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Exploring Materials for Carbon Capture: A Project-Based Lab for the First-Year Chemistry Course

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ABSTRACT

The field of carbon capture, utilization, and storage is a popular area of research due to its connection to environmental issues and continually rising carbon dioxide (CO_2) levels. To incorporate this important topic into the undergraduate chemistry curriculum, a test apparatus for CO_2 capture was adapted for a 3-week, project-based lab designed to compare the efficacy of different capture materials. The experimental design of the apparatus is intuitive, using a gravimetric approach. It is also versatile, with the potential to test a variety of emerging materials for CO_2 capture. The apparatus used does not require any expensive equipment, making it possible to implement with limited resources or even at the high school level. The 3-week lab was piloted in second-semester general chemistry labs for both undergraduate majors and non-majors at Bridgewater State University, in Spring 2022. Instead of completing a cook-book style laboratory with an expected outcome, students worked together through class discussion, reading science news articles, and completing laboratory experiments to evaluate the efficacy and sustainability of sodium hydroxide versus deep eutectic solvents for carbon capture. Student teams were able to complete data analysis, evaluate the precision of their collected data, identify experimental limitations, and suggest ways for future students to continue the project. In the latest iteration, more emphasis was placed on science communication and oral presentation of data. Finally, surveys were developed to gather quantitative data on the impact of this authentic research project in the future.

INTRODUCTION

In the effort to increase student engagement and relevance of curricula to real-world issues, Project-Based Learning (PBL) has received considerable attention in education research.[1–4] PBL is a student-centered teaching/learning model that has students investigate a real-world problem or question through a longer-term project, with the teacher as a facilitator. Participation in PBL courses is correlated with more perceived relevance to career goals.[1] PBL can also improve student life skills and student understanding of chemical concepts. A survey study was conducted on graduate students who were studying education and enrolled in a PBL-style course. The results indicated observable improvement in students' perception of skill in responsibility, problem-solving, self-direction, communication, and creativity.[2] The PBL model has been shown to be effective in the context of science, technology, engineering and math (STEM) education, including chemistry courses. In a study of about 500 college students, it was found that students who took at least one PBL course were more likely to have career aspirations in STEM. One study on undergraduate chemistry students showed that students in a PBL course performed significantly better in final exams, post-tests, and interviews than those who were enrolled in a traditional course,[3] while another demonstrated the role of PBL in encouraging undergraduate chemistry students to exercise metacognition. Through semi-structured interviews with students, the researchers found that students were developing their planning, evaluating, reflection, and teamwork skills, and were learning to take individual responsibility.[4] PBL has also been shown to have a positive impact on marginalized students.[5,6]

Many project-based labs have been developed for higher education chemistry courses, especially on analytical chemistry and biochemistry topics[7–10], and there is ample research on the benefits of PBL as a pedagogical tool. There is also literature on semester-long PBL experiences.[11–13]

However, shorter term project-based labs for first-year general chemistry courses are currently lacking in the literature. One of the few published PBL labs of this kind was developed by Bopegedera et. al, and explores the chemistry and geology of alkaline surface lake waters in Washington state.[14] This PBL laboratory was designed as an interdisciplinary collaboration for a joint chemistry-geology course. There have been other studies on the benefits of PBL in the general chemistry classroom[15,16], but many of them do not outline a specific project that can be adapted widely in the curriculum.

One of the requirements of PBL is that it must connect to an authentic problem.[17] The present issue of climate change is one with which undergraduate students largely possess some level of familiarity. In a preliminary survey conducted on students in a first-year chemistry course at Bridgewater State University, it was found that 93% felt they could already contribute to a conversation on environmental issues, making it an ideal topic to engage students. Additionally, in a survey of 13,749 U.S adults, Pew Research Center found that out of Gen Z, Millennial, Gen X, Boomer, and older generations, Gen Z was the most concerned with addressing climate change and has engaged with the issue the most, both socially and through taking action.[18] One major issue connected to climate change is that of the perpetually rising levels of carbon dioxide (CO_2),[19] and naturally, mitigation efforts via carbon capture. Resources and time have been poured into developing better, more efficient carbon capture technologies, but the difficulty is in designing techniques that avoid doing more harm to the environment than good. Green chemistry principles must be adhered to as much as possible in the process. Leclaire and Heldebrant outline three problems that must be considered when designing carbon capturing techniques: energy efficiency, cost of manufacture, and life cycle, which includes synthesis, use, and waste.[20] This offers a dynamic, highly relevant problem with which students can interact.

Considerable efforts have been made to integrate topics of climate change to undergraduate education in many scientific disciplines, especially earth science and geology.[21] However, carbon capture and other topics on CO_2 emissions are rarely addressed in the undergraduate science laboratory. In chemistry specifically, even laboratory curricula containing broader climate change topics are lacking. To our knowledge, there are only three published papers that describe chemistry laboratory experiments on this topic as of 2022, and although valuable exercises, they are designed for upper-level courses and none of them employ PBL.[22–24] Thus, there is a need for thoughtfully designed introductory chemistry lab projects that connect students' learning to relevant, authentic research challenges like carbon capture.

To address this gap, we developed a three-week, PBL lab with a focus on carbon capture. In this research-style lab, students explore different materials for CO_2 capture, with consideration for green chemistry principles. In the final week, students prepare and give an oral presentation to their classmates summarizing their hypotheses, results, and proposed future research directions. According to the ACS guidelines for bachelor's degree programs, six overarching skills must be developed in an ACS-accredited program: problem-solving, chemical literature and information management, laboratory safety, communication, team skills, and ethics.[25] The project described here addresses all but ethics. Also in alignment with the ACS guidelines, students engage with the scientific method and perform synthesis of molecules. This PBL lab was also developed according to the Bridgewater State University chemistry program learning outcomes; in particular, that students will be able to: solve problems by applying the scientific method, communicate chemical information and scientific findings to both the general public and scientific communities, work in a team environment, develop chemical literacy skills, and

demonstrate safe laboratory practices. The specific learning objectives of this lab are outlined as follows:

- Through guiding questions and group discussion, students will be able to identify a problem, develop a research question, and state a hypothesis. Students will be able to draw conclusions from the data and reflect on the results, thinking about experiment limitations.
- Students will be able to apply their lecture-learned skills to perform gravimetric analysis, compare data, and demonstrate mastery of solution chemistry.
- Students will be able to consume and understand science media, which has been created for audiences with a general knowledge of science and is related to their own lab project.
- Students will be able to compile and communicate scientific findings to their peers through oral presentation.

EXPERIMENTAL OVERVIEW

In light of rising levels of carbon dioxide in the environment, carbon capture technologies have been developed as one way to combat climate change via mitigation of environmental CO_2, either by pulling it from the atmosphere or by catching it directly at sources of high CO_2 emissions such as burning fossil fuels ("carbon," in this case, is used synonymously with "carbon dioxide"). One subset of carbon capture techniques uses materials to absorb or adsorb CO_2, with some well-studied sorbents including sodium hydroxide (NaOH)[26–28] and amines such as ethylenediamine (EDA).[29–31] More recently, metal organic frameworks (MOFs) have received

considerable attention.[31,32] Another, novel material that shows some promise is the deep eutectic solvent (DES).[33] A DES forms by mixing two chemicals that lower each other's melting points to the eutectic point, allowing them to mix as a liquid even if they started out as two solid materials. DESs are considered green due to their energy- and atom-efficient synthesis. The synthesis produces no wasteful byproducts, it requires minimal heating, and the reagents have relatively low toxicity, in alignment with the EPA's green chemistry principles.[34] They have also been shown to be readily biodegradable, with a low cytotoxicity.[35]

Three choline chloride-based DESs were chosen as testing materials for this lab to compare to NaOH: choline chloride-urea (ChCl-Urea), choline chloride-ethylene glycol (ChCl-EG), and choline chloride-glycerol (ChCl-Glycerol). Their synthesis, based on procedures by Mulia et al., is shown in Figure 1.[33] Although NaOH is corrosive and not considered green, it is useful to examine as a well-studied conventional material and as a lesson for students to think critically about the cost vs. benefit of using a material that is effective at capturing CO_2 but is harmful to the environment.

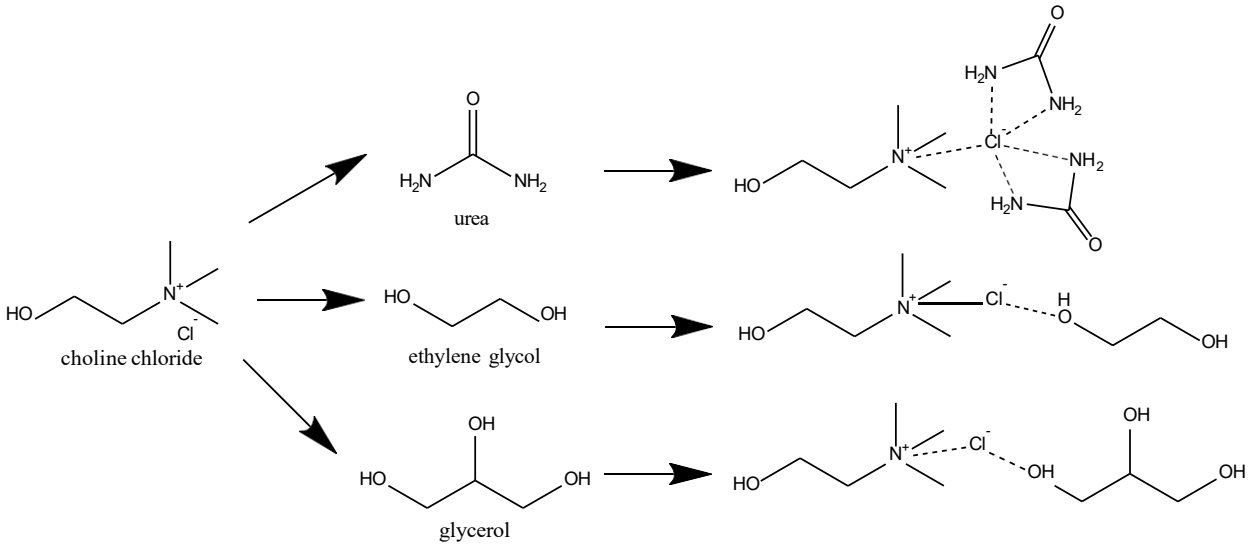

Figure 1: Synthesis of ChCl-Urea, ChCl-EG, and ChCl-Glycerol.

An image of the experimental apparatus used is shown in Figure 2, that was originally designed and developed by BSU undergraduate student Myrria Lyncee in 2019. It measures mass of CO_2 captured using a gravimetric approach. CO_2 gas is generated in a vacuum flask using citric acid injected through a syringe and sodium bicarbonate. A balloon is added to regulate pressure. The carbon capturing material of interest is placed inside the conical vial and set to stir at 200 rpm. The CO_2 gas flows through the tubing, some of it is captured, and excess CO_2 flows into a beaker of water, bubbling into the open air. The difference in mass of the test vial before and after CO_2 exposure can be used to determine the mass of CO_2 captured.

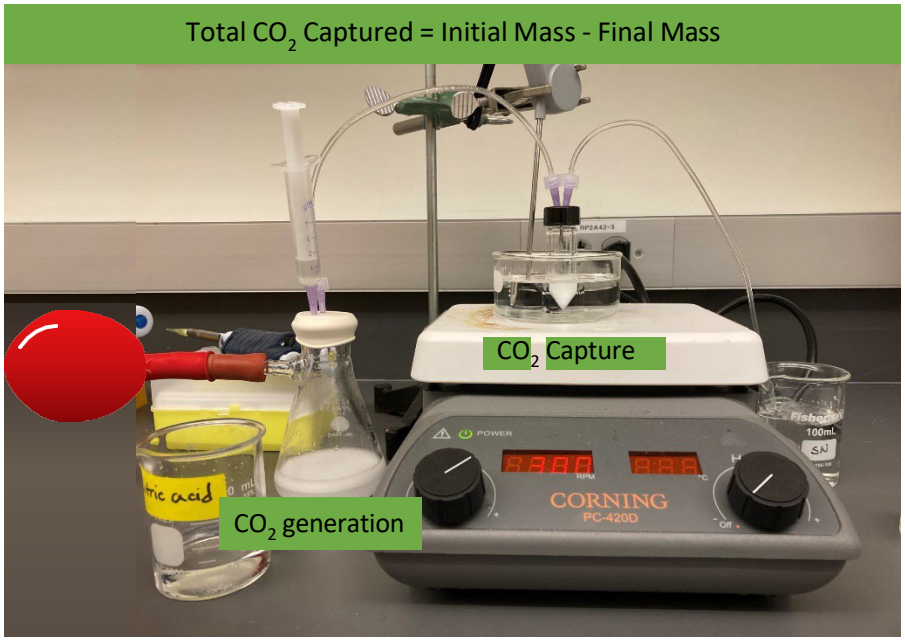

Figure 2: Carbon capture apparatus adapted from design by Maria Lyncee.

In Week 1, students use this apparatus to test 0.5 M NaOH (positive control). They are given previously obtained data on DI water (neutral control) with which to compare. They also synthesize a choline chloride DES, and each pair of students is assigned one of three types: choline chloride-urea, chlorine chloride-ethylene glycol, or choline chloride-glycerol. In Week 2, students use the apparatus to test the DES they synthesized, and then compare data across all the

materials. In Week 3, students prepare and give oral presentations to their peers. This is detailed in Table 1.

Table 1: Overview of PBL lab.

Week	Learning Activities	Skills
1	• Read *Chemical & Engineering News* article as introduction to deep eutectic solvents • Introduction to carbon capture and class discussion • Test a positive control (NaOH) • Synthesize deep eutectic solvents	• Form testable hypothesis • Exercise awareness of reagent hazards • Analyze data
2	• Learn about the Twelve Principles of Green Chemistry as an introduction to green chemistry • Class discussion and data analysis (Compare positive control to neutral DI water control) • Test deep eutectic solvents	• Analyze and compile data • Think critically about cost vs benefit with regards to materials of varying "greenness" and CO_2 capturing efficacy
3	• Prepare and give oral presentations in groups or pairs	• Oral communication

INITIAL TESTING OF CO_2 CAPTURE APPARATUS

In preparation for designing the lab, we gathered data on some well-studied materials as well as a MOF (specifically, ZIF-93 suspended in aqueous solution) and DESs. The MOFs were synthesized at room temperature using a water-based method developed by Ramos et al.[36] The DESs were synthesized using the method described above in Experimental Overview. For this data, 2 mL of each material was used, and CO_2 was slowly generated over the course of 20 minutes. The CO_2 absorption or adsorption efficacy of five of these materials is summarized in Figure 3. The apparatus was reasonably precise, with a %RSD ranging from 2%-8% for the data shown.

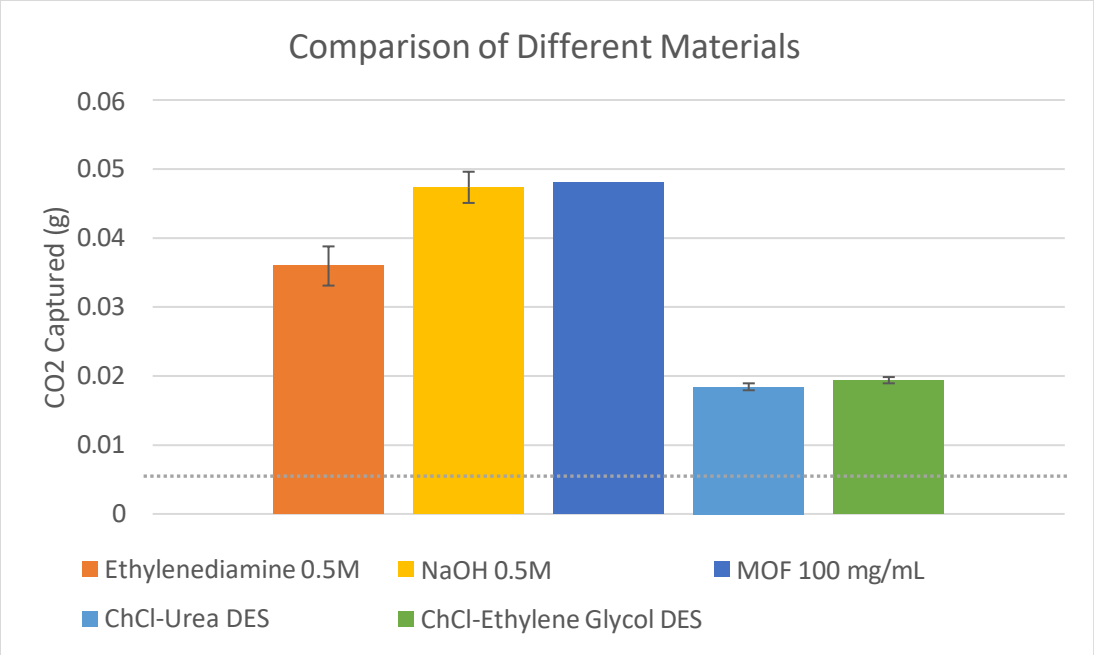

Figure 3: Amount of CO_2 captured by different materials. The gray dotted line represents the control, DI water. Error bars represent standard deviation. Data was taken for 2 mL of material after 20 min of exposure to a continual stream of CO_2 gas.

Figure 4 shows the amount of CO_2 absorbed by 2 mL of NaOH or ethylenediamine at different concentrations, all at room temperature and after 20 min of exposure to a continual stream of CO_2 gas. Ethylenediamine was chosen due to its bifunctionality (two amine groups are required to bind CO_2) and because it should theoretically be able to produce only sweet gases. NaOH was chosen due to its role as a conventional material and its straightforward CO_2 mechanism. The apparatus yielded precise data, and the error bars represent standard deviation. Carbon captured by DI water is represented by the gray dotted line.

