Sample Plan for Easy, Inexpensive, Safe, and Relevant Hands-On, At-Home Wet Organic Chemistry Laboratory Activities

Supporting Information – Experiment Handouts, Safety Contract, Bin Contents, Instructor Notes, and Student Surveys

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The experiment handouts, safety contract, and supply bin contents list have been slightly tweaked since their use in the 2020-2021 organic chemistry and are provided here in their refined form for other instructors to use for their courses.

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Laboratory Safety Contract

Course: XXXXSemester(s): XXXXAcademic Year: XXXXInstructor: XXXXInstructor Contact: XXXXSchool: XXXXEmergencies: call 911 or a 24-hour Poison Control Center Hotline at 1-800-222-1222

Purpose: This contract outlines expectations surrounding behavior, attire, safety practices, and other safety-related aspects of conducting laboratory work. Submission of this signed form is **required** to engage in any laboratory activity. Safety is the top priority and non-negotiable.

- Experimental chemistry presents many hazards and risks which can, either through neglect, carelessness, ignorance, environmental conditions, equipment failure/malfunction, chemical instability/incompatibility/contamination, human mistake, or accident, lead to various forms of property damage, bodily injury (acute/chronic, temporary/permanent, mental/physical, etc.), and even death.
 - Although the lab activities for this course have deliberately been designed to be as low-risk as possible, the inherently hazardous nature of chemistry lab work makes this safety contract necessary.
- Failure to follow safety practices is a serious concern in the chemistry laboratory and will result in corrective, academic, and/or disciplinary actions in order to safeguard the wellbeing of students, instructor, other individuals, property, and the environment.
 - Corrective warnings will usually be given for first-time offenses, but, due to the seriousness of lab safety, a first-time offense may also incur academic and/or disciplinary action depending on the situation.
 - Conducting labs is a privilege, not a right. The instructor and/or parent/guardian may revoke a student's lab privileges at any time for any safety concern.

Instructions: Read this form thoroughly with your parent/guardian. Fill out any relevant personal information, and sign. Both student and parent/guardian must sign to indicate that you will abide by all safety rules throughout the course. Submit the signed version before the first lab.

Due: by start of class on XXXX

• Students may not participate in any lab activities pending instructor receipt of a completed, signed safety contract.

Safety Rules: The following outlines standard safety policies associated with the chemistry laboratory. Additional policies may be issued during the course either verbally or in writing (including online equivalents).

Behavior

- 1. Read all instructions fully before beginning lab activities. Follow all directions. Demonstrate responsible behavior. Use common sense.
- 2. Be alert and focused. Never work sleepy or distracted. Pay attention to your work, your environment, and the actions of those around you. Always work in a well-lit area.
- 3. Do not engage in horseplay, practical jokes, or pranks. Do not engage in any distracting, alarming, physically vigorous, or dangerous behavior (ex: yelling, running).

- 4. Manage your time efficiently, but do not rush or run.
- 5. Do not panic. Keep calm in all situations.
- 6. Do not work alone. Never touch, interact, or be around lab items without a supervisor. Another person should always be in the vicinity during a lab activity in case you require emergency assistance.
- 7. Do not use or manipulate any lab items without explicit instructor permission.
- 8. Immediately correct behavior when prompted by the instructor, another adult, or an informed peer.
- 9. React to anything which seems odd (ex: solution becoming discolored, noticing an odor, a container is swelling, hissing sound from somewhere, a general feeling that something is wrong, etc.) by reporting immediately to the instructor.

