are in a unique position to tackle issues of sustainability in their future academic and professional lives, which singles them out vis a vis other undergraduates. Having such an individualized approach to the mini-research challenge involved planning, as well as one-on-one meetings to ensure that students were going down a reasonable path in their research, which can be difficult in a larger department.

Second, the responses gathered in the survey were self-reported. The purpose was to gauge students' perceptions of their knowledge and attitudes. Though it was clearly observed that students showed greater mastery of ideas of sustainability in their laboratory reports as the semester progressed, this was difficult to quantify. Thus, self-reporting was used as a technique in an effort to gain insight into how students perceived issues of sustainability, as it pertained to chemistry and chemical research.

CONCLUSION

On the basis of anonymous self-reporting by students, as well as general observations and modified behaviors in the laboratory, it was evident that having an introductory laboratory curriculum well rooted in sustainability concepts had far reaching consequences. Students used problem solving and critical thinking skills to propose novel, greener syntheses, akin to the process used in chemical research. Some examples of assessing critical thinking included grading laboratory reports with an eye toward elaborated data interpretation, as well as generating new ideas about more sustainable approaches to the work done in each laboratory session. Students had to think critically through numerous tests for their final project and defend their interpretations of the data. Students also gained an appreciation and knowledge of environmental issues to consider when performing chemical experiments.

Future improvements would consist of polling a larger sample size to assess a larger impact. Although the sample size was small at present, follow up work with more students in future years would allow for a greater statistical understanding of the impact of this curriculum, as well as the ability to track students into their later years to gauge impact of this first-year experience. Having a small class size is necessary to the success of this curricular approach. Spending more time with chemical designations in order to improve awareness of this personal and environmental safety issue would be an improvement to the curriculum. Though students unknowingly employed various aspects of the Twelve Principles of Green Chemistry,[6] a more conscious emphasis should be placed on familiarizing students with these early in the semester in order to encourage broader thinking on how to improve existing laboratory practices. Additionally, a focus on EcoScale[7] calculations will help tighten student understanding and attempt to do a better job of quantifying what it means for a synthesis to be greener.

Implications of this research are a need for greater focus on sustainability issues, as well as practice with SDS information for undergraduates. In an effort to promote a "culture of safety," early introduction into ideas of environmental awareness and personal protection encompasses a more global view of the chemist's responsibility for the earth. Additionally, this study clearly indicated that student outcomes were enhanced, not inhibited, by introducing a sustainability lens into the laboratory. Students demonstrated mastery of critical thinking ideas through novel synthetic schemes and successfully compared results from multiple syntheses, both from a chemical and from a sustainability standpoint, preparing students for research with an eye toward global responsibility.

ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00281.

Syllabus for the course. (PDF)

Separate survey results. (XLSX)

Outline for what each section of the laboratory report should include. (PDF)

Outline for what each section of the laboratory report should include. (DOCX)

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Notes

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