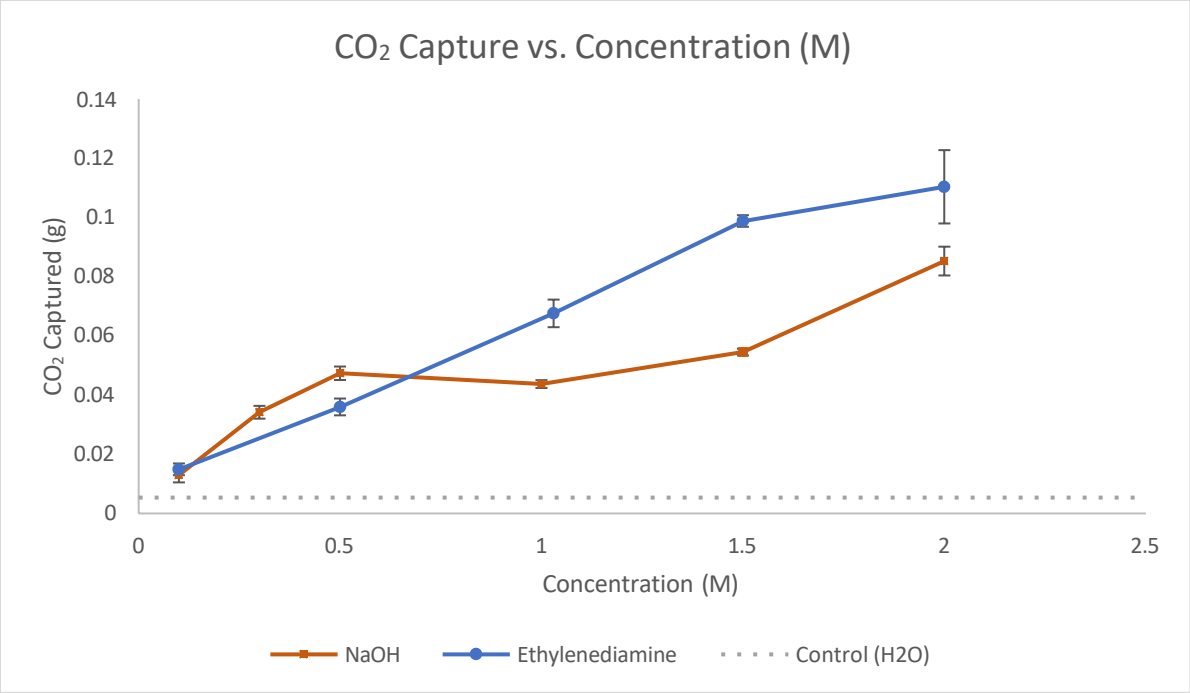

Figure 4: Amount of CO_2 absorbed by NaOH (red squares) and ethylenediamine (blue circles) at different concentrations. Data was taken for 2 mL of each material after 20 min of exposure to CO_2 gas.

Kinetics were also explored for varying temperature and concentration. It was found that at higher concentrations of EDA and NaOH, the absorption of CO_2 would generally take less time to reach a steady state, and the material had a larger absorption capacity. There is a discrepancy in that the lowest concentration of EDA reaches the steady state sooner than either 1 M or 1.5 M EDA, and 1.5 M NaOH similarly reaches the steady state sooner than 2M NaOH. This is shown in Figure 5 and Figure 7. Then, keeping concentration constant, we modulated the temperature and found that at higher temperatures in EDA and NaOH, the absorption of CO_2 would increase rapidly, reach a steady state, and then decrease once again. This data is shown in Figure 6 and Figure 8, respectively. This suggests that the absorption is reversible. However, it is possible that the decrease is due to other factors, such as evaporation of the solution, so further testing would be required to confirm this.

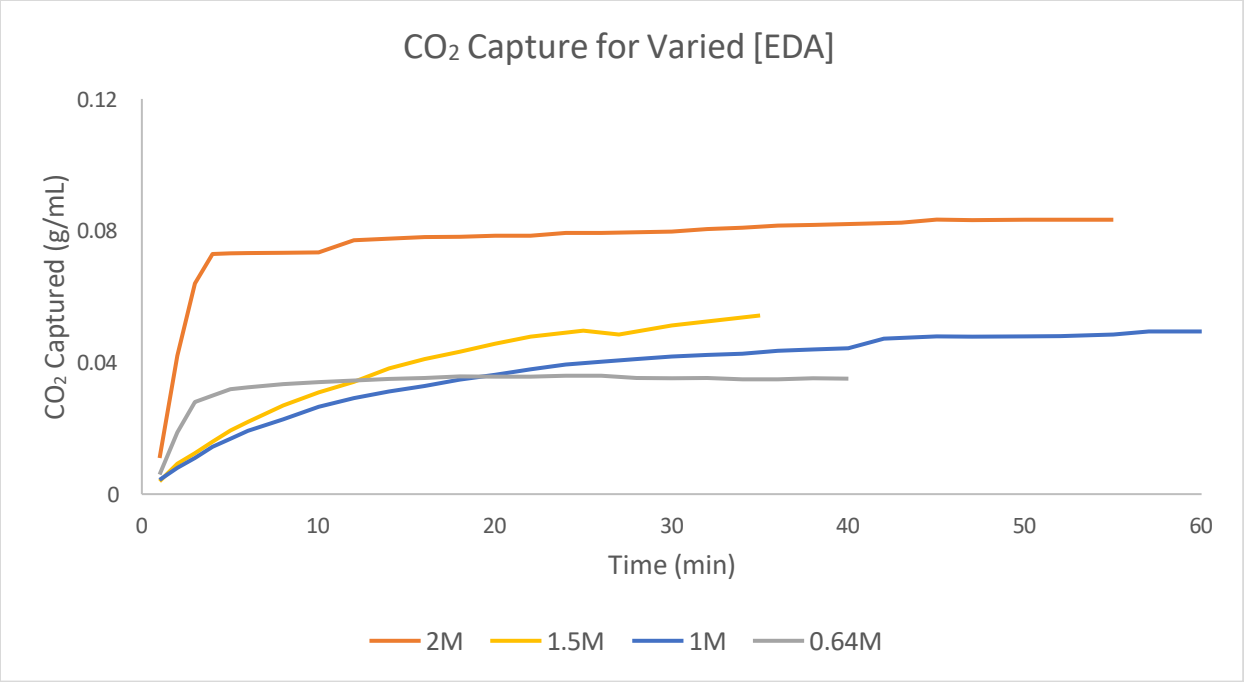

Figure 5: Kinetics of CO_2 capture by different concentrations of ethylenediamine at room temperature.

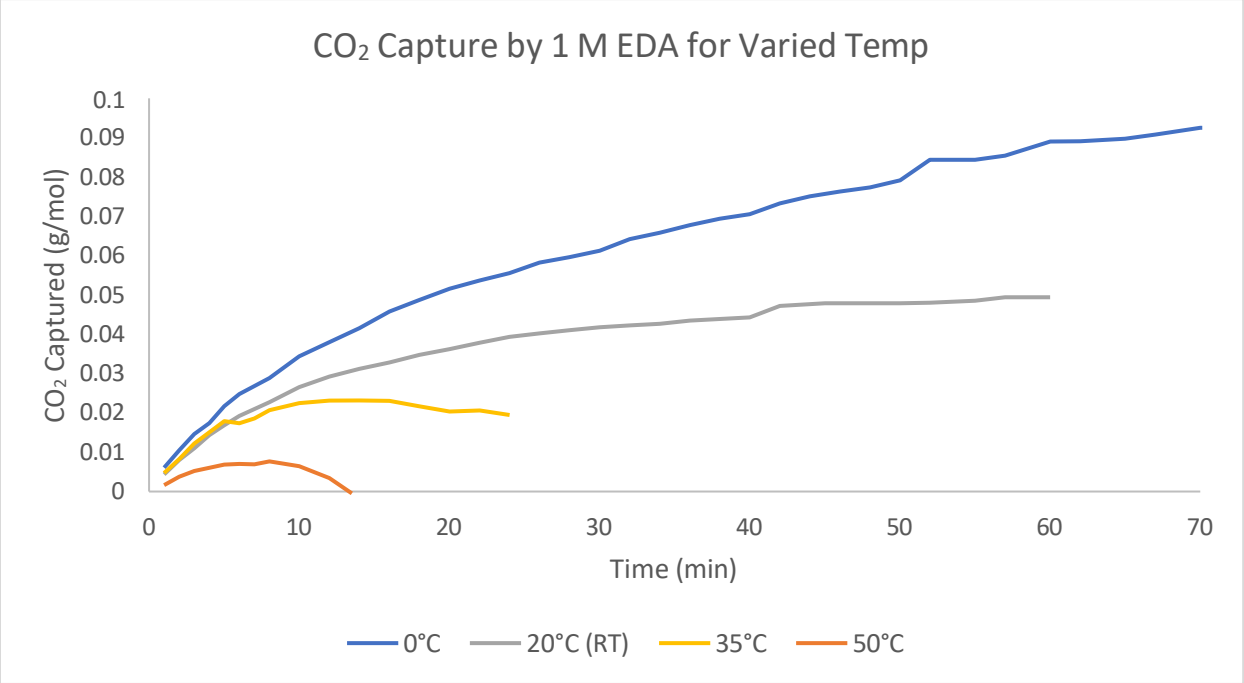

Figure 6: Kinetics of CO_2 capture by 1 M ethylenediamine at varied temperatures.

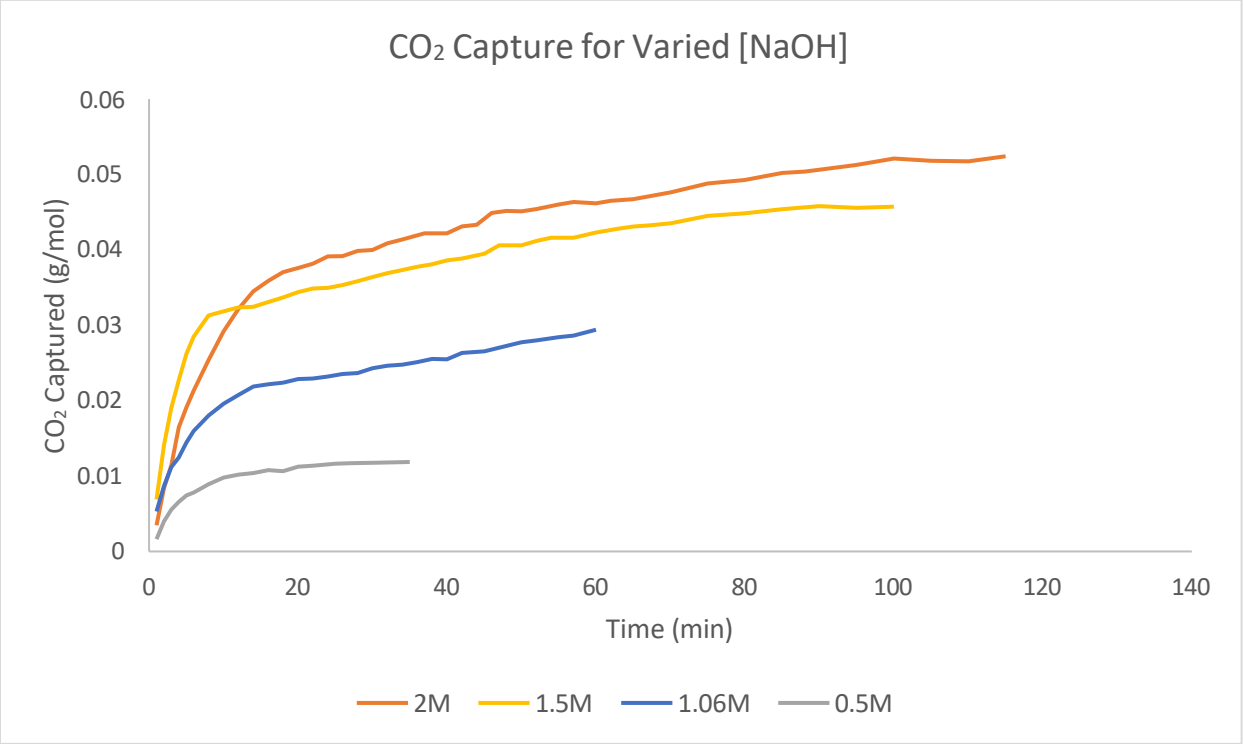

Figure 7: Kinetics of CO_2 capture by different concentrations of sodium hydroxide at room temperature.

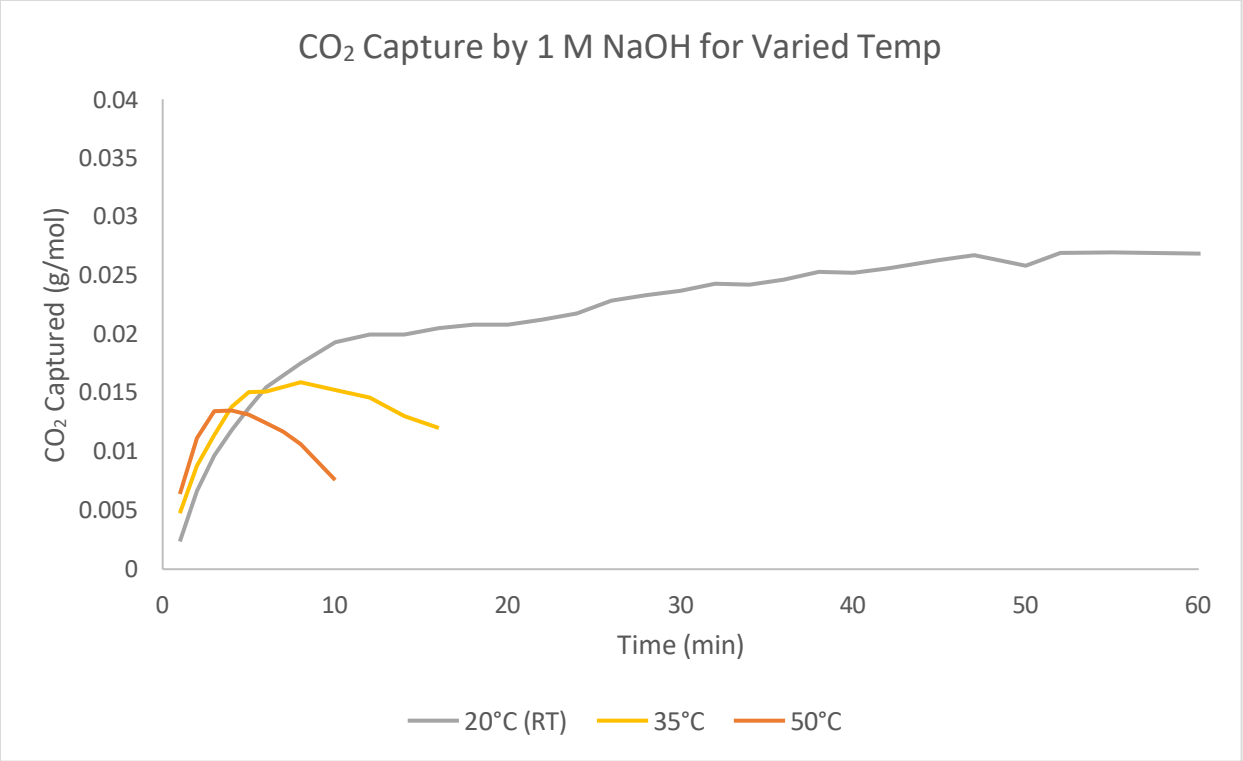

Figure 8: Kinetics of CO_2 capture by 1 M sodium hydroxide at varied temperatures.

IMPLEMENTATION AND ITERATIVE DESIGN

This study was performed as part of the second semester general chemistry curriculum (Chemical Principles II) at Bridgewater State University, a primarily undergraduate institution in Massachusetts. 126 students participated across 12 sections, taught by up to 6 different instructors (Table 2). Most students are biology majors, some in chemistry, and a few students in other majors. The changes made between iterations is summarized in Figure 9.

Table 2: History of implementation and number of student participants.

Semester	Sections	# Instructors	# Students
Fall 2021	1*	1	16
Spring 2022	9	6	95
Summer 2022	2	2	15
Total # of students			**126**

**This initial pilot session was performed in an Organic Chemistry I Lab instead of General Chemistry.*

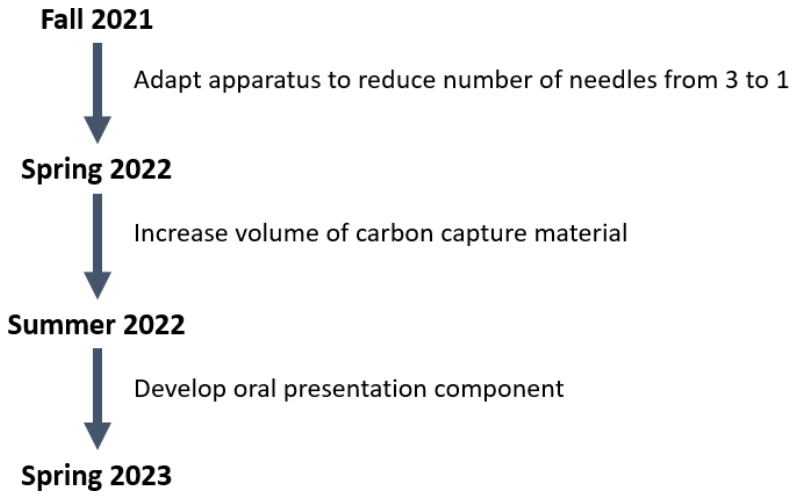

Figure 9: Summary of iterative curriculum design.