Personal Protective Equipment (PPE) and the Body

- 10. Wear chemical splash goggles (not just safety glasses or prescription glasses), gloves, and other PPE when instructed.
- 11. Wear prescription glasses instead of contact lenses when medically feasible and especially when handling or generating any gaseous, volatile, or other substance harmful to eyes.
- 12. Tie back hair. Hair should neither interfere with vision nor droop into chemicals or flames.
- 13. Avoid loose or baggy clothing, flowy sleeves, short shorts (anything above the thigh midway point), skirts, and dangling jewelry (ex: earrings, necklaces, bracelets).
- 14. Wear closed-toe, closed-top, closed-heel shoes with traction (ex: sneakers).
- 15. Do not eat, drink, or chew gum. Do not bring any consumable items into a lab.
- 16. Do not apply cosmetics, lotions, etc. in lab (you may arrive with cosmetics on). Do not touch contact lenses or your face.
- 17. Remove gloves and/or wash your hands before leaving the lab. Do not touch doorknobs, light switches, or other common touchpoints with contaminated gloves or hands.
- 18. Alert the instructor of any allergies, disabilities, and other medical conditions which may either manifest during labs, impair your ability to conduct lab work, and/or affect your ability to engage in safety behaviors.
- 19. Wash skin and/or eyes with fast-running, cold water for several minutes immediately after direct contact with any chemical or if any tingling/itching/burning/reddening is noticed. Remove contact lenses during eye washing, and continue rinsing eyes. Notify instructor of the incident while washing. Help another person if they need to rinse off, but do not touch them.
- 20. If you ingest a chemical (which you never should), immediately leave the lab to drink water and eat food (with supervision) after alerting the instructor. Do not induce vomiting unless instructed by a Poison Control Center.

Safety Equipment and the Physical Environment

- 21. Always have the ability to call 911.
- 22. Know the location of fire suppression equipment (ex: fire extinguisher, fire blanket), first aid equipment (ex: sink, eye wash, safety shower), spill and breakage management supplies (ex: paper towels, dustpan), fire alarms, an emergency phone, and the fire exit route.
- 23. If a fire or other extremely hazardous situation occurs, handle the situation if it is within the limits of your abilities. If not, leave the area immediately, and alert others (during your

exit, close doors/windows and pull a fire alarm if it is a fire). Never attempt to fight a fire which is larger than the size of a small trashcan unless it is between you and your only exit.

- 24. Keep walkways clear. No backpacks or other non-lab items are permitted in the lab. Keep chairs and other items orderly and out of the way. Do not congregate around or congest a walkway or doorway.
- 25. Work in a ventilated area. Use a fume hood, kitchen vent, opened window/door, or outdoor location when directed.
- 26. Keep your work area clean, organized, tidy, and clear of extraneous materials. Label all containers with chemicals in a clear, legible, and unambiguous manner to identity the chemical, date, and yourself.
- 27. Never leave anything near the edge of a table or where it could be easily knocked over.
- 28. Store your lab supply bin in a cool location, preferably also dark and dry.
- 29. Notify the instructor of any potentially unsafe conditions.

Handling Chemicals and Equipment

- 30. Consider all chemicals and equipment as potentially hazardous, especially if you do not know what its hazards are.
- 31. Do not use chemicals or equipment for unauthorized activities or experiments. Return or dispose of any unused supplies as instructed.
- 32. Label all chemicals. Do not handle chemicals which are not labeled or if you can't read/understand the label. Doublecheck all labels, safety information, and instructions before use.
- 33. Do not use very old chemicals or contaminated chemicals without instructor approval.
- 34. Do not touch chemicals with bare skin.
- 35. Be extra careful around flames and other heat sources such as hot plates and ovens. Never reach over an open flame. Never leave unattended an open flame, gas-operated equipment, or other equipment/experiment where the heat could "run away".
- 36. Carry sharp objects with the points aimed toward the floor. Carry chemicals and equipment securely in your hands, and never try to juggle multiple items at once.
- 37. Smell chemicals using the wafting technique only. Do not directly inhale.
- 38. Never shake or heat a volatile substance.
- 39. Never expose a flammable or volatile substance to any source of heat. Be aware that areas next to windows can become hotter than you anticipate.
- 40. Do not contaminate stock supplies of chemicals.
- 41. Handle glassware and other equipment delicately. Examine equipment before each use for cracks, cleanliness, and other issues.
- 42. Never suddenly expose hot items to cold and vice versa.
- 43. Carefully make sure glassware is not hot before picking it up or putting it down on a work surface.
- 44. Never combine chemicals unless directed.
- 45. Follow any specific instructions given by instructor about any specific chemical, equipment, technique, procedure, etc.