The first iteration of this PBL lab was piloted in one section of Organic Chemistry I (CHEM 243L) in Fall 2021 and followed this outline:

- Week 1 – Introduction to carbon capture. Students watch National Geographic video on climate change as homework. Students participate in group/class discussion and test a control (DI water).
- Week 2 – Introduction to green chemistry. Students read a Chemistry & Engineering News article on DESs. They participate in discussion, test a conventional carbon capturing material (NaOH), and synthesize a DES, then use information on the physical properties of the different materials to form a hypothesis about their relative efficacy.
- Week 3 – Data analysis. Students test DESs, then engage in class discussion and analyze results.

In the second iteration, which encompasses both Spring and Summer of 2022, the apparatus was modified to reduce the number of needles involved, and then the volume of testing material was increased from 2 mL to 5 mL. Instead of inserting two needles into the septum cap on the conical vial and one into the vacuum flask, the two needles in the conical vial were foregone and the tubing was inserted directly into the septum cap using a corkscrew. The basic procedure provided to students was also written more clearly, and slight modifications were made to the pre-lab discussion questions. This second iteration was administered in all sections of Chemical Principles II (CHEM 142L) in Spring 2022 and Summer 2022. Lab handouts and list of materials can be referenced in Appendix A.

As for the third iteration, which will be piloted in Spring 2023, testing the DI water control was removed, both to save frustration due to very small numbers for CO_2 mass capture data and to

make room for an oral presentation. However, the DI water data is provided to the students and is still used as a comparison against other materials. The outline is as follows:

- Week 1 – Introduction to carbon capture. Students watch National Geographic video on climate change as homework and read a Chemistry & Engineering News article on DESs. Students participate in group/class discussion and test a conventional carbon capturing material (NaOH). They also synthesize a DES, then use information on the physical properties of the different materials to form a hypothesis about their relative efficacy.
- Week 2 – Introduction to green chemistry. Students read about the principles of green chemistry and identify which principles are being addressed in this project. They participate in discussion, test DESs, then engage in class discussion and data analysis.
- Week 3 – Oral presentations. In groups or pairs, students create short presentations detailing their hypotheses, experimental results, conclusions, and a new research question for future students or researchers to explore. Near the end of the period, students will present to the class.

Lab handouts and rubric for this version can be found in Appendix B.

ASSESSMENT DESIGN

Pre- and post-surveys were designed to be given in-class to assess students' perceived understanding of green chemistry and environmental issues, as well as attitude toward chemistry courses. For big-picture understanding, students are asked about their awareness of green chemistry, climate change, and environmental issues. Students also self-assess their specific laboratory skills such as analyzing precision in data and solution chemistry. To assess students'

attitudes, they are asked about how relevant they perceived the course to be to their career goals and their historical attitude towards chemistry courses in general. Participation in this survey is voluntary, and students are allowed to skip any questions they may feel uncomfortable answering. See Appendix C for pre- and post-surveys.

FURTHER EXTENSIONS

This laboratory project has been integrated into the curriculum at Bridgewater State University and will run in CHEM 142L in Spring 2023. The surveys will be administered in-class. I will also prepare an article based on this work to submit to the *Journal of Chemical Education*.

In the future, a researcher could extend this work in one of several ways. From an educational standpoint, this PBL work could be adapted for a high school chemistry lab, as the apparatus requires no specialized equipment. This would lay a good foundation for students with science-related aspirations, and encourage those who may not have seen themselves as future chemists to consider it. It could likewise be adapted for a higher-level course such as analytical chemistry. Currently, this project addresses all the ACS learning standards except for ethics. Future iterations could incorporate a discussion of scientific integrity of results and data.

In a preliminary post-survey, students suggested research problems that future students could pursue. One proposed problem involved testing CO_2 sorption vs. time. Although the current apparatus is not robust enough to thoroughly investigate sorption rate kinetics, it could be used qualitatively to help students connect their own experimental data to kinetics concepts. A second student posited that they could add a dye to the CO_2 to visualize its capture. Dyeing the carbon capturing material instead of the CO_2 would be a feasible option, as already demonstrated in a

lab intended for an inorganic chemistry course.[22] They utilized CO_2 acidity and dyed MOFs with methyl red indicator, to measure pH change as a proxy for CO_2 adsorption.

From a chemistry standpoint, future researchers could explore other materials for carbon capture such as different MOFs, or they could study existing materials more in-depth, perhaps by modulating conditions of DESs, combining materials, or studying their absorption or adsorption kinetics. These types of research problems could also be integrated into the PBL curriculum for students to explore.

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Thesis Appendices

Appendix A – Procedure Handouts and Laboratory Preparation Materials for Iteration 2 of PBL

Appendix B – Procedure Handouts and Laboratory Preparation Materials for Iteration 3 of PBL

Appendix C – Pre- and Post-Surveys

CHEMISTRY 142 – Chemical Principles II Laboratory – Spring 2022
Lab 2: Testing Materials for CO_2 Capture
Week 1 of 3

Project Goal. In this project-based lab, you will evaluate the effectiveness of different test materials in "capturing" carbon dioxide (CO_2).

Experimental Goals and Introduction. Most people are familiar with or at least aware of current environmental issues, whether it be air pollution, excessive waste, or coastal flooding. One major aspect of these issues is the steadily increasing amount of carbon dioxide (CO_2) gas in the air.[1] CO_2 is a necessary *greenhouse gas*, keeping the earth from freezing, but there is too much of it in the air. This leads to rising global temperatures and an unstable climate. Excess CO_2 also contributes to air pollution and pH imbalance in seawater, which becomes acidic as it absorbs increasing amounts of CO_2.[2] To combat this, researchers are developing methods to remove CO_2 from the air and then convert it to a material that can be used for other purposes. "Carbon Capture Utilization and Storage" (CCUS) refers to the technology and science designed around this challenge (in this context, "Carbon" means "CO_2").[3]

In this project-based lab, you will be using a simple apparatus to generate CO_2 gas and study the efficiency of different test materials in carbon capture using *gravimetric analysis*. This project has two major objectives:

Objective 1: Evaluate the precision of the carbon capture apparatus.
Objective 2: Evaluate the relative ability of test materials to capture CO_2 gas.

You will generate CO_2 gas, expose different test materials to the CO_2 gas, and then use the change in mass of the test material to calculate how much CO_2 was captured. When doing a *quantitative analysis* of the change in mass, it is important to know the precision and reliability of your experimental setup and technique. To do this, you will run the same experiment in triplicate, and then check the precision of your data by calculating the relative standard deviation, (%RSD). Precision refers to the reproducibility of repetitive sets of data and can be expressed as %RSD. %RSD is calculated by taking the standard deviation of the data points, and dividing it by the mean value of the data points:

$$\%RSD = \frac{standard\ deviation}{mean} \times 100$$

This can be quickly determined in Excel using the AVERAGE() function to calculate the mean, and the STDEV.S() function to calculate the standard deviation. The lower the %RSD, the better the precision. A high %RSD means that the measurements varied a lot when the same experiment was repeated..

In the first week of this experiment, you will test a control material, water, to which the other materials will be compared. We expect that the water will not absorb very much CO_2 gas. The CO_2 gas dissolves in water, forming an *aqueous solution*. Aqueous CO_2 then slowly reacts with water to form carbonic acid. This is why pure water in contact with air tends to have a slightly acidic pH rather than a neutral pH of 7. This process is described by the chemical reactions below:

$$\mathbf{CO_2(g) \rightleftarrows CO_2(aq)}$$

$$\mathbf{CO_2(aq) + H_2O(l) \rightleftarrows H_2CO_3(aq)}$$

*Glossary:
Greenhouse gas – a gas that absorbs infrared radiation and prevents too much heat from leaving the atmosphere.
Eutectic point – in a mixture, the lowest possible melting point for all ratios of components.
Precision – in chemical analysis, the reproducibility of a measurement (how close replicate measurements are to each other). This is different from accuracy, which is how close a measurement is to the true value.
Quantitative analysis - a technique to determine how much of a certain component is present in a sample
Gravimetric analysis - a technique that uses the measurement of mass to make a conclusion about the chemicals present in a sample. (This is the same *gravi-* as in *gravity*.)
Project-based learning - in education, a method of teaching/learning that has students engaging in meaningful, longer-term projects that connect to the real world.
Aqueous solution – a solution where water is the solvent.

Learning Outcomes for this Lab:
After completing this project-based lab, you will be able to…

1. Identify a problem, develop a research question, and state a hypothesis.
2. Perform gravimetric analysis and prepare solutions accurately and precisely.
3. Draw conclusions from experimental data and identify potential limitations.
4. Access and comprehend scientific media designed for general audiences.

Pre-lab Discussion.
As part of the pre-lab introduction with your instructor, you will have a discussion with the other students at your lab table about the following topics:

1. With your team, come up with 3 examples of global environmental problems that chemists can help solve.
2. What is the problem that you are investigating in this project-based lab?
3. Why is this problem important to study?

Qualitative Observations. As this is a new lab experiment, you will be graded on the quality of your observations. For each step be sure to record not only what you "see", but also any challenges or questions. You will be graded on the accuracy and completeness of these observations, and you will use your observations answer post-lab questions.

Experimental Procedure: This week, you will evaluate the precision of our carbon capture apparatus by using gravimetric analysis to determine the efficiency of a water control to capture CO_2 gas. Because water is a control material that we will compare other materials to later, we do not expect the water to capture very much CO_2 gas.

Materials.

- 2-3 5-mL reaction vials
- Stiff tubing piercing rubber septa and septum cap
- 5 mL plastic syringe with needle (pink 18 gauge)
- P1000 micropipette
- 1 mid-sized spin vane
- 1 Vacuum flask (125 mL, with arm)
- 2 Beakers (100mL)
- Balloon

Carbon Capture Apparatus

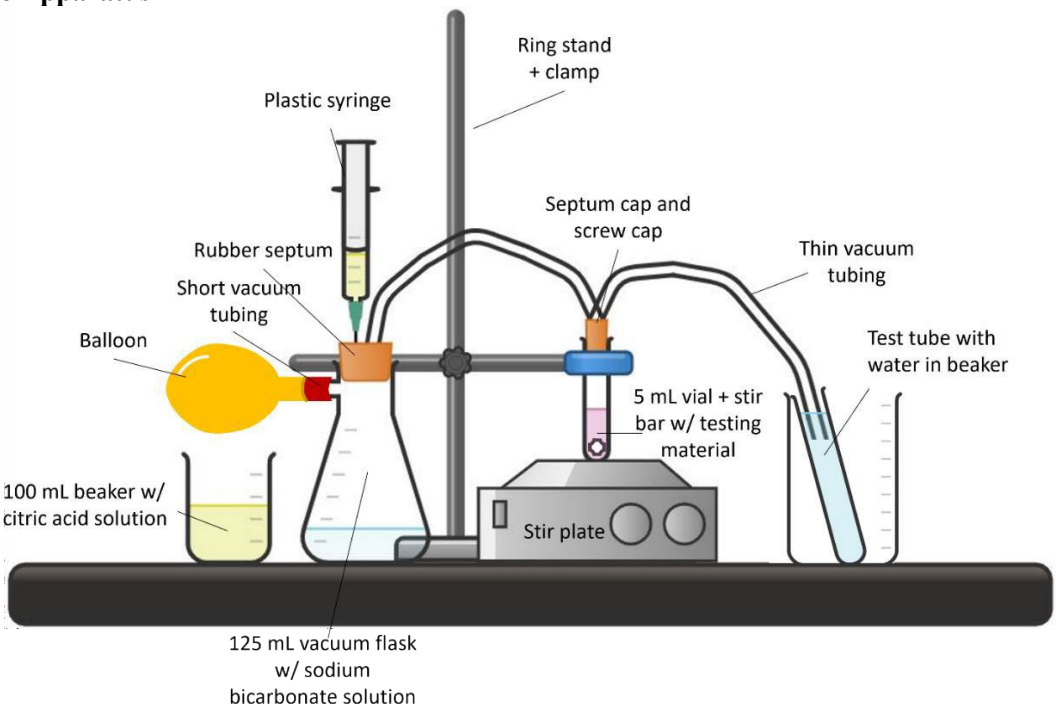


Figure 1: Carbon capture apparatus. Original design by Myrria Lyncee. Illustration made in Chemix by Elise Eng.

Week 1 Protocol

1. Add 10 g citric acid to a beaker. Using a graduated cylinder, measure out 10 mL DI water and add to the citric acid. Make sure to stir the solution with a stirring rod until it is fully dissolved.
2. Add 6.5 g sodium bicarbonate and 20 mL DI water to the 125 mL vacuum flask. Stir the solution with a stirring rod to mix, but note that the sodium bicarbonate will not fully dissolve.
3. Using Figure 1 and the example apparatus as a guide, construct the carbon capture apparatus.
4. Using a P1000 micropipette, add 2.00 mL of DI water, 1 mL at a time, to the 5-mL reaction vial. DO NOT try to pick up all 2 mL of water at once with the micropipette. This will damage the micropipette. Add the spin vane to the reaction vial. Record the initial mass of the reaction vial, water, and spin vane (with no cap on the vial) to 3 decimal places.
5. Cap your reaction vial with the septum cap that contains the tubing and check to see that the carbon capture apparatus is fully assembled. Turn the stir plate on and allow the vial to stir at approximately 200 rpm. Do NOT turn up the heat. This is a room-temperature experiment.
6. Using the plastic syringe with needle **(Be careful! Sharps!)**, slowly add the citric acid solution to the sodium bicarbonate over the course of around 20 minutes. **Make sure citric acid does not get in the tube that connects to the 5 mL vial!** Record your observations. After completing the addition of citric acid, allow the reaction vial to stir for an additional 5 minutes.

   **Note: To save time, one member of your team should begin preparing the citric acid, sodium bicarbonate, and water for the next trial of the experiment.*
7. Remove the 5 mL vial from the apparatus, reweigh it, and record your data to 3 decimal places.
8. Repeat the experiment two more times. If you need to reuse the same 5 mL reaction vial, be sure to rinse and dry the vial before beginning the next trial.
9. Wash and put everything away. Your lab instructor will direct you on how to properly dispose of needles in the sharps container.

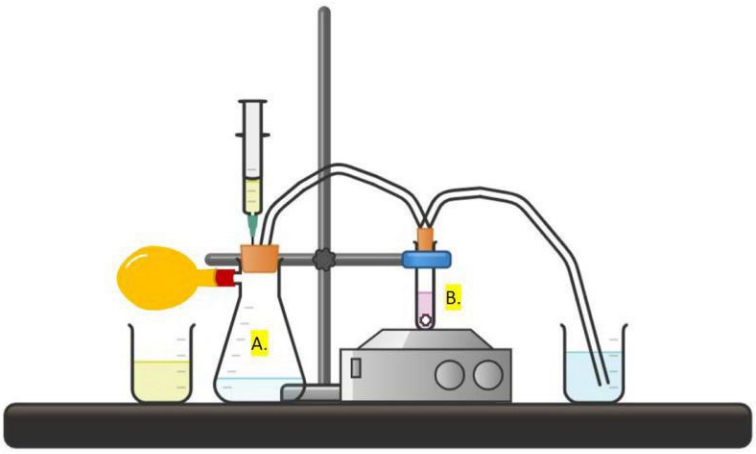
Data Sheet:

Table 1: Mass of CO_2 Captured (report masses to 3 decimal places) and % Relative Standard Deviation

	Trial 1	**Trial 2**	**Trial 3**
Mass of 5 mL reaction vial before CO_2 capture (g)			
Mass of 5 mL reaction vial after CO_2 capture (g)			
Mass of CO_2 captured (after – before) (g)			
Moles of CO_2 captured (mol)			
Average moles CO_2 captured (mol)			
Standard Deviation			
%RSD			

Post-Lab Questions:

1. Based on your %RSD and your observations, how repeatable is your experiment? **%RSD < 20% is ideal.**
2. Where is the CO_2 being generated? Where is it being captured? Label on the picture below or type your answer:



3. Where did you have the most trouble? State one way you will improve your technique next time and suggest one change to the apparatus for the future. Keep in mind that the goal is to increase precision (i.e., decrease %RSD).

Pre-lab assignment.

(1) Watch the YouTube video, linked below. Take notes while you are watching it, and then answer the question below. Causes and Effects of Climate Change | National Geographic | **https://www.youtube.com/watch?v=G4H1N_yXBiA.**

What is the big-picture problem you are trying to help solve in this lab? Why is this important to study?

(2) In your own words, and in complete sentences, define *precision* as related to chemical analysis.

(3) Below is the data from a test of carbon capture using 0.5 M ethylenediamine as the test material. Please read the experimental procedure to better understand where these numbers come from. Go through the calculations you would do to figure out the amount of CO_2 captured, in grams and moles (hint: start with grams and convert to moles). Show your work for one calculation.

Trial Name	**Initial Mass (g)**	**Final Mass (g)**	**CO_2 Captured (g)**	**CO_2 Captured (mol)**
Ethylenediamine 1	31.1752	31.2143		
Ethylenediamine 2	30.5885	30.625		
Ethylenediamine 3	30.5973	30.6295		

Sample calculation:

References:
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(2) Climate Change: Atmospheric Carbon Dioxide | NOAA Climate.gov https://www.climate.gov/news-features/understanding-climate/climate-change-atmospheric-carbon-dioxide (accessed 2021 -06 -04).
(3) Leclaire, J.; Heldebrant, D. J. A Call to (Green) Arms: A Rallying Cry for Greenchemistry and Engineering for CO2 Capture, Utilisation and Storage. *Green Chem.* **2018**, *20* (22), 5058–5081.
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Additional resources:
https://www.youtube.com/watch?v=2cWa5ENWxAg "How to make Carbon Dioxide (The Old-Fashioned Way)" by Periodic Videos
Better Carbon Capture Through Chemistry (Fossil Fuels vs Carbon Capture, and other topics): https://cen.acs.org/articles/93/web/2015/12/Better-Carbon-Capture-Through-Chemistry.html
Video about deep eutectic solvents to make the paper industry greener. "Introducing: PROVIDES - Deep Eutectic Solvents": https://www.youtube.com/watch?v=2oDgVDhUXAY

CHEMISTRY 142 – Chemical Principles II Laboratory – Spring 2022
Lab 2: Testing Materials for CO_2 Capture
Week 2 of 3

Project Goal. In this Project-Based lab, you will evaluate the effectiveness of different test materials in "capturing" carbon dioxide (CO_2).

Experimental Goals and Introduction. In Part 1 of this lab, you tested the carbon-capturing abilities of a neutral control, water. This week, you will test a material that has been shown to capture carbon effectively: sodium hydroxide (NaOH). You will also synthesize a deep eutectic solvent (DES), to test next week. You will learn more about DES materials in your pre-lab assignment.

Carbon capture has the goal of undoing the release of CO_2 into the environment to prevent environmental devastation. However, carbon capture technologies will not be very helpful in this goal if they involve materials or processes that damage the environment in other ways! To consider these potential trade-offs, we can look to the field of "green chemistry. Green chemistry is "the design of chemical products and processes that reduce or eliminate the use or generation of hazardous substances,"[1] meaning it focuses on the prevention of environmental hazards rather than repairing those hazards after the fact. There are twelve major principles of green chemistry that can be used as a guide when considering whether something is "green." These are summarized on the next page.

In the second week of this experiment, you will test an aqueous solution of sodium hydroxide (NaOH), which is a commonly used material for carbon capture. After CO_2 gas dissolves in the solution, it reacts with NaOH to form sodium carbonate (Na_2CO_3). The Na_2CO_3 can then react with more CO_2 to form sodium bicarbonate ($NaHCO_3$). This process is described by the chemical reactions below:

$$\mathbf{CO_2(g) \rightleftarrows CO_2(aq)}$$

$$\mathbf{CO_2(aq) + 2NaOH(aq) \rightleftarrows Na_2CO_3(aq) + H_2O(l)}$$

$$\mathbf{Na_2CO_3(aq) + H_2O(l) + CO_2(aq) \rightleftarrows 2NaHCO_3(aq)}$$

Below is a table summarizing the materials you have tested and will test throughout this project, along with their freezing points, viscosity, and pH. These are not necessarily all properties that affect CO_2 capture, but they are a good starting point when making hypotheses.

Table 1: Materials tested in this project and their properties[2–6]

Material	Composition	Freezing Point	Viscosity at ~25°C	pH	Density
DI water	water	0 °C	0.89 cP	7.0 (5.8 if exposed to air)	0.997 g/mL
0.5 M NaOH	NaOH and water	0 °C	0.9145 cP	13.7	1.02 g/mL
Reline (DES)	choline chloride + urea	12.7 °C	748.09 cP	10.39	1.19 g/mL
Ethaline (DES)	choline chloride + ethylene glycol	-66 °C	32.1 cP	4.68	1.31 g/mL
Glyceline (DES)	choline chloride + glycerol	< -40°C	113 cP	6.0	1.19 g/mL

The 12 Principles of GREEN CHEMISTRY

Green chemistry is an approach to chemistry that aims to maximize efficiency and minimize hazardous effects on human health and the environment. While no reaction can be perfectly 'green', the overall negative impact of chemistry research and the chemical industry can be reduced by implementing the 12 Principles of Green Chemistry wherever possible.

1. WASTE PREVENTION

Prioritize the prevention of waste, rather than cleaning up and treating waste after it has been created. Plan ahead to minimize waste at every step.

2. ATOM ECONOMY

Reduce waste at the molecular level by maximizing the number of atoms from all reagents that are incorporated into the final product. Use atom economy to evaluate reaction efficiency.

3. LESS HAZARDOUS CHEMICAL SYNTHESIS

Design chemical reactions and synthetic routes to be as safe as possible. Consider the hazards of all substances handled during the reaction, including waste.

4. DESIGNING SAFER CHEMICALS

Minimize toxicity directly by molecular design. Predict and evaluate aspects such as physical properties, toxicity, and environmental fate throughout the design process.

5. SAFER SOLVENTS & AUXILIARIES

Choose the safest solvent available for any given step. Minimize the total amount of solvents and auxiliary substances used, as these make up a large percentage of the total waste created.

6. DESIGN FOR ENERGY EFFICIENCY

Choose the least energy-intensive chemical route. Avoid heating and cooling, as well as pressurized and vacuum conditions (i.e. ambient temperature & pressure are optimal).

7. USE OF RENEWABLE FEEDSTOCKS

Use chemicals which are made from renewable (i.e. plant-based) sources, rather than other, equivalent chemicals originating from petrochemical sources.

8. REDUCE DERIVATIVES

Minimize the use of temporary derivatives such as protecting groups. Avoid derivatives to reduce reaction steps, resources required, and waste created.

9. CATALYSIS

Use catalytic instead of stoichiometric reagents in reactions. Choose catalysts to help increase selectivity, minimize waste, and reduce reaction times and energy demands.

10. DESIGN FOR DEGRADATION

Design chemicals that degrade and can be discarded easily. Ensure that both chemicals and their degradation products are not toxic, bioaccumulative, or environmentally persistent.

11. REAL-TIME POLLUTION PREVENTION

Monitor chemical reactions in real-time as they occur to prevent the formation and release of any potentially hazardous and polluting substances.

12. SAFER CHEMISTRY FOR ACCIDENT PREVENTION

Choose and develop chemical procedures that are safer and inherently minimize the risk of accidents. Know the possible risks and assess them beforehand.

Learning Outcomes for this Lab:
After completing this project-based lab, you will be able to…

1. Identify a problem, develop a research question, and state a hypothesis.
2. Perform gravimetric analysis and prepare solutions accurately and precisely.
3. Draw conclusions from experimental data and identify potential limitations.
4. Access and comprehend scientific media designed for general audiences.

Pre-lab Discussion.

After the pre-lab introduction with your instructor, please have a discussion with the other students at your lab table about the following topics:

1. What are two of the green chemistry principles being addressed in this project?
2. What are some advantages and disadvantages to using NaOH for carbon capture?
3. Based on how the experiment went last week, what is a tip you would give to the class moving forward, or to someone else repeating the experiment?

Qualitative Observations. As this is a new lab experiment, you will be graded on the quality of your observations. For each step be sure to record not only what you "see", but also any challenges or questions. You will be graded on the accuracy and completeness of these observations, and you will use your observations answer post-lab questions.

Experimental Procedure: This week, you will test the efficiency of NaOH to capture CO_2 gas using *gravimetric analysis* and your carbon capture apparatus. You will also continue to evaluate the precision of the apparatus.

Materials.

- 2-3 20 mL glass reaction vials
- Stiff tubing piercing rubber septa and septum cap
- 5 mL plastic syringe with needle
- P1000 micropipette
- 1 mid-sized spin vane
- 1 vacuum flask (125 mL, with short piece of vacuum tubing attached)
- 2 Beakers (100 mL)
- Balloon

Week 2 Protocol

Note: To take full advantage of your time and teamwork, it is highly recommended that you have one team member complete Part I (Testing Carbon Capture), while another team member completes Part II (Synthesis of DES). A hot plate will be kept in the fume hood for the DES heating and mixing.

Part I: Testing Carbon Capture

This is the almost same procedure as the one followed last week. Remember to record the mass with three decimal places and write down your observations.

1. Add 10 g citric acid to a beaker. Using a graduated cylinder, measure out 10 mL DI water and add to the citric acid. Make sure to stir the solution with a stirring rod until it is fully dissolved.
2. Add 6.5 g sodium bicarbonate and 20 mL DI water to the 125 mL vacuum flask. Stir the solution with a stirring rod to mix, but note that the sodium bicarbonate will not fully dissolve.
3. Using Figure 1 and the example apparatus as a guide, construct the carbon capture apparatus.
4. Using a P1000 micropipette, add 5.00 mL of 0.5 M NaOH, 1 mL at a time, to the glass reaction vial and weigh (with spin vane) to get its mass. DO NOT try to pick up all 5 mL of NaOH at once with the micropipette.

This will damage the micropipette. Add the spin vane to the reaction vial. Record the initial mass of the reaction vial, water, and spin vane (with no cap on the vial) to 3 decimal places.

5. Cap your reaction vial with the septum cap that contains the tubing and check to see that the carbon capture apparatus is fully assembled. Turn the stir plate on and allow the vial to stir at approximately 200 rpm. Do NOT turn up the heat. This is a room-temperature experiment.
6. Using the plastic syringe with needle **(Be careful! Sharps!)**, slowly add the citric acid solution to the sodium bicarbonate over the course of around 20 minutes. **Make sure citric acid does not get in the tube that connects to the vial!** Record your observations. After completing the addition of citric acid, allow the reaction vial to stir for an additional 5 minutes.**Note: To save time, one member of your team should begin preparing the citric acid, sodium bicarbonate, and water for the next trial of the experiment.*
7. Remove the 5 mL vial from the apparatus, reweigh it, and record your data to 3 decimal places.
8. Repeat the experiment two more times. If you need to reuse the same 5 mL reaction vial, be sure to rinse and dry the vial before beginning the next trial.
9. Wash and put everything away. Your lab instructor will direct you on how to properly dispose of needles in the sharps container. Do not throw away the balloon (unless it is broken) or remove the tubing from the rubber septa.

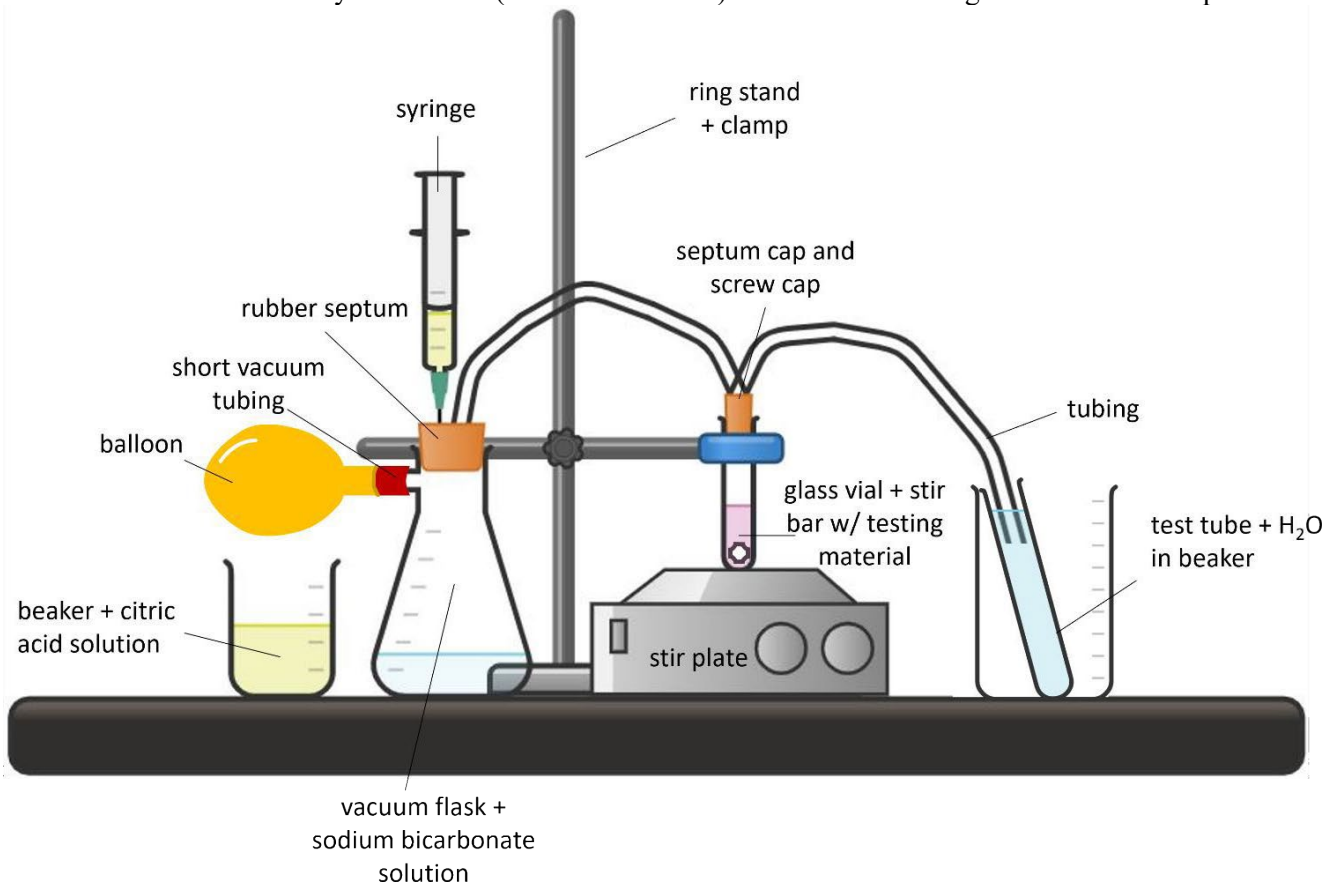


Figure 1: Carbon capture apparatus. Original design by Myrria Lyncee. Illustration made in Chemix by Elise Eng

Part II: Synthesis of Deep Eutectic Solvent (DES)

Your lab instructor will assign your team one of the three DES materials:

- Reline: Composed of 10.00 g choline chloride and 8.60 g of urea
- Ethaline: Composed of 10.00 g choline chloride and 9.16 g of ethylene glycol
- Glyceline: Composed of 5.00 g choline chloride and 13.20 g of glycerol

1. Using weigh paper (remember to tare the scale after placing the paper), measure out 10.00 g of choline chloride and add it to a 50 mL Erlenmeyer flask.

2. Weigh the appropriate amount of your second material. Refer to the list of DES materials above to determine the appropriate material and quantity. Add this to the Erlenmeyer flask.
3. Using tape, label your Erlenmeyer flask with the name of the DES, the date, and your initials.
4. Add a stir bar and stir on the hot plate at 70 ºC for 30 minutes or until liquid. A hot plate for this purpose will be kept in the fume hood so that we do not accidentally heat our carbon capture experiment. Write down your observations. How did the consistency and/or color of your mixture change over time? Cover the top with foil and set it aside. This will be stored in a 60 ºC oven until it is used in the next experiment.

Data Sheet:

Table 1: Mass of CO_2 Captured with NaOH solution (report masses to 3 decimal places) and % Relative Standard Deviation

	Trial 1	**Trial 2**	**Trial 3**
Mass of reaction vial before CO_2 capture (g)			
Mass of reaction vial after CO_2 capture (g)			
Mass of CO_2 captured (after – before) (g)			
Moles of CO_2 captured (mol)			
Average moles CO_2 captured (mol)			
Average moles CO_2 captured / mL of solution (mol/mL)			
Standard Deviation			
%RSD			

Table 2: Synthesis of DES

Name of DES Prepared by Team	
Exact mass of choline chloride added, g	
Exact mass of {insert material name} added, g	

Post-Lab Questions:

1. Based on your %RSD and your observations, how repeatable is your experiment? **%RSD < 5% is ideal in this case.** How does this compare to your data from last week?

2. Previous studies have shown that 0.5 M NaOH will capture about 0.024 g CO_2/mL. How does your data compare to this?

3. Making a hypothesis: Which material (water, 0.5 M NaOH, reline, ethaline, or glyceline) do you think will be the most effective at capturing CO_2 and why? It may be helpful to reference the information in Table 1. Write your answer and justification in complete sentences.

Pre-Lab Assignment:

(1) Read the C&EN (Chemical & Engineering News) article titled, "Mixed Solids Form Low-Freezing Liquids" by Michael Freemantle. Based on what you learned, define a deep eutectic solvent (DES) in your own words. Use complete sentences. What is one unique property of DESs? What is one potential application of DESs?

(2) Here are the 12 principles of green chemistry: https://www.compoundchem.com/2015/09/24/green-chemistry/ What are two principles being addressed in this 3-week laboratory project? Briefly explain your response.

(3) Based on the SDS for 0.5 M NaOH and the principles of green chemistry, what are some advantages and disadvantages to using NaOH for carbon capture?

(4) Consider the two sets of data provided below. Which one would be considered more precise? In complete sentences, justify your answer.

Table 2: Sample data from carbon capture with 0.3 M NaOH and 0.1 M EDA

Test Material A	Initial Mass (g)	Final Mass (g)	CO_2 Captured (g)	Test Material B	Initial Mass (g)	Final Mass (g)	CO_2 Captured (g)
0.3 M NaOH Trial 1	31.2722	31.3093	0.0371	0.1 M EDA Trial 1	30.5654	30.5775	0.0121
0.3 M NaOH Trial 2	30.6079	30.6399	0.032	0.1 M EDA Trial 2	31.2222	31.2388	0.0166
0.3 M NaOH Trial 3	31.2756	31.309	0.0334	0.1 M EDA Trial 3	30.5654	30.5813	0.0159
		Avg	0.03416667			**Avg**	0.01486667
		Std Dev	0.002151			**Std Dev**	0.001977
		%RSD	6.2970%			**%RSD**	13.2988%

References:

(1) US EPA, O. Basics of Green Chemistry https://www.epa.gov/greenchemistry/basics-green-chemistry (accessed 2021 -11 -06).

(2) Vázquez, G.; Alvarez, E.; Varela, R.; Cancela, A.; Navaza, J. M. Density and Viscosity of Aqueous Solutions of Sodium Dithionite, Sodium Hydroxide, Sodium Dithionite + Sucrose, and Sodium Dithionite + Sodium Hydroxide + Sucrose from 25 °C to 40 °C. *J. Chem. Eng. Data* **1996**, *41* (2), 244–248. https://doi.org/10.1021/je950243k.