Spills, Breakages, Waste Disposal, and Clean-Up

46. Dispose of chemicals and equipment in appropriate waste containers depending on classification.

- 47. Never touch broken glass with your hands, either bare or gloved. Use a dustpan to sweep up broken glass, and dispose of in an appropriate glass receptacle. If the glass is contaminated, either clean the glass before disposal or place in an appropriately ventilated receptable. Use a damp paper towel to wipe up remaining micro shards.
- 48. Consult with the instructor about how to appropriately clean up spilled chemicals. Avoid stepping in, touching, or contaminating other materials with spilled chemicals. Always be prepared with paper towels on hand to prevent a liquid spill from migrating.
- 49. Report any accidents, spills, breakages, and any other incidents to the instructor, even if at home and/or already cleaned up. Never leave a spill or breakage unattended to.
- 50. Clean your work area, and wipe down the surface with a wet paper towel at the end of every experiment. Store all supplies neatly in an organized manner in a cool and preferably dark and dry location.

Personal Information: Please report any medical conditions or other situations which may affect your safety, the safety of others, and your ability to execute laboratory work, regardless of whether or not this information is already in school databases.

Colorblindness	Yes / No
• Description:	
• Dyslexia	Yes / No
• Description:	
Visual impairment and prescriptions	
 Contact lenses 	Yes / No
o Glasses	Yes / No
• Other:	
Hand/Fine motor impairment	Yes / No
 Description: 	
Other physical motor impairment	Yes / No
 Description: 	
• Allergies	Yes / No
 Description: 	
• Tendency toward contact dermatitis/skin irritation	No / Sometimes / Frequently
• Do you have a private place to work with ventilation?	Yes / No
• This work location is	
• Does your family know where your lab bin is stored?	Yes / No
• Other:	

• Other:

Signatures:

[Student] By signing below, I attest that...

- □ I have read the above information and agree to follow all rules throughout the course.
- □ I understand and accept the risks involved with chemistry laboratory work. I accept the self-responsibility to adhere to all safety rules and actively work towards safety, especially when working at home during remote learning.

- □ I understand that I may ask for clarification and assistance from the instructor about all safety rules and instructions at any time. I will keep a copy of this contract handy for quick reference.
- □ I understand and accept that failure to follow these rules and any additional instructions given by the instructor may result in academic, corrective, and/or disciplinary action. My parent/guardian may be requested to confiscate lab supplies by the instructor and may also do so on their own initiative if a safety concern manifests.
- □ I promise to inform my instructor of any mishaps, spills, injuries, or other accidents which occur during lab activities, whether during class or outside of class meeting times, so that my instructor can keep track of incidents and respond appropriately.
- I understand that I am responsible for returning my take-home lab supply bin and all its contents at the end of this course, unless instructed to dispose of an item by the instructor. If I do not return the complete kit, I understand I may be charged a replacement fee for missing items.
- □ I release my instructor from liability for damages resulting from freak accidents and/or my failure to pay attention, exercise common sense, and/or follow rules and instructions.
- □ I understand that the provided list of safety rules are curated for this specific course, that more rules may be added later on, and that future chemistry courses may require additional rules depending on the situation, materials handled, and techniques conducted.

Name (Print)

Name (Signature)

Date

[Parent/Guardian] By signing below, I attest that...

- □ I have read through this document with my student. I understand and accept the instructor's rules regarding safety. I am confident that my student understands the safety rules and expectations.
- □ I promise to uphold my responsibility as a familial supervisor of my student's behavior and will do my best to help ensure that my student adheres to all rules.
- □ I know where my student has stored their lab supply bin, and I affirm that it is not easily accessible by younger siblings and pets.
- □ I will confiscate my student's lab supplies if requested by the instructor. I understand that I also have the right to independently confiscate the supplies if I am concerned about safety.
- □ I promise to raise any questions or concerns I have with the instructor directly so that they may respond appropriately to my concerns.
- □ I release the instructor from liability for damages resulting from freak accidents and/or my student's failure to pay attention, exercise common sense, and/or follow rules and instructions.

Name (Print)

Name (Signature)

Date

Relationship to Student

Laboratory Supply Bin Contents List & Required Student Supplies

Course: XXXXSemester(s): XXXXAcademic Year: XXXXInstructor: XXXXInstructor Contact: XXXXSchool: XXXXEmergencies: call 911 or a 24-hour Poison Control Center Hotline at 1-800-222-1222

Keep this contents list in the bin.

WARNING! Contents include flammable and volatile* solvents, fragile glass and other breakable items, acidic and basic solutions, and potential chemical irritants/allergens.

*"volatile" = a material which evaporates easily and has high vapor pressure – does not mean it is inherently explosive or a bomb (common misconception) – don't shake anything that's volatile

Storage & Handling Instructions: Failure to follow instructions will result in your parent/guardian being requested to confiscate your lab supplies. Your parent/guardian may also independently confiscate your supplies at any time if they deem it prudent.

- Do not expose bin to heat. Handle gently. Store in a room-temperature or cooler location (ideally also dark and dry) away from potential access from younger siblings and pets. Notify a parent/guardian of the storage location.
- Do not open any bags, touch anything, or eat anything unless instructed. Just leave the bin alone until instructed.
- Immediately contact your instructor if you are concerned about the contents, any spills or breakages occur, you notice an odd odor, something looks wrong, etc. Call 911 in case of a medical emergency.
- Never use supplies for purposes other than designated class activities.
- Notify your instructor of any relevant allergies, especially latex, plants, and food.
- Follow all safety rules and other instructions issued for each activity.
- Never throw away any used supplies unless instructed to do so. Hold on to everything.

Contents: organized according to labeled bags

- *No bag*: chemical splash goggles (if you didn't say that you already have one) and a capacitive computer stylus
- Not included: a camera or the model kit listed as required student materials on the syllabus

Note: You'll need a way to take photos during lab activities to upload images of your work for online submission. You'll also be requested to provide some supplies yourself for each lab.

Bag	Items	Safety
Balloons (Lab 1)	 2 x latex balloons Plastic 1 mL pipette 2 x toothpicks ~ 1 mL hexanes in orange plastic Eppendorf vial You'll need to bring * Pro balloon tying skills * A little bit of water 	 Latex is a potential ALLERGEN. Notify your instructor if you have a latex allergy, and don't touch the balloons. Hexanes are FLAMMABLE and VOLATILE. Handle the vial gently; it will be brittle and easy to damage due to the hexanes contained within. If the vial breaks or you spill the hexanes, no biggie; the hexanes will evaporate quickly and pose no significant health concerns in such a small amount.
Hydrogels (Lab 2)	 1.5 mL ~ 1 M aqueous HCl in yellow plastic Eppendorf vial 1.5 mL ~ 1 M aqueous NaOH in green Eppendorf vial 3 x 1-Day Acuvue Moist contact lenses 2 x toothpicks You'll need to bring A ruler (preferably measuring cm) A piece of white paper A way to tell time (minutes) 	 HCl is an ACID, and NaOH is a BASE. Rinse off skin with cool water if you get it on you. Rinse out your eyes immediately if you get it in your eyes. Wipe off surfaces with a damp paper towel.
Essential Oil Distillation (Lab 4)	 1 L glass canning jar with hole (~ 0.25 in. diameter) in center of lid Thin strand of ~ 1 ft of green wire Loose-woven fabric (~ 6-8 in. x 6-8 in.) Empty 1.5 mL clear plastic Eppendorf vial Small clear plastic cup (~ 1 in. x 2.5 in., condiment cup size) 1 mL plastic pipette ~ 14 in. clear vinyl tubing (0.25 in. outer diameter; 0.170 in. inner diameter) You'll need to bring * Water and ice (smaller pieces are more convenient) * Two cups (to hold ice and to pour water, respectively) * A pan which can hold water while also holding the jar in it (placed in the water) and be heated on the stove safely * A stovetop or other way to boil water using the pan 	 The canning jar is BREAKABLE GLASS. If broken, do not pick up the pieces with bare hands. Use a dustpan and broom to dispose of. Wipe the cleared area with a wet paper towel to safely remove small glass remnants (you might not be able to see them easily). Plants are potential ALLERGENS. Make sure you and your household are not allergic to any plant you handle. Ovens are potential BURN and FIRE HAZARDS. Never leave unattended. Exercise common sense and care. Treat any burns immediately with cool water; you may require further medical treatment if it's a bad burn. Knives are SHARP OBJECTS. Exercise common sense and care. Treat any cuts by immediately cleaning, applying pressure to stop the bleeding, and bandaging; you may require further medical treatment if it's a deep cut.