(3) Ibrahim, R. K.; Hayyan, M.; AlSaadi, M. A.; Ibrahim, S.; Hayyan, A.; Hashim, M. A. Physical Properties of Ethylene Glycol-Based Deep Eutectic Solvents. *Journal of Molecular Liquids* **2019**, *276*, 794–800. https://doi.org/10.1016/j.molliq.2018.12.032.

(4) Manurung, R.; Simanjuntak, G.; Perez, R.; Syahputra, A.; Alhamdi, M.; Siregar, H.; Zuhri, R. Production of Choline Chloride-Based Deep Eutectic Solvent with Hydrogen Bond Donor D-Glucose and Ethylene Glycol. *IOP Conference Series: Materials Science and Engineering* **2019**, *505*, 012134. https://doi.org/10.1088/1757-899X/505/1/012134.

(5) Mjalli, F. S.; Ahmed, O. U. Physical Properties and Intermolecular Interaction of Eutectic Solvents Binary Mixtures: Reline and Ethaline: Reline-Ethaline. *Asia-Pac. J. Chem. Eng.* **2016**, *11* (4), 549–557. https://doi.org/10.1002/apj.1978.

(6) Abbott, A. P.; Harris, R. C.; Ryder, K. S.; D'Agostino, C.; Gladden, L. F.; Mantle, M. D. Glycerol Eutectics as Sustainable Solvent Systems. *Green Chem.* **2011**, *13* (1), 82–90. https://doi.org/10.1039/C0GC00395F.

Additional resources:

- Basics of green chemistry: https://www.epa.gov/greenchemistry/basics-green-chemistry
- Video about deep eutectic solvents to make the paper industry greener. "Introducing: PROVIDES - Deep Eutectic Solvents": https://www.youtube.com/watch?v=2oDgVDhUXAY

CHEMISTRY 142 – Chemical Principles II Laboratory – Spring 2022
Lab 2: Testing Materials for CO_2 Capture
Week 3 of 3

Project Goal. In this Project-Based lab, you will evaluate the effectiveness of different test materials in "capturing" carbon dioxide (CO_2).

Experimental Goals and Introduction. As you learned last week, one potential method of capturing carbon is to use deep eutectic solvents (DESs). A DES forms by mixing two chemicals that lower each other's melting points to the *eutectic point*, allowing them to mix as a liquid even if they started out as two solid materials. These have several potential applications, such as in metal polishing and chrome plating,[1] but we are interested in their ability to absorb CO_2. Currently, researchers do not know the exact mechanism for CO_2 absorption by DES materials. Therefore, we do not have a known chemical reaction for this week's experiment.

In this experiment, you will test the DES you previously prepared at a 1:2 ratio of choline chloride with a hydrogen-bond donor (urea, ethylene glycol, or glycerol). You will then compare this material's data with the data for DI water and NaOH to determine which material was most effective in carbon capture.

Glossary:
Eutectic point – the point at which the conditions are such that the substance of interest has the lowest melting/freezing point possible

Learning Outcomes for this Lab:

After completing this project-based lab, you will be able to…
1. Identify a problem, develop a research question, and state a hypothesis.
2. Perform gravimetric analysis and prepare solutions accurately and precisely.
3. Draw conclusions from experimental data and identify potential limitations.
4. Access and comprehend scientific media designed for general audiences.

Pre-lab Discussion.
After the pre-lab introduction with your instructor, please have a discussion with the other students at your lab table about the following topics:

1. What was your hypothesis? Did anyone at your table convince you to change your hypothesis?
2. How did your precision from week 1 (DI water) compare with week 2 (NaOH)?

Qualitative Observations. As this is a new lab experiment, you will be graded on the quality of your observations. For each step be sure to record not only what you "see," but also any challenges or questions. You will be graded on the accuracy and completeness of these observations which you will use in answering post-lab questions.

Materials.

- 2-3 20 mL glass reaction vials
- Stiff tubing piercing rubber septa and septum cap
- 5 mL plastic syringe with needle
- P1000 micropipette
- 1 mid-sized spin vane
- 1 vacuum flask (125 mL, with short piece of vacuum tubing attached)
- 2 Beakers (100 mL)
- Balloon

Carbon Capture Apparatus

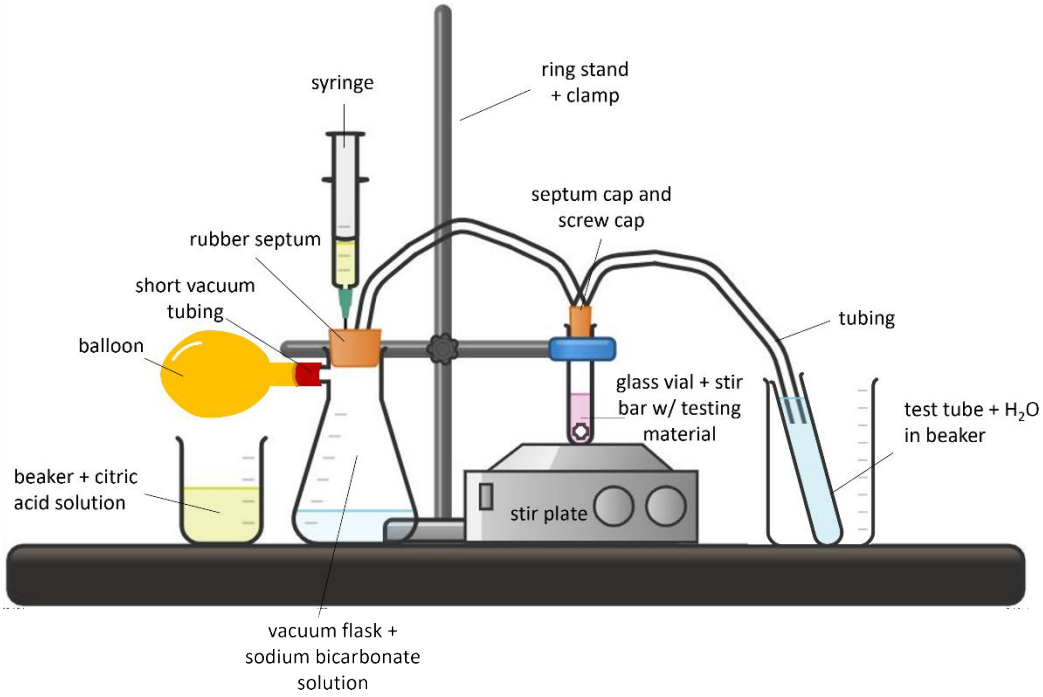


Figure 1: Carbon capture apparatus. Original design by Myrria Lyncee. Illustration made in Chemix by Elise Eng.

Week 3 Protocol

This is the almost same procedure as the one followed in the past two weeks. Remember to record the mass with three decimal places and write down your observations, including any changes from the protocol.

1. Add 10 g citric acid to a beaker. Using a graduated cylinder, measure out 10 mL DI water and add to the citric acid. Make sure to stir the solution with a stirring rod until it is fully dissolved.
2. Add 6.5 g sodium bicarbonate and 20 mL DI water to the 125 mL vacuum flask. Stir the solution with a stirring rod to mix, but note that the sodium bicarbonate will not fully dissolve.
3. Using Figure 1 and the example apparatus as a guide, construct the carbon capture apparatus.
4. DESs are very viscous and difficult to measure and transfer volumetrically. Instead of measuring out your DES into the vial with a micropipette or graduated cylinder, use a disposable plastic pipette to add the correct mass of DES. This is the amount you determined in Question 5 of the Pre-Lab Assignment.
5. Add the spin vane to the reaction vial. Record the initial mass of the reaction vial, water, and spin vane (with no cap on the vial) to 3 decimal places.
6. Cap your reaction vial with the septum cap that contains the tubing and check to see that the carbon capture apparatus is fully assembled. Turn the stir plate on and allow the vial to stir at approximately 200 rpm. Do NOT turn up the heat. This is a room-temperature experiment.
7. Using the plastic syringe with needle **(Be careful! Sharps!)**, slowly add the citric acid solution to the sodium bicarbonate over the course of around 20 minutes. **Make sure citric acid does not get in the tube that connects to the vial!** Record your observations. After completing the addition of citric acid, allow the reaction vial to stir for an additional 5 minutes.
   **Note: To save time, one member of your team should begin preparing the citric acid, sodium bicarbonate, and DES for the next trial of the experiment.*
8. Remove the vial from the apparatus, reweigh it, and record your data to 3 decimal places.
9. Repeat the experiment two more times. If you need to reuse the same reaction vial, be sure to rinse and dry the vial before beginning the next trial.
10. Wash and put everything away. Your lab instructor will direct you on how to properly dispose of needles in the sharps container. Do not throw away the balloon (unless it is broken) or remove the tubing from the rubber septa.

Data Sheet:

Table 1: Mass of CO_2 Captured with DES solution (report masses to 3 decimal places) and % Relative Standard Deviation

	Trial 1	**Trial 2**	**Trial 3**
Mass of 5 mL reaction vial before CO_2 capture (g)			
Mass of 5 mL reaction vial after CO_2 capture (g)			
Mass of CO_2 captured (after – before) (g)			
Moles of CO_2 captured (mol)			
Average moles CO_2 captured (mol)			
Average moles CO_2 captured / mL solution (mol/mL)			
Standard Deviation			
%RSD			

Figure 1: Comparison of CO_2 Capture Capability of Different Materials

{Insert the Figure from the Excel Template}

Post-Lab Questions:

1. Which of the three testing materials was the most effective at capturing CO_2? Use your data from Weeks 2 & 3 and the previously obtained data for the DI water for comparison. Was your hypothesis from Week 2 correct? Why or why not?

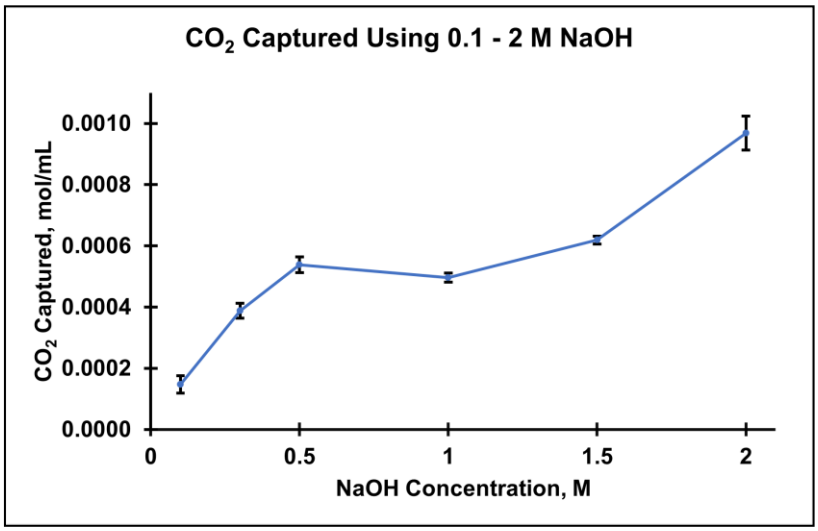
2. If you were to continue this research, what do you think you could try next? What changes would you make to the experimental setup to improve precision?

3. Read the sample research question below. Develop a new research question that future students could answer, and then write a few sentences about how you would test this in the lab. Think about what other variables could be changed… materials, conditions like temperature, the apparatus, etc. A research question should be specific, measurable, and complex (not a yes or no question).

   Example: "How do deep eutectic solvents compare with sodium hydroxide for effectiveness in carbon capture?"

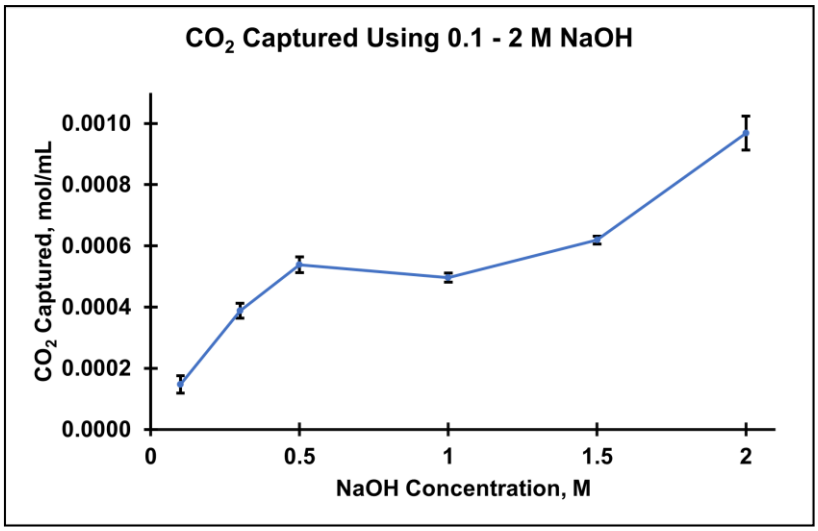
4. List your %RSD for each week of the lab. How did your precision change over the course of the 3-week lab?

5. Below is a figure showing the amount of CO_2 captured at different concentrations of NaOH. What concentration of NaOH would you estimate has the equivalent CO_2 capture capability as your DES?



Pre-Lab Assignment:

1. Unlike some carbon capturing materials, NaOH does not have a direct correlation between concentration and amount of CO_2 captured; instead, it is more like a curve with multiple bends. Consider the data for different concentrations of NaOH shown below (Note: concentration is on the x-axis and CO_2 capture in mol/mL solution is on the y-axis):



This data was gathered using various concentrations of NaOH, and the error bars on each point represent the standard deviation (so smaller error bars indicate a more precise measurement). **How does your data from Week 2 compare to this data? Did you measure higher CO_2 capture, lower, or about the same?**

2. What are some skills you have learned or improved upon as part of this lab? Did your overall precision improve from Week 1 to Week 2?

3. Due to their high viscosity, the DESs are difficult to measure by volume.

DES	Density
Choline Chloride + Urea (Reline)	1.188 g/mL
Choline Chloride + Ethylene Glycol (Ethaline)	1.313 g/mL
Choline Chloride + Glycerol (Glyceline)	1.192 g/mL

Calculate how many grams of the DES you prepared last week would need to be weighed out to be equivalent to 5 mL. Show your calculation.

References:

(1) Smith, E. L.; Abbott, A. P.; Ryder, K. S. Deep Eutectic Solvents (DESs) and Their Applications. *Chem. Rev.* **2014**, *114* (21), 11060–11082. https://doi.org/10.1021/cr300162p.

Carbon Capture Lab Prep – Materials Summary

Week 1

Materials:

Quantity	Quantity/Pair of students	Material
8	1	20 mL glass reaction vial
8	1	Rubber septum and septum cap attached by thin tubing (Dr. Brush is assembling)
8	1	Short piece of vacuum tubing
8	1	125-mL vacuum flask with arm
16	2	100-mL beaker
8	1	5-mL syringe with needle (pink 18 gauge)
8	1	P1000 micropipette
8	1	Mid-sized spin vane
8	1	Balloon

Dry chemicals:

Quantity	Quantity/Pair of students	Chemical
240 g	30 g	Citric acid
160 g	20 g	Sodium bicarbonate

Week 2

Materials:

Quantity	Quantity/Pair of students	Material
8	1	20 mL glass reaction vial
8	1	Rubber septum and septum cap attached by thin tubing (Dr. Brush is assembling)
8	1	Short piece of vacuum tubing
8	1	125-mL vacuum flask with arm
16	2	100-mL beaker
8	1	5-mL syringe with needle (pink 18 gauge)
8	1	P1000 micropipette
8	1	Mid-sized spin vane
8	1	Balloon
8	1	50-mL Erlenmeyer flask
8	1	Mid-sized stir bar

Reagents:

Quantity	Quantity/Pair of students	Chemical
240 g	30 g	Citric acid
160 g	20 g	Sodium bicarbonate
40 g	10 g	Choline Chloride
~20 g	8.6 g	Urea
~20 mL	8 mL	Ethylene glycol
~25 mL	10.46 mL	Glycerol
~25 mL	10.46 mL	Propylene Glycol

Pre-made solutions:

Quantity	Quantity/Pair of students	Solution
32 mL	15 mL	0.5 M aqueous solution of NaOH

Week 3

Materials:

Quantity	Quantity/Pair of students	Material
8	1	20 mL glass reaction vial
8	1	Rubber septum and septum cap attached by thin tubing (Dr. Brush is assembling)
8	1	Short piece of vacuum tubing
8	1	125-mL vacuum flask with arm
16	2	100-mL beaker
8	1	5-mL syringe with needle (pink 18 gauge)
8	1	P1000 micropipette
8	1	Mid-sized spin vane
8	1	Balloon
4	--	Plastic disposable transfer pipet

Dry chemicals:

Quantity	Quantity/Pair of students	Chemical
240 g	30 g	Citric acid
160 g	20 g	Sodium bicarbonate

Other chemicals:

- DES solutions prepared in Week 2

CHEMISTRY 142 – Chemical Principles II Laboratory
Lab 2: Testing Materials for CO_2 Capture
Week 1 of 3

Project Goal. In this project-based lab, you will evaluate the effectiveness of different test materials in "capturing" carbon dioxide (CO_2).

Experimental Goals and Introduction. Most people are familiar with or at least aware of current environmental issues, whether it be air pollution, excessive waste, or coastal flooding. One major aspect of these issues is the steadily increasing amount of carbon dioxide (CO_2) gas in the air.[1] CO_2 is a necessary *greenhouse gas*, keeping the earth from freezing, but there is too much of it in the air. This leads to rising global temperatures and an unstable climate. Excess CO_2 also contributes to air pollution and pH imbalance in seawater, which becomes acidic as it absorbs increasing amounts of CO_2.[2] To combat this, researchers are developing methods to remove CO_2 from the air and then convert it to a material that can be used for other purposes. "Carbon Capture Utilization and Storage" (CCUS) refers to the technology and science designed around this challenge (in this context, "carbon" means "CO_2").[3]

In this project-based lab, you will be using a simple apparatus to generate CO_2 gas and study the efficiency of different test materials in carbon capture using *gravimetric analysis*. This project has two major objectives:

Objective 1: Evaluate the precision of the carbon capture apparatus.
Objective 2: Evaluate the relative ability of test materials to capture CO_2 gas.

You will generate CO_2 gas, expose different test materials to the CO_2 gas, and then use the change in mass of the test material to calculate how much CO_2 was captured. When doing a *quantitative analysis* of the change in mass, it is important to know the precision and reliability of your experimental setup and technique. To do this, you will run the same experiment in triplicate, and then check the precision of your data by calculating the relative standard deviation, (%RSD). Precision refers to the reproducibility of repetitive sets of data and can be expressed as %RSD. %RSD is calculated by taking the standard deviation of the data points, and dividing it by the mean value of the data points:

$$\%RSD = \frac{standard\ deviation}{mean} \times 100$$

This can be quickly determined in Excel using the AVERAGE() function to calculate the mean, and the STDEV.S() function to calculate the standard deviation. The lower the %RSD, the better the precision. A high %RSD means that the measurements varied a lot when the same experiment was repeated.

You will be provided with data on using water to absorb CO_2 gas. As you will see, not much CO_2 is captured, but this type of data is called a "control," to which you will compare your data from testing other materials. In this case, the CO_2 gas dissolves in water, forming an *aqueous solution*. Aqueous CO_2 then slowly reacts with water to form carbonic acid. This is why pure water in contact with air tends to have a slightly acidic pH rather than a neutral pH of 7. This process is described by the chemical reactions below:

$$\mathbf{CO_2(g) \rightleftarrows CO_2(aq)}$$

$$\mathbf{CO_2(aq) + H_2O(l) \rightleftarrows H_2CO_3(aq)}$$

In this week, you will test an aqueous solution of sodium hydroxide (NaOH), which is a commonly used material for carbon capture. After CO_2 gas dissolves in the solution, it reacts with NaOH to form sodium carbonate (Na_2CO_3). The Na_2CO_3 can then react with more CO_2 to form sodium bicarbonate ($NaHCO_3$).
This process is described by the chemical reactions below:

$$\mathbf{CO_2(g) \rightleftarrows CO_2(aq)}$$

$$\mathbf{CO_2(aq) + 2NaOH(aq) \rightleftarrows Na_2CO_3(aq) + H_2O(l)}$$

$Na_2CO_3(aq) + H_2O(l) + CO_2(aq) \rightleftarrows 2NaHCO_3(aq)$

You will also synthesize a deep eutectic solvent (DES), to test next week. You will learn more about DES materials in week two.

*Glossary:
Greenhouse gas – a gas that absorbs infrared radiation and prevents too much heat from leaving the atmosphere.
Eutectic point – in a mixture, the lowest possible melting point for all ratios of components.
Precision – in chemical analysis, the reproducibility of a measurement (how close replicate measurements are to each other). This is different from accuracy, which is how close a measurement is to the true value.
Quantitative analysis - a technique to determine how much of a certain component is present in a sample
Gravimetric analysis - a technique that uses the measurement of mass to make a conclusion about the chemicals present in a sample. (This is the same *gravi-* as in *gravity*.)
Project-based learning - in education, a method of teaching/learning that has students engaging in meaningful, longer-term projects that connect to the real world.
Aqueous solution – a solution where water is the solvent.

Learning Outcomes for this Lab:
After completing this project-based lab, you will be able to…
1. Identify a problem, develop a research question, and state a hypothesis.
2. Perform gravimetric analysis and prepare solutions accurately and precisely.
3. Draw conclusions from experimental data and identify potential limitations.
4. Communicate findings to peers through oral presentation.
5. Access and comprehend scientific media designed for general audiences.

Pre-lab Discussion.
As part of the pre-lab introduction with your instructor, you will have a discussion with the other students at your lab table about the following topics:
1. With your team, come up with 3 examples of global environmental problems that chemists can help solve.
2. What is the problem that you are investigating in this project-based lab?
3. Why is this problem important to study?

Qualitative Observations. As this is a new lab experiment, you will be graded on the quality of your observations. For each step be sure to record not only what you "see", but also any challenges or questions. You will be graded on the accuracy and completeness of these observations, and you will use your observations answer post-lab questions.

Experimental Procedure: This week, you will test the efficiency of NaOH to capture CO_2 gas using *gravimetric analysis* and your carbon capture apparatus. You will also continue to evaluate the precision of the apparatus.

Materials.
- 2-3 20 mL glass reaction vials
- Stiff tubing piercing rubber septa and septum cap
- 5 mL plastic syringe with needle
- P1000 micropipette
- 1 mid-sized spin vane
- 1 vacuum flask (125 mL, with short piece of vacuum tubing attached)
- 2 Beakers (100 mL)
- Balloon
- 1 Erlenmeyer flask (50 mL)

Week 2 Protocol

Note: To take full advantage of your time and teamwork, it is highly recommended that you have one team member complete Part I (Testing Carbon Capture), while another team member completes Part II (Synthesis of DES). A hot plate will be kept in the fume hood for the DES heating and mixing.

Part I: Testing Carbon Capture

This is the almost same procedure as the one followed last week. Remember to record the mass with three decimal places and write down your observations.

1. Add 10 g citric acid to a beaker. Using a graduated cylinder, measure out 10 mL DI water and add to the citric acid. Make sure to stir the solution with a stirring rod until it is fully dissolved.
2. Add 6.5 g sodium bicarbonate and 20 mL DI water to the 125 mL vacuum flask. Stir the solution with a stirring rod to mix, but note that the sodium bicarbonate will not fully dissolve.
3. Using Figure 1 and the example apparatus as a guide, construct the carbon capture apparatus.
4. Using a P1000 micropipette, add 5.00 mL of 0.5 M NaOH, 1 mL at a time, to the glass reaction vial and weigh (with spin vane) to get its mass. DO NOT try to pick up all 5 mL of NaOH at once with the micropipette. This will damage the micropipette. Add the spin vane to the reaction vial. Record the initial mass of the reaction vial, water, and spin vane (with no cap on the vial) to 3 decimal places.
5. Cap your reaction vial with the septum cap that contains the tubing and check to see that the carbon capture apparatus is fully assembled. Turn the stir plate on and allow the vial to stir at approximately 200 rpm. Do NOT turn up the heat. This is a room-temperature experiment.
6. Using the plastic syringe with needle **(Be careful! Sharps!)**, slowly add the citric acid solution to the sodium bicarbonate over the course of around 20 minutes. **Make sure citric acid does not get in the tube that connects to the vial!** Record your observations. After completing the addition of citric acid, allow the reaction vial to stir for an additional 5 minutes.
   **Note: To save time, one member of your team should begin preparing the citric acid, sodium bicarbonate, and sodium hydroxide for the next trial of the experiment.*
7. Remove the 5 mL vial from the apparatus, reweigh it, and record your data to 3 decimal places.
8. Repeat the experiment two more times. If you need to reuse the same 5 mL reaction vial, be sure to rinse and dry the vial before beginning the next trial.
9. Wash and put everything away. Your lab instructor will direct you on how to properly dispose of needles in the sharps container. Do not throw away the balloon (unless it is broken) or remove the tubing from the rubber septa.

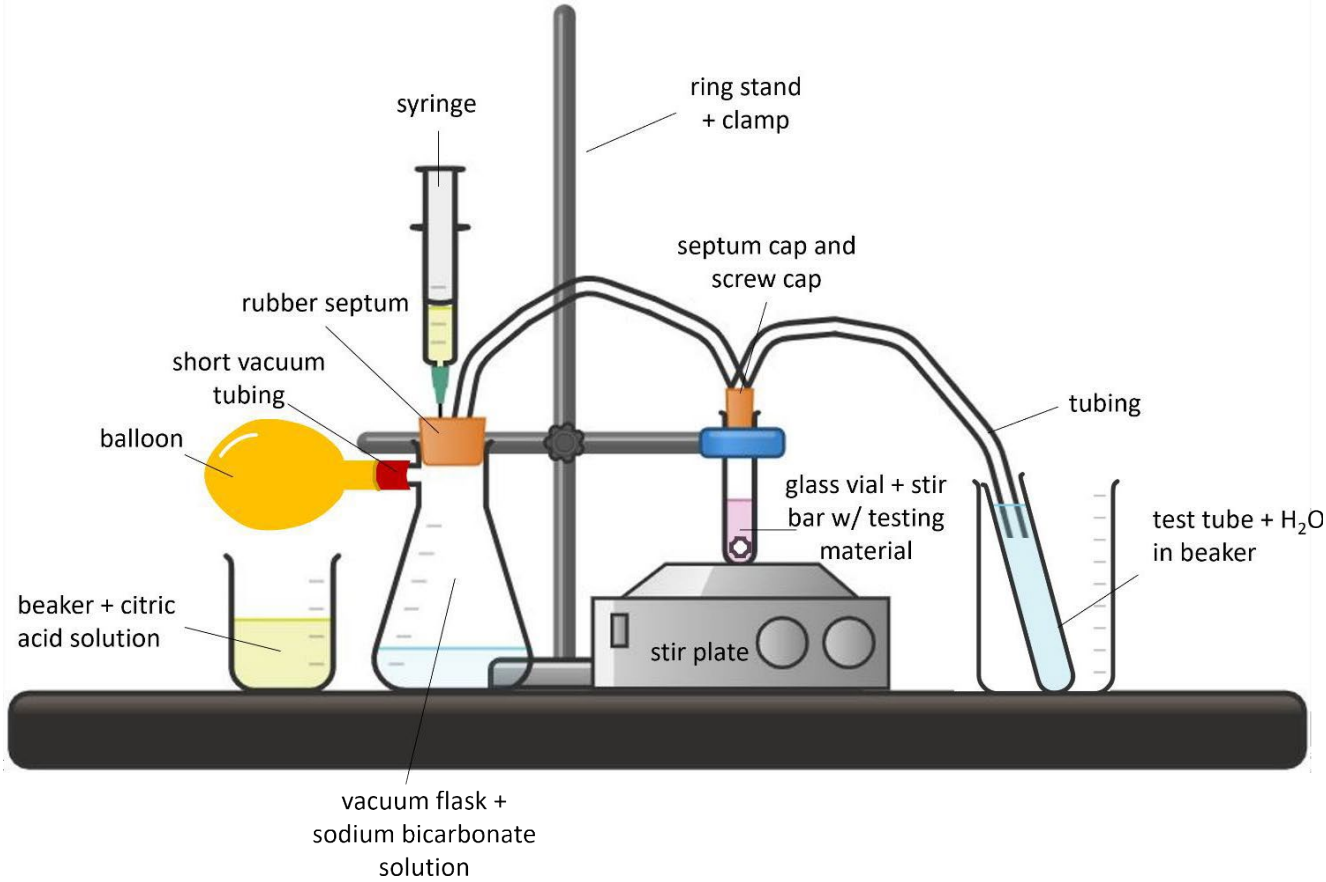


Figure 1: Carbon capture apparatus. Original design by Myrria Lyncee. Illustration made in Chemix by Elise Eng

Part II: Synthesis of Deep Eutectic Solvent (DES)

Your lab instructor will assign your team one of the three DES materials:

- Reline: Composed of 10.00 g choline chloride and 8.60 g of urea
- Ethaline: Composed of 10.00 g choline chloride and 9.16 g of ethylene glycol
- Glyceline: Composed of 5.00 g choline chloride and 13.20 g of glycerol

1. Using weigh paper (remember to tare the scale after placing the paper), measure out 10.00 g of choline chloride and add it to a 50 mL Erlenmeyer flask.
2. Weigh the appropriate amount of your second material. Refer to the list of DES materials above to determine the appropriate material and quantity. Add this to the Erlenmeyer flask.
3. Using tape, label your Erlenmeyer flask with the name of the DES, the date, and your initials.
4. Add a stir bar and stir on the hot plate at 70 ºC for 30 minutes or until liquid. A hot plate for this purpose will be kept in the fume hood so that we do not accidentally heat our carbon capture experiment. Write down your observations. How did the consistency and/or color of your mixture change over time? Cover the top with foil and set it aside. This will be stored in a 60 ºC oven until it is used in the next experiment.

Data Sheet:

Table 1: Mass of CO_2 Captured with NaOH solution (report masses to 3 decimal places) and % Relative Standard Deviation

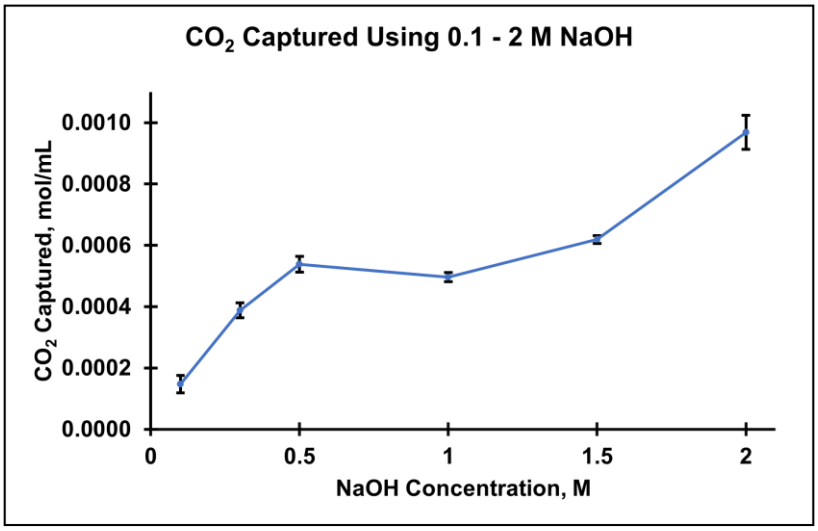
	Trial 1	**Trial 2**	**Trial 3**
Mass of reaction vial before CO_2 capture (g)			
Mass of reaction vial after CO_2 capture (g)			
Mass of CO_2 captured (after – before) (g)			
Moles of CO_2 captured (mol)			
Moles CO_2 captured / mL of solution (mol/mL)			
Average moles CO_2 captured / mL of solution (mol/mL)			
Standard Deviation			
%RSD			

Table 2: Synthesis of DES

Name of DES Prepared by Team (g)	
Exact mass of choline chloride added (g)	
Exact mass of {insert material name} added, (g)	

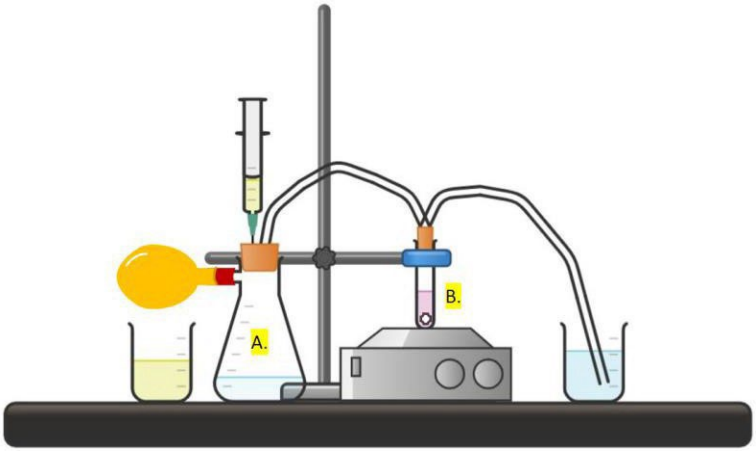
Post-Lab Questions:

1. In your own words, and in complete sentences, define *precision* as related to chemical analysis.
2. Based on your %RSD and your observations, how repeatable is your experiment? **%RSD < 5% is ideal in this case.**
3. Unlike some carbon capturing materials, NaOH does not have a direct correlation between concentration and amount of CO_2 captured; instead, it is more like a curve with multiple bends. Consider the data for different concentrations of NaOH shown below (Note: concentration is on the x-axis and CO_2 capture in mol/mL solution is on the y-axis):



This data was gathered using various concentrations of NaOH, and the error bars on each point represent the standard deviation (so smaller error bars indicate a more precise measurement). **How does your data from Week 1 compare to this data? Did you measure higher CO_2 capture, lower, or about the same?** (Hint: make sure you convert from grams to moles and take into account the volume of NaOH used. MM of CO_2 = 44.01 g/mol)

4. Where is the CO_2 being generated? Where is it being captured? Label on the picture below or type your answer:



5. Where did you have the most trouble? State one way you will improve your technique next time and suggest one change to the apparatus for the future. Keep in mind that the goal is to increase precision (i.e., decrease %RSD).

Pre-lab assignment.

(1) Watch the YouTube video, linked below. Take notes while you are watching it, and then answer the question below. Causes and Effects of Climate Change | National Geographic | **https://www.youtube.com/watch?v=G4H1N_yXBiA.**

What is the big-picture problem you are trying to help solve in this lab? Why is this important to study?

(2) Read the C&EN (Chemical & Engineering News) article titled, "Mixed Solids Form Low-Freezing Liquids" by Michael Freemantle. Based on what you learned, define a deep eutectic solvent (DES) in your own words. Use complete sentences. What is one unique property of DESs? What is one potential application of DESs?

(3) Below is the data from a test of carbon capture using 0.5 M ethylenediamine as the test material. Please read the experimental procedure to better understand where these numbers come from. Go through the calculations you would do to figure out the amount of CO_2 captured, in grams and moles (hint: start with grams and convert to moles). Show your work for one calculation.

Trial Name	**Initial Mass (g)**	**Final Mass (g)**	**CO_2 Captured (g)**	**CO_2 Captured (mol)**
Ethylenediamine 1	31.1752	31.2143		
Ethylenediamine 2	30.5885	30.625		
Ethylenediamine 3	30.5973	30.6295		

Sample calculation:

References:
(1) Keeling, R. F.; Piper, S. C.; Bellenbacher, A. F.; Walker, J. S. *Atomspheric Carbon Dioxide Record from Mauna Loa*. CDIAC. https://data.ess-dive.lbl.gov/view/doi:10.3334/CDIAC/ATG.035 (accessed 2021-06-02).
(2) *Climate Change: Atmospheric Carbon Dioxide | NOAA Climate.gov*. https://www.climate.gov/news-features/understanding-climate/climate-change-atmospheric-carbon-dioxide (accessed 2021-06-04).
(3) Leclaire, J.; Heldebrant, D. J. A Call to (Green) Arms: A Rallying Cry for Greenchemistry and Engineering for CO2 Capture, Utilisation and Storage. *Green Chem.* **2018**, *20* (22), 5058–5081.

Additional resources:
https://www.youtube.com/watch?v=2cWa5ENWxAg "How to make Carbon Dioxide (The Old-Fashioned Way)" by Periodic Videos
Better Carbon Capture Through Chemistry (Fossil Fuels vs Carbon Capture, and other topics): https://cen.acs.org/articles/93/web/2015/12/Better-Carbon-Capture-Through-Chemistry.html
Video about deep eutectic solvents to make the paper industry greener. "Introducing: PROVIDES - Deep Eutectic Solvents": https://www.youtube.com/watch?v=2oDgVDhUXAY

CHEMISTRY 142 – Chemical Principles II Laboratory
Lab 2: Testing Materials for CO_2 Capture
Week 2 of 3

Project Goal. In this Project-Based lab, you will evaluate the effectiveness of different test materials in "capturing" carbon dioxide (CO_2).

Experimental Goals and Introduction. In Part 1 of this lab, you tested the carbon-capturing abilities of well-tested material, sodium hydroxide (NaOH), which has been shown to capture carbon effectively. As you learned last week, one potential method of capturing carbon is to use deep eutectic solvents (DESs), which you also synthesized. A DES forms by mixing two chemicals that lower each other's melting points to the *eutectic point*, allowing them to mix as a liquid even if they started out as two solid materials. These have several potential applications, such as in metal polishing and chrome plating,[2] but we are interested in their ability to absorb CO_2. Currently, researchers do not know the exact mechanism for CO_2 absorption by DES materials. Therefore, we do not have a known chemical reaction for this week's experiment.

In this experiment, you will test the DES you previously prepared at a 1:2 ratio of choline chloride with a hydrogen-bond donor (urea, ethylene glycol, or glycerol). You will then compare this material's data with the data for DI water and NaOH to determine which material was most effective in carbon capture.

When testing these materials, it is important to consider advantages and disadvantages beyond and in addition to efficiency. Carbon capture has the goal of undoing the release of CO_2 into the environment to prevent environmental devastation. However, carbon capture technologies will not be very helpful in this goal if they involve materials or processes that damage the environment in other ways! To consider these potential trade-offs, we can look to the field of "green chemistry." Green chemistry is "the design of chemical products and processes that reduce or eliminate the use or generation of hazardous substances,"[1] meaning it focuses on the prevention of environmental hazards rather than repairing those hazards after the fact. There are twelve major principles of green chemistry that can be used as a guide when considering whether something is "green." These are summarized on the next page.

Glossary:
Eutectic point – the point at which the conditions are such that the substance of interest has the lowest melting/freezing point possible (melting/freezing point can be affected by pressure, concentration, formation or breaking of chemical bonds, etc.)

Below is a table summarizing the carbon capturing materials explored throughout this project-based lab, along with their freezing points, viscosity, and pH. These are not necessarily all properties that affect CO_2 capture, but they are a good starting point when making hypotheses.

Table 1: Materials tested in this project and their properties[3–7]

Material	Composition	Freezing Point	Viscosity at ~25°C	pH	Density
DI water	water	0 °C	0.89 cP	7.0 (5.8 if exposed to air)	0.997 g/mL
0.5 M NaOH	NaOH and water	0 °C	0.9145 cP	13.7	1.02 g/mL
Reline (DES)	choline chloride + urea	12.7 °C	748.09 cP	10.39	1.19 g/mL
Ethaline (DES)	choline chloride + ethylene glycol	-66 °C	32.1 cP	4.68	1.31 g/mL
Glyceline (DES)	choline chloride + glycerol	< -40°C	113 cP	6.0	1.19 g/mL

The 12 Principles of GREEN CHEMISTRY

Green chemistry is an approach to chemistry that aims to maximize efficiency and minimize hazardous effects on human health and the environment. While no reaction can be perfectly 'green', the overall negative impact of chemistry research and the chemical industry can be reduced by implementing the 12 Principles of Green Chemistry wherever possible.

1. WASTE PREVENTION

Prioritize the prevention of waste, rather than cleaning up and treating waste after it has been created. Plan ahead to minimize waste at every step.

2. ATOM ECONOMY

Reduce waste at the molecular level by maximizing the number of atoms from all reagents that are incorporated into the final product. Use atom economy to evaluate reaction efficiency.

3. LESS HAZARDOUS CHEMICAL SYNTHESIS

Design chemical reactions and synthetic routes to be as safe as possible. Consider the hazards of all substances handled during the reaction, including waste.

4. DESIGNING SAFER CHEMICALS

Minimize toxicity directly by molecular design. Predict and evaluate aspects such as physical properties, toxicity, and environmental fate throughout the design process.

5. SAFER SOLVENTS & AUXILIARIES

Choose the safest solvent available for any given step. Minimize the total amount of solvents and auxiliary substances used, as these make up a large percentage of the total waste created.

6. DESIGN FOR ENERGY EFFICIENCY

Choose the least energy-intensive chemical route. Avoid heating and cooling, as well as pressurized and vacuum conditions (i.e. ambient temperature & pressure are optimal).

7. USE OF RENEWABLE FEEDSTOCKS

Use chemicals which are made from renewable (i.e. plant-based) sources, rather than other, equivalent chemicals originating from petrochemical sources.

8. REDUCE DERIVATIVES

Minimize the use of temporary derivatives such as protecting groups. Avoid derivatives to reduce reaction steps, resources required, and waste created.

9. CATALYSIS

Use catalytic instead of stoichiometric reagents in reactions. Choose catalysts to help increase selectivity, minimize waste, and reduce reaction times and energy demands.

10. DESIGN FOR DEGRADATION

Design chemicals that degrade and can be discarded easily. Ensure that both chemicals and their degradation products are not toxic, bioaccumulative, or environmentally persistent.

11. REAL-TIME POLLUTION PREVENTION

Monitor chemical reactions in real-time as they occur to prevent the formation and release of any potentially hazardous and polluting substances.

12. SAFER CHEMISTRY FOR ACCIDENT PREVENTION

Choose and develop chemical procedures that are safer and inherently minimize the risk of accidents. Know the possible risks and assess them beforehand.

Learning Outcomes for this Lab:
After completing this project-based lab, you will be able to…

1. Identify a problem, develop a research question, and state a hypothesis.
2. Perform gravimetric analysis and prepare solutions accurately and precisely.
3. Draw conclusions from experimental data and identify potential limitations.
4. Communicate findings to peers through oral presentation.
5. Access and comprehend scientific media designed for general audiences.

Pre-lab Discussion.

After the pre-lab introduction with your instructor, please have a discussion with the other students at your lab table about the following topics:

1. What are two of the green chemistry principles being addressed in this project?
2. What are some advantages and disadvantages to using NaOH for carbon capture?
3. What was your hypothesis? Did anyone at your table convince you to change your hypothesis?
4. Based on how the experiment went last week, what is a tip you would give to the class moving forward, or to someone else repeating the experiment?

Qualitative Observations. As this is a new lab experiment, you will be graded on the quality of your observations. For each step be sure to record not only what you "see", but also any challenges or questions. You will be graded on the accuracy and completeness of these observations, and you will use your observations answer post-lab questions.

Experimental Procedure: This week, you will test the efficiency of a DES to capture CO_2 gas using *gravimetric analysis* and your carbon capture apparatus. You will also continue to evaluate the precision of the apparatus.

Materials.

- 2-3 20 mL glass reaction vials
- Stiff tubing piercing rubber septa and septum cap
- 5 mL plastic syringe with needle
- P1000 micropipette
- 1 mid-sized spin vane
- 1 vacuum flask (125 mL, with short piece of vacuum tubing attached)
- 2 Beakers (100 mL)
- Balloon

Carbon Capture Apparatus

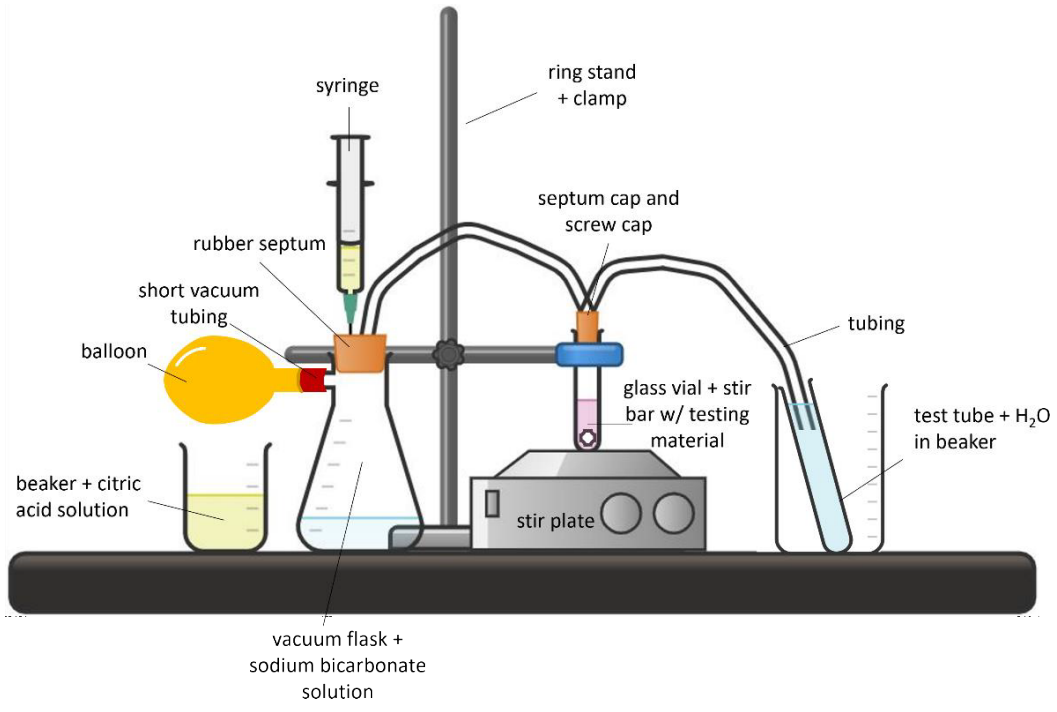


Figure 1: Carbon capture apparatus. Original design by Myrria Lyncee. Illustration made in Chemix by Elise Eng.

Week 2 Protocol

This is the almost same procedure as the one followed in the last week. Remember to record the mass with three decimal places and write down your observations, including any changes from the protocol.

1. Add 10 g citric acid to a beaker. Using a graduated cylinder, measure out 10 mL DI water and add to the citric acid. Make sure to stir the solution with a stirring rod until it is fully dissolved.
2. Add 6.5 g sodium bicarbonate and 20 mL DI water to the 125 mL vacuum flask. Stir the solution with a stirring rod to mix, but note that the sodium bicarbonate will not fully dissolve.
3. Using Figure 1 and the example apparatus as a guide, construct the carbon capture apparatus.
4. DESs are very viscous and difficult to measure and transfer volumetrically. Instead of measuring out your DES into the vial with a micropipette or graduated cylinder, use a disposable plastic pipette to add the correct **mass** of DES. This is the amount you determined in Question 5 of the Pre-Lab Assignment.
5. Add the spin vane to the reaction vial. Record the initial mass of the reaction vial, water, and spin vane (with no cap on the vial) to 3 decimal places.
6. Cap your reaction vial with the septum cap that contains the tubing and check to see that the carbon capture apparatus is fully assembled. Turn the stir plate on and allow the vial to stir at approximately 200 rpm. Do NOT turn up the heat. This is a room-temperature experiment.
7. Using the plastic syringe with needle **(Be careful! Sharps!)**, slowly add the citric acid solution to the sodium bicarbonate over the course of around 20 minutes. **Make sure citric acid does not get in the tube that connects to the vial!** Record your observations. After completing the addition of citric acid, allow the reaction vial to stir for an additional 5 minutes.
   **Note: To save time, one member of your team should begin preparing the citric acid, sodium bicarbonate, and DES for the next trial of the experiment.*
8. Remove the vial from the apparatus, reweigh it, and record your data to 3 decimal places.
9. Repeat the experiment two more times. If you need to reuse the same reaction vial, be sure to rinse and dry the vial before beginning the next trial.
10. Wash and put everything away. Your lab instructor will direct you on how to properly dispose of needles in the sharps container. Do not throw away the balloon (unless it is broken) or remove the tubing from the rubber septa.

Data Sheet:

Table 1: Mass of CO_2 Captured with DES Solution (report masses to 3 decimal places) and % Relative Standard Deviation

	Trial 1	**Trial 2**	**Trial 3**
Mass of 5 mL reaction vial before CO_2 capture (g)			
Mass of 5 mL reaction vial after CO_2 capture (g)			
Mass of CO_2 captured (after – before) (g)			
Moles of CO_2 captured (mol)			
Moles CO_2 captured / mL solution (mol/mL)			
Average moles **CO_2 captured / mL solution** (mol/mL)			
Standard Deviation, **mols CO_2 / mL solution**			
%RSD			

Table 2: Mass of CO_2 Captured with Different Materials

	DI Water	0.5 NaOH	{Insert DES here}
Average moles CO_2 captured / mL solution (mol/mL)	0.00267		
Standard Deviation, mols CO_2 / mL solution	0.000327		
% RSD	19.6%		

Figure 1: Comparison of CO_2 Capture Capability of Different Materials

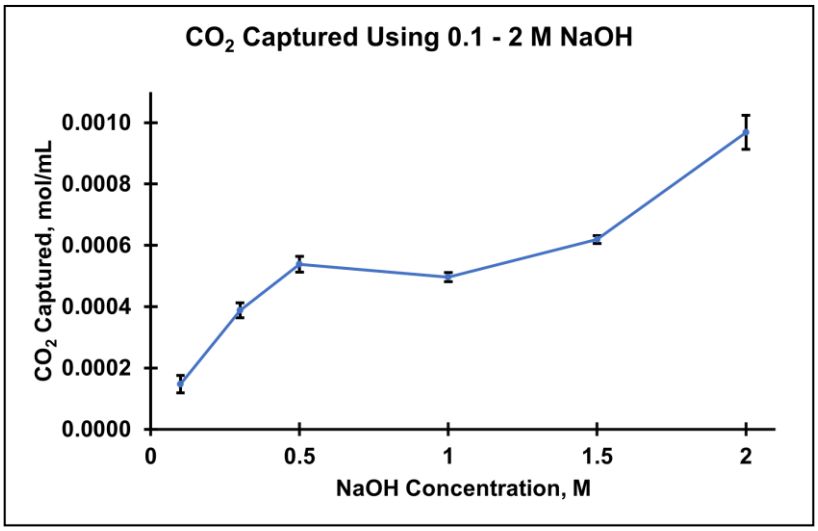
{Insert the Figure from the Excel Template}

Post-Lab Questions:

1. Based on your %RSD and your observations, how repeatable is your experiment? How does this compare to your data from last week?

2. Which of the testing materials was the most effective at capturing CO_2? Use your data from Weeks 1 & 2 and the previously obtained data for the DI water for comparison. Was your hypothesis from the pre-lab correct? Why or why not?

3. Below is a figure showing the amount of CO_2 captured at different concentrations of NaOH. What concentration of NaOH would you estimate has the equivalent CO_2 capture capability as your DES?



4. If you were to continue this research, what do you think you could try next? Develop a new research question that future students could answer, and then write a few sentences about how you would test this in the lab. Think about what other variables could be changed… materials, conditions like temperature, the apparatus, etc. A research question should be specific, measurable, and complex (not a yes or no question).

   Example: "How do deep eutectic solvents compare with sodium hydroxide for effectiveness in carbon capture?"

Pre-Lab Assignment:

(1) Here are the 12 principles of green chemistry: https://www.compoundchem.com/2015/09/24/green-chemistry/ What are two principles being addressed in this 3-week laboratory project? Briefly explain your response.

(2) Based on the SDS for 0.5 M NaOH and the principles of green chemistry, what are some advantages and disadvantages to using NaOH for carbon capture?

(3) Making a hypothesis: Which material (water, 0.5 M NaOH, reline, ethaline, or glyceline) do you think will be the most effective at capturing CO_2 and why? It may be helpful to reference the information in Table 1. Write your answer and justification in complete sentences.

(4) Consider the two sets of data provided below. Which one would be considered more precise? In complete sentences, justify your answer.

Table 2: Sample data from carbon capture with 0.3 M NaOH and 0.1 M EDA

Test Material A	**Initial Mass (g)**	**Final Mass (g)**	**CO_2 Captured (g)**	**Test Material B**	**Initial Mass (g)**	**Final Mass (g)**	**CO_2 Captured (g)**
0.3 M NaOH Trial 1	31.2722	31.3093	0.0371	0.1 M EDA Trial 1	30.5654	30.5775	0.0121
0.3 M NaOH Trial 2	30.6079	30.6399	0.032	0.1 M EDA Trial 2	31.2222	31.2388	0.0166
0.3 M NaOH Trial 3	31.2756	31.309	0.0334	0.1 M EDA Trial 3	30.5654	30.5813	0.0159
		Avg	0.03416667			**Avg**	0.01486667
		Std Dev	0.002151			**Std Dev**	0.001977
		%RSD	6.2970%			**%RSD**	13.2988%

(5) Due to their high viscosity, the DESs are difficult to measure by volume.

DES	Density
Choline Chloride + Urea (Reline)	1.188 g/mL
Choline Chloride + Ethylene Glycol (Ethaline)	1.313 g/mL
Choline Chloride + Glycerol (Glyceline)	1.192 g/mL

Calculate how many grams of the DES you prepared last week would need to be weighed out to be equivalent to 5 mL. Show your calculation.

References:

(1) US EPA, O. *Basics of Green Chemistry*. Environmental Protection Agency. https://www.epa.gov/greenchemistry/basics-green-chemistry (accessed 2021-11-06).

(2) Smith, E. L.; Abbott, A. P.; Ryder, K. S. Deep Eutectic Solvents (DESs) and Their Applications. *Chem. Rev.* **2014**, *114* (21), 11060–11082. https://doi.org/10.1021/cr300162p.

(3) Vázquez, G.; Alvarez, E.; Varela, R.; Cancela, A.; Navaza, J. M. Density and Viscosity of Aqueous Solutions of Sodium Dithionite, Sodium Hydroxide, Sodium Dithionite + Sucrose, and Sodium Dithionite + Sodium Hydroxide + Sucrose from 25 °C to 40 °C. *J. Chem. Eng. Data* **1996**, *41* (2), 244–248. https://doi.org/10.1021/je950243k.

(4) Ibrahim, R. K.; Hayyan, M.; AlSaadi, M. A.; Ibrahim, S.; Hayyan, A.; Hashim, M. A. Physical Properties of Ethylene Glycol-Based Deep Eutectic Solvents. *Journal of Molecular Liquids* **2019**, *276*, 794–800. https://doi.org/10.1016/j.molliq.2018.12.032.

(5) Manurung, R.; Simanjuntak, G.; Perez, R.; Syahputra, A.; Alhamdi, M.; Siregar, H.; Zuhri, R. Production of Choline Chloride-Based Deep Eutectic Solvent with Hydrogen Bond Donor D-Glucose and Ethylene Glycol. *IOP Conference Series: Materials Science and Engineering* **2019**, *505*, 012134. https://doi.org/10.1088/1757-899X/505/1/012134.

(6) Mjalli, F. S.; Ahmed, O. U. Physical Properties and Intermolecular Interaction of Eutectic Solvents Binary Mixtures: Reline and Ethaline: Reline-Ethaline. *Asia-Pac. J. Chem. Eng.* **2016**, *11* (4), 549–557. https://doi.org/10.1002/apj.1978.

(7) Abbott, A. P.; Harris, R. C.; Ryder, K. S.; D'Agostino, C.; Gladden, L. F.; Mantle, M. D. Glycerol Eutectics as Sustainable Solvent Systems. *Green Chem.* **2011**, *13* (1), 82–90. https://doi.org/10.1039/C0GC00395F.

Additional resources:

- Basics of green chemistry: https://www.epa.gov/greenchemistry/basics-green-chemistry
- Video about deep eutectic solvents to make the paper industry greener. "Introducing: PROVIDES - Deep Eutectic Solvents": https://www.youtube.com/watch?v=2oDgVDhUXAY

CHEMISTRY 142 – Chemical Principles II Laboratory
Lab 2: Testing Materials for CO_2 Capture
Week 3 of 3

Project Goal. In this Project-Based lab, you will evaluate the effectiveness of different test materials in "capturing" carbon dioxide (CO_2).

Oral Presentation. Science does not exist in a vacuum. For it to begin to make a difference in the real world, it is important to know how to communicate scientific discoveries to colleagues, other people in STEM, and to the wider public. To practice that, you and your lab partner will use the lab period to prepare a 5-minute PowerPoint presentation to present to the class on the same day. There will not be an experimental lab today. Use your answers to the pre-lab questions as a guide, as well as the pre- and post-lab questions from the previous two weeks.

Your PowerPoint should consist of 4 slides, including the title slide. Use the template posted in BlackBoard.

- **Title Slide –** Include a title and the names of you and your partner(s).
- **Goal** – What is the purpose of your research experiment? State your hypothesis.
- **Results –** Talk about your observations during the experiment and any key data points. Include a graph summarizing your results.
- **Conclusion and Reflection**– Was your hypothesis correct? Why or why not? What is a piece of advice you would give to future CHEM142L students working on this project? What could a future class investigate further?

Table 1: (for use in class)

	DI Water	Material 1	Material 2
Average moles CO2 captured / mL solution (mol/mL)	0.00267		
Standard Deviation / mL solution	0.000327		

Pre-Lab Assignment:

1. What are some skills you have learned or improved upon as part of this lab? Did your overall precision improve from Week 1 to Week 2?

References:

(1) Smith, E. L.; Abbott, A. P.; Ryder, K. S. Deep Eutectic Solvents (DESs) and Their Applications. *Chem. Rev.* **2014**, *114* (21), 11060–11082. https://doi.org/10.1021/cr300162p.

Grading Rubric for Final Carbon Capture Lab Student Presentations: _____/ 120 pts

	60%: Beginning	80%: Meets Expectations	100%: Exceeds Expectations
Research Goals & Hypothesis (20 pts)	□ There is little to no discussion of the research goals and hypothesis.	□ The research goal and hypothesis are stated. □ The connection to carbon capture or the scientific justification for the hypothesis are unclear.	□ The research goal is clearly stated and connects to evaluating materials for carbon capture. □ The research hypothesis and justification is clearly stated.
Experimental Results (30 pts)	□ There is little to no presentation of experimental results.	□ Provides a figure summarizing the performance of two carbon capture materials, but the legibility or labeling of the figure needs improvement or there are errors in the interpretation of the data.	□ Provides a figure summarizing the performance of two carbon capture materials. Figure is legible and axes are clearly labeled. □ 1-2 bullet-points are provided that clearly explain the main takeaways of the experimental data.
Conclusions & Reflections (30 pts)	□ There is little to no discussion of the hypothesis, recommendations, or future ideas.	□ The analysis of the hypothesis contains errors, or the recommendation or idea for future research has been omitted.	□ The hypothesis is clearly confirmed or refuted based on the experimental data. □ One recommendation for future 142L students is provided. □ One idea for future research is provided.
PowerPoint Slides (20 pts)			□ The completed PowerPoint slides are uploaded to Blackboard as a PPT or PDF file.
In-Class Presentation (20 pts)			□ Both partners contribute to the in-class oral presentation.

Post-Survey

1. What is your current class year?
    a. Freshman
    b. Sophomore
    c. Junior
    d. Senior
    e. Graduate Student
    f. Non-degree/other: _____

2. What is your race/ethnicity? Check all that apply.
    a. American Indian/Alaskan Native
    b. Asian descent
    c. Bi-racial/Multi-racial
    d. Black/African descent
    e. Black/Caribbean descent
    f. Hispanic/Latinx
    g. Middle Eastern descent
    h. Native Hawaiian or Other Pacific Islander
    i. White/European descent
    j. Other: _____

3. What is your gender identity? Check all that apply.
    a. Female
    b. Male
    c. Transgender
    d. Non-binary
    e. Other: _____

4. How confident are you that your chemistry courses will prepare you for your future career?
    a. Very confident; My chemistry courses will be critical in preparing me for my future career.
    b. Moderately confident; My chemistry courses will be useful in preparing me for my future career.
    c. Slightly confident; I may be able to apply a few things that I have learned during my chemistry courses to my future career.

d. Not confident; My chemistry courses are completely irrelevant to my future career.

5. How confident are you that your chemistry training will prepare you to understand and solve global problems?
    a. Very confident; My chemistry training will help me contribute to solving global problems.
    b. Moderately confident; My chemistry training will help me identify potential solutions to global problems.
    c. Slightly confident; My chemistry training will help me learn more about global problems.
    d. Not confident; My chemistry training will not be useful in preparing me to understand or solve global problems.

6. How confident are you in your ability to write a research question?
    a. Very confident; I know what a research question is, and I can develop my own answerable question.
    b. Moderately confident; I know what a research question is, but I'm not sure how to develop my own.
    c. Slightly confident; I have heard of a research question, but I don't have a complete understanding of the term.
    d. Not confident; I am unfamiliar with the term "research question."

7. How confident are you in your ability to write a hypothesis?
    a. Very confident; I know what a hypothesis is, and I can develop my own testable hypothesis.
    b. Moderately confident; I know what a hypothesis is, but I'm not sure how to develop my own hypothesis.
    c. Slightly confident; I have heard of a hypothesis, but I don't have a complete understanding of the term.
    d. Not confident; I am unfamiliar with the term "hypothesis."

8. How confident are you in your ability to perform dimensional analysis calculations (e.g., convert between units or physical quantities such as moles --> grams --> milliliters)?
    a. Very confident; I could do it right now.
    b. Moderately confident; After a bit of thought, I could do it.
    c. Slightly confident; I could do it with some help.
    d. Not confident; I have no idea how to do that.

9. How confident are you in your ability to determine the accuracy and precision of a measurement?

a. Very confident; I could calculate values representing both the accuracy and precision of a measurement, and interpret these numbers.
b. Moderately confident; I could qualitatively estimate the accuracy and precision of a measurement, but I couldn't quantitatively calculate these values.
c. Slightly confident; I have heard of accuracy and precision, but I don't know the difference between them.
d. Not confident; I am unfamiliar with accuracy and precision.

10. How confident are you in your ability to communicate scientific findings/data through oral presentation?
    a. Very confident; I am comfortable speaking in front of others and can communicate clearly, professionally, and effectively, using data to back my claims in an organized way.
    b. Moderately confident; I am okay with speaking in front of others and can communicate somewhat effectively, using data to back my claims.
    c. Slightly confident; I am somewhat uncomfortable speaking in front of others, but I can get the idea across.
    d. Not confident; I dread speaking in front of others and can't communicate clearly or effectively yet.

11. How confident are you in your ability to understand scientific media (e.g., social media posts, scientific magazine articles, science-related videos, etc.)?
    a. Very confident; I can understand and evaluate it and am able to critique processes that are talked about, thinking about experiment limitations.
    b. Moderately confident; I mostly understand it and can identify the main point. In a study, I can identify the purpose and get the gist of the process used.
    c. Slightly confident; I could do it with some help.
    d. Not confident; I am not sure how to do that.

12. How much do you know about current environmental issues surrounding climate change?
    a. I am knowledgeable about environmental issues surrounding climate change, and I feel very comfortable contributing to a discussion about them.
    b. I know of a few environmental issues surrounding climate change, and I could have some conversation on that topic.
    c. I have heard of some environmental issues surrounding climate change but don't know what they are exactly.
    d. I don't know much at all about environmental issues surrounding climate change.

13. How familiar are you with the topic of green chemistry?

a. I understand the principles of green chemistry and could apply this thinking to real-life scientific problems.
b. I know the principles of green chemistry, but I am not sure how to apply them.
c. I have heard of green chemistry, but I don't know what it is exactly.
d. I have never heard of green chemistry.

14. How invested did you feel in the project?
    a. So invested that I want to learn more about the topic on my own.
    b. I was motivated to participate as I was asked to.
    c. I was invested enough to do the bare minimum.
    d. I couldn't care less.

15. How relevant do you feel that this project was to real-world issues?
    a. Very relevant
    b. Moderately relevant
    c. Slightly relevant
    d. Not relevant

16. How relevant was this project to your personal interests and/or experience?
    a. Very relevant
    b. Moderately relevant
    c. Slightly relevant
    d. Not relevant

17. How engaging was the time spent in class?
    a. Very engaging
    b. Somewhat engaging
    c. Not very engaging
    d. Not engaging at all

18. How engaging was the pre- and post-lab work?
    a. Very engaging
    b. Somewhat engaging
    c. Not very engaging
    d. Not engaging at all

19. How useful was the pre- and post-lab work in preparing you for in-class discussions?
    a. Very useful
    b. Somewhat useful
    c. They had no effect on my preparation.

d. They made me more confused than before.

20. What is something future students could try?

_____
_____
_____

21. What was the most confusing or frustrating part of the lab project? Why?

_____
_____
_____

22. What part of the lab project was the most fulfilling or interesting to you? Why?

_____
_____
_____

Pre-Survey

1. What is your current class year?
    a. Freshman
    b. Sophomore
    c. Junior
    d. Senior
    e. Graduate Student
    f. Non-degree/other: _____

2. What is your race/ethnicity? Check all that apply.
    a. American Indian/Alaskan Native
    b. Asian descent
    c. Bi-racial/Multi-racial
    d. Black/African descent
    e. Black/Caribbean descent
    f. Hispanic/Latinx
    g. Middle Eastern descent
    h. Native Hawaiian or Other Pacific Islander
    i. White/European descent
    j. Other: _____

3. What is your gender identity? Check all that apply.
    a. Female
    b. Male
    c. Transgender
    d. Non-binary
    e. Other: _____

4. What is your academic major (or majors)? _____

5. What are your career goals? _____

6. What is your general attitude toward taking chemistry courses?
    a. Very positive; I enjoy taking chemistry courses, and I intend to seek out additional chemistry courses beyond my requirements.
    b. Somewhat positive; I enjoy my required chemistry courses, but I will not seek out additional chemistry courses.
    c. Neutral; I do not have positive or negative feelings toward my required chemistry courses.

d. Somewhat negative; I do not enjoy my required chemistry courses, and I have slight feelings of apprehension toward taking these courses.
e. Very negative; I do not enjoy my required chemistry courses, and I have strong feelings of apprehension toward taking these courses.

7. How confident are you that your chemistry courses will prepare you for your future career?
    a. Very confident; My chemistry courses will be critical in preparing me for my future career.
    b. Moderately confident; My chemistry courses will be useful in preparing me for my future career.
    c. Slightly confident; I may be able to apply a few things that I have learned during my chemistry courses to my future career.
    d. Not confident; My chemistry courses are completely irrelevant to my future career.

8. How confident are you that your chemistry training will prepare you to understand and solve global problems?
    a. Very confident; My chemistry training will help me contribute to solving global problems.
    b. Moderately confident; My chemistry training will help me identify potential solutions to global problems.
    c. Slightly confident; My chemistry training will help me learn more about global problems.
    d. Not confident; My chemistry training will not be useful in preparing me to understand or solve global problems.

9. How confident are you in your ability to write a research question?
    a. Very confident; I know what a research question is, and I can develop my own answerable question.
    b. Moderately confident; I know what a research question is, but I'm not sure how to develop my own.
    c. Slightly confident; I have heard of a research question, but I don't have a complete understanding of the term.
    d. Not confident; I am unfamiliar with the term "research question."

10. How confident are you in your ability to write a hypothesis?
    a. Very confident; I know what a hypothesis is, and I can develop my own testable hypothesis.
    b. Moderately confident; I know what a hypothesis is, but I'm not sure how to develop my own hypothesis.

c. Slightly confident; I have heard of a hypothesis, but I don't have a complete understanding of the term.
d. Not confident; I am unfamiliar with the term "hypothesis."

11. How confident are you in your ability to perform dimensional analysis calculations (e.g., convert between units or physical quantities such as moles --> grams --> milliliters)?
    a. Very confident; I could do it right now.
    b. Moderately confident; After a bit of thought, I could do it.
    c. Slightly confident; I could do it with some help.
    d. Not confident; I have no idea how to do that.

12. How confident are you in your ability to determine the accuracy and precision of a measurement?
    a. Very confident; I could calculate values representing both the accuracy and precision of a measurement, and interpret these numbers.
    b. Moderately confident; I could qualitatively estimate the accuracy and precision of a measurement, but I couldn't quantitatively calculate these values.
    c. Slightly confident; I have heard of accuracy and precision, but I don't know the difference between them.
    d. Not confident; I am unfamiliar with accuracy and precision.

13. How confident are you in your ability to communicate scientific findings/data through oral presentation?
    a. Very confident; I am comfortable speaking in front of others and can communicate clearly, professionally, and effectively, using data to back my claims in an organized way.
    b. Moderately confident; I am okay with speaking in front of others and can communicate somewhat effectively, using data to back my claims.
    c. Slightly confident; I am somewhat uncomfortable speaking in front of others, but I can get the idea across.
    d. Not confident; I dread speaking in front of others and can't communicate clearly or effectively yet.

14. How confident are you in your ability to understand scientific media (e.g., social media posts, scientific magazine articles, science-related videos, etc.)?
    a. Very confident; I can understand and evaluate it and am able to critique processes that are talked about, thinking about experiment limitations.
    b. Moderately confident; I mostly understand it and can identify the main point. In a study, I can identify the purpose and get the gist of the process used.
    c. Slightly confident; I could do it with some help.
    d. Not confident; I am not sure how to do that.

15. How much do you know about current environmental issues surrounding climate change?
    a. I am knowledgeable about environmental issues surrounding climate change, and I feel very comfortable contributing to a discussion about them.
    b. I know of a few environmental issues surrounding climate change, and I could have some conversation on that topic.
    c. I have heard of some environmental issues surrounding climate change but don't know what they are exactly.
    d. I don't know much at all about environmental issues surrounding climate change.

16. How familiar are you with the topic of green chemistry?
    a. I understand the principles of green chemistry and could apply this thinking to real-life scientific problems.
    b. I know the principles of green chemistry, but I am not sure how to apply them.
    c. I have heard of green chemistry, but I don't know what it is exactly.
    d. I have never heard of green chemistry.