	 Oven mitts or some other way to insulate your hands from a heat source (ex: a kitchen towel) At <i>least</i> 8 inches of a fragrant plant's stem with leaves (ex: rosemary) or a couple handfuls of fragrant petals – best if the cuttings/petals are fresh – the more plant material, the easier the lab Knife for chopping ~ 1 tablespoon salt A chair to sit on A kitchen towel for cleaning up any spilled water 	
Candle Magic (Lab 5)	 2 x birthday candles Small plastic Petri dish and cover (~ 2 in. diameter) Glass test tube (13 mm x 100 mm), wrapped in paper towel Copper wire (~ 6 in.) You'll need to bring Water A way to safely light candles a few times Oven mitts or small towel or small tongs or large pliers or some other way to insulate your hands from a heat source while holding the test tube 	 The test tube is FRAGILE GLASS. If broken, do not pick up the pieces with bare hands. Use a dustpan and broom to dispose of. Wipe the cleared area with a wet paper towel to safely remove small glass remnants (you might not be able to see them easily). Flames and hot wax are potential BURN and FIRE HAZARDS. Never leave unattended or place near flammable objects. Exercise common sense and care. Do not operate a flame underneath a smoke detector, or you may set off a nuisance alarm. Treat any burns immediately with cool water; you may require further medical treatment if it's a bad burn.
Extraction (Lab 6)	 ~ 0.75 mL ethyl acetate (EtOAc) in clear plastic Eppendorf vial, with toothpick taped to vial 2 x empty 1.5 mL clear plastic Eppendorf vials Plastic red cup 5 x 1 mL plastic pipettes ~ 5 x coffee filters You'll need to bring * Water * Scissors * Spoon * ¹/₄ cup ground cinnamon powder 	

	 ½ cup 70 % (or higher) isopropanol (aka rubbing alcohol – higher percentage should be easier) Timer (phone timer or clock is fine) A pan which can hold water while also holding a glass in it (placed in the water) and be heated on the stove safely A stovetop or other way to boil water using the pan Oven mitts or some other way to insulate your hands from a heat source (ex: a kitchen towel) Glass drinking cup Flat glass pan (like a baking pan)
Glowmatography (Lab 7)	 2 x nitrile gloves (size: large) 2 x non-toxic glowsticks Be careful not to activate them prematurely aka do not bend at all. 3 x ~ 3-inch chalk, wrapped in paper towel 2 x plastic 1 mL pipettes Small clear plastic cup (condiment cup size) 2 x small clear plastic lids (from condiment cups) 60 mL Nalgene dropper bottle, partially filled with ~ 40 mL denatured 95 % ethanol You'll need to bring Scissors A couple paper towels A pencil (soft tips are better) A reavid ark room A flashlight (ex: smartphone light) You. Handle with gloves to be extra safe to prevent skin absorption. If it spills, it will evaporate, but you should go ahead and wipe it up with a paper towel just to get rid of it expediently. Ethanol may also mar wood stains or other surfaces covered in finishes or lacquers. It will not harm granite or other common kitchen stone countertops.
M&M TLC (Lab 8)	 3 x red, orange, yellow, green, blue, and brown M&Ms, each M&Ms are a rare ALLERGEN but are often processed in facilities that process tree nuts. Notify your instructor if you

	 3x qualitative filter paper (~ 5 in. diameter), wrapped in printer paper to keep clean Avoid touching the filter paper with your fingers as much as possible. Keep clean. Handle around the edges. 5 x toothpicks ~ 0.5 mL yellow food coloring (yellow #5) in yellow plastic Eppendorf vial 	>	 have a relevant food allergy. Do not eat the M&M's. Food coloring is a rare ALLERGEN. Allergies to food coloring are rare, but please notify your instructor if you have such an allergy. Food coloring might stain a surface if you spill it. Clean up promptly with a damp paper towel.
	 You'll need to bring Scissors Tape Plain graphite pencil (soft tips are better) A ruler (preferably measuring cm) A small, flat-bottomed bowl (<i>must</i> be flat) or short, wide cup Water A "pinch" of salt (< ¼ teaspoon) 		
Column Chromatography (Lab 9)	 A pinch of salt (< ¼ teaspoon) 2 x 1 mL plastic pipettes Cotton ball Bag of corn starch (~ 1/3 cup) 10 mL plastic syringe with small drilled hole between the 6 mL and 7 mL gradations Syringe plunger ~ 0.2 mL green food coloring in green Eppendorf vial Paper towel 2 x coffee stirrers Thin popsicle stick 2 x paint trays You'll need to bring Plain graphite pencil (blunt tip is better than sharp/pointy) A marker Bottle of 70 % (or higher) isopropanol (aka rubbing alcohol) Several colored plant leaves, flowers, vegetable roots, etc. (your choice) – and a way to mash up the plant material 	A	Food coloring is a rare ALLERGEN. Allergies to food coloring are rare, but please notify your instructor if you have such an allergy. Food coloring might stain a surface if you spill it. Clean up promptly with a damp paper towel. Isopropanol is FLAMMABLE and VOLATILE. Wipe it up with a paper towel if it spills. Isopropanol may mar wood stains or other surfaces covered in finishes or lacquers. It will not harm granite or other common kitchen stone countertops. Plants are potential ALLERGENS. Make sure you and your household are not allergic to any plant you handle.

# Activity	Stockroom/Class Supplies ^a	Out-of-Pocket Supplies b, c	Out-of-Pocket Expenses ^b
1 Unsafe Lab Practical	pipettes, hexanes, vials	balloons, toothpicks	balloons: \$2.99 / pack toothpicks: \$1.59 / 250
2 Hydrogels	HCl, NaOH, vials	toothpicks, contact lenses	lenses: \$43.20/ 36 lenses canning jar: \$25.51 / 12 jars
4 Essential Oil Distillation	vials, pipettes	canning jar, green wire, netting, shot cups, [original] straws, [revised] clear vinyl tubing	netting: \$1.99 / 30-in. x 60-in. paddle wire: \$2.07 / paddle plastic straws: \$0.99 / 100 shot cups: \$1.99 / 25
5 Candle Magic	Petri dishes, test tubes, Cu wire	birthday candles	birthday candles: \$0.99 / pack
6 Extraction	EtOAc, vials, pipettes, gloves, plastic cups	toothpicks, condiment cups, coffee filters	condiment cups (with lids): \$3.00 / 30 coffee filters: \$2.99 / pack
7 Glowmatography	gloves, chalk, pipettes, Nalgene dropper bottles, EtOH	glowsticks, condiment cups	glowsticks: \$5.99 / 36
8 M&M TLC	filter paper, printer paper, vials	M&Ms, toothpicks, yellow #5 food coloring (tartrazine)	yellow food coloring: \$3.89 / 1 fl. oz. M&Ms: \$5.99 / family-sized bag
9 Colum Chromatography	pipettes, cotton balls, syringes, green food coloring, vials, paper towels, coffee stirrers, thin popsicle sticks	corn starch, paint trays	corn starch: \$1.99 / 1 lb. paint trays: \$4.00 / 24 bins (with lids): \$30.00 / 12 Total: \$139.17 / 12 bins = \$11.60 / bin

Bin Cost Estimate for Instructor

^a also: chemical splash goggles were provided to students from the classroom set

^b from the instructor to make an original 12 kits during summer (later resulting in a surplus of kits as well as leftover materials with actual enrollment of 7 students) – excludes supplies the students must purchase

^c contact lenses (1-Day Acuvue Moist) from Walmart; jars and green wire (plant DIY/decor supplies section) from Michaels; glowsticks and netting from Party City; paint trays from Dollar Tree; bins from Target – everything else could be found in the local grocery store

Special Assembly Instructions for Syringe and Canning Jar

Syringe Modification: Drilling the hole in the syringe was necessary to prevent cracking and drawing air up the packed column. After filling the column with corn starch and adding eluent, depressing the plunger into the syringe was necessary to achieve a desirable drip rate; gravity elution was prohibitively slow. However, the author was unable to replicate the reported procedure's method of slowly twisting up the plunger without adverse results. An easy fix to this issue was drilling (with a power drill) a very small hole between the 6 mL and 7 mL mark (with the top of the column around the 4 mL mark). Do not drill any higher than 7 mL, and take great care to not allow the drill bit to touch the other side of the syringe once it breaks through the syringe in making the intended hole. If the moving drill bit touches the other side of the syringe with forward momentum, it will likely crack the syringe and render it unusable (you would be able to visibly see the crack and know to discard the syringe).

After then preparing the column with cotton and corn starch, place over the hole the fleshy part of your thumb of the hand holding the column steady. Use your other hand to add eluent to the 10 mL mark and slowly depress the plunger (note that the chosen eluent must be safe for skin contact – an appropriate example is rubbing alcohol). Apply pressure with your thumb against the drilled hole to prevent the eluent from squirting out of the hole. When the plunger reaches the hole, release your thumb so that pressure can equalize in the column before pulling the plunger back out. If a student has trouble with eluent squirting out of the hole while depressing the plunger, advise them to place some Play-Do or other clay-like substance over the hole before applying pressure with their thumb. The student may then need to use a toothpick to clear the hole out for the pressure to equalize. Repeat for each round.

Alternatively, another article which reported using a plastic syringe for column chromatography instead attached a length of tubing to the syringe tip and then to a second, empty syringe; pulling the depressed plunger slowly out on the second syringe (while the first syringe was securely attached vertically to a ring stand) facilitated elution.[†]

Canning Jar Modification: In making the hole in the jar lid, a micro wire cutter was found to be very effective whereas the classroom scissors were incapable of puncturing the lid. A micro wire cutter was smoothly twisted (while closed) repetitively back and forth against the top center of the lid (face down on a sturdy tabletop which can afford getting a few scratches) for half a minute to create the hole. A straw (~ 0.25 cm outer diameter) was inserted into the hole to test for appropriate fit. If the wire cutter opened up a hole that was too large for the straw, the metal lip protruding from the underside of the lid could be pushed back into the hole to create a snugger fit. Sharp, jagged metal edges protruding from the underside of the hole were cut off with the wire cutter to reduce the odds that a student might cut themselves on a sharp edge.

[†]Miles, D. T.; Wells, W. G. Lab-in-a-Box: A Guide for Remote Laboratory Instruction in an Instrumental Analysis Course. *J. Chem. Educ.* **2020**, *97*(9), 2971-2975. DOI: 10.1021/acs.jchemed.0c00709

#	Wet Activity	Special Hazards	Uncommon Items	Possible Alternatives
1	Unsafe Lab Practical	 i. Allergen (latex, must touch) ii. Flammable (1 mL hexanes) iii. Volatile (1 mL hexanes) iv. Health^a (1 mL hexanes) 	a. Plastic pipette b. Hexanes	 a. Pour hexanes (vs. squirting at balloon with pipette). b. Orange peel^b/limonene/ heptane/toluene^c/WD-40/ spray paint/stain remover/spray adhesive/paint thinner (vs. hex.)
2	Hydrogels	i. Acid (1.5 mL 1 M aq. HCl) ii. Base (1.5 mL 1 M aq. NaOH)	a. HCl b. NaOH c. Contact lenses	 a. Vinegar/AcOH (vs. HCl) b. NaHCO₃ (vs. NaOH) c. Use one lens sequentially, washing in lens solution between vials (vs. using 3 lenses).
4	Essential Oil Distillation	 i. Irritant/Allergen (plant, essential oil) ii. Open flame (if gas stovetop) iii. Glass (jar) iv. Sharp object (knife) 	a. Eppendorf vial	a. Shot cups/ice cube trays/paint trays from column chromatography bag (vs. vials)
5	Candle Magic	i. Open flame (candles, lighter not in kit)ii. Hot wax (candles)iii. Glass (test tube)	a. Plastic petri dish b. Glass test tube	a. Small, nonflammable plate/bowl (vs. Petri dish)b. Thin shot/dessert glass (vs. test tube)
6	Extraction	 i. Open flame (if gas stovetop) ii. Flammable (0.5 mL EtOAc, <i>i</i>-PrOH not in kit) iii. Volatile (0.5 mL EtOAc, <i>i</i>-PrOH not in kit) 	a. EtOAc b. Plastic pipettes	a. Vegetable oil (vs. EtOAc)b. Kitchen basters (vs. pipettes)
7	Glowmatography	 i. Flammable (~40 mL EtOH) ii. Volatile (~40 mL EtOH) iii. Toxic (denat. EtOH, if drunk) iv. Glass (in glowstick, exposed when cut) 	a. Plastic pipettes b. Denat. 95 % EtOH	a. Q-tips (vs. pipettes)b. <i>i</i>-PrOH (vs. EtOH, though poorer elution)
8	M&M TLC	i. Allergen (chocolate, yellow food coloring [rare], if eaten)	a. Filter paper	a. Paper towel/coffee filter (vs. filter paper)
9	Column Chromatography	 i. Irritant/Allergen (plant, green food coloring [rare]) ii. Flammable (<i>i</i>-PrOH not in kit) iii. Volatile (<i>i</i>-PrOH not in kit) 	a. Plastic pipettes b. 10 mL plastic syringe	a. Careful pouring (vs. pipetting eluent)b. Emptied (cut open) marker/highlightertube (vs. syringe)

Special Hazards and Supply Alternatives for Wet Activities

^aHexanes, which are present in the atmosphere and many household products, have been reported as a neurotoxin for cases of chronic, high exposure (i.e. certain industrial settings). The amount suggested here does not reach those levels.[†]

^bOranges, shown in a popular ChemEdX demo^{††}, did not work well for many students in the spring, possible since many balloons are vulcanized.

^cToluene is a regulated substance in several states and can be misused as a recreational inhalant.

[†]EPA. *Hexane. 110-54-3.* <u>https://www.epa.gov/sites/production/files/2016-</u> 09/documents/hexane.pdf (accessed Aug 2020).

^{††}Kuntzleman, T.; Talaski, T.; Schaerer, C. How Does an Orange Peel Pop a Balloon? Chemistry, Of Course!. *ChemEdX* **2015**. <u>https://www.chemedx.org/blog/how-does-orange-peel-pop-balloon-chemistry-course</u> (accessed Mar 2020).

Supplemental Instructor Notes

# Activity	Alterations to Published	Notes
1 Unsafe Lab Practical ⁹⁻¹⁰	 Video: → Filming videos of instructor working in lab instead of physical student walk-through of lab. Balloon Popping: → Using toothpicks instead of bamboo skewers. → Wetting toothpicks with water (mess-free) instead of oil. → Using hexanes instead of toluene (recreational inhalant). → Squirting hexanes using a pipette onto balloon from a couple feet away instead of next to it. 	 Video: → It can be hard to see small details on a video, especially with poor pixels and a lagging computer. Film slowly, and pan in/out. → Students noticed safety problems and good behavior more easily than expected. Balloon Popping: → The hexanes will fully evaporate in the vial within a week or less, depending on environment. → Tell students to be careful not to miss with the hexanes; there isn't much solvent after a few days. → WD-40, stain remover, and spray paint were successful substitutes for students whose hexanes evaporated. → Despite the warnings, students were surprised and jumped when the balloons popped. This is the intended effect to cause self-reflection on calmly dealing with emergencies and surprise in a lab.
2 Hydrogels ¹¹	 → Using stronger acid and base. → Placing contact lenses directly in acidic and basic Eppendorf vials, shaking, and leaving for 10 minutes before removing for comparison instead of placing concurrently in sectional Petri dish. → Using toothpicks to remove lenses from vials and place side-by-side on white paper for measurement with a ruler. 	 → Emphasize keeping track of which lens is which. → Consider adding color to visualize the lenses in solution or keep track of which is which. To do so, dip (<i>just barely</i>) a toothpick into a food coloring vial (from other labs) to obtain a <i>small</i> amount. → Changes in lens size are very slight. Acid contraction can be seen when directly comparing, but basic expansion is harder to see.
3 Modeling	not applicable → Using Darling model kit (required course material, ~ \$25). ¹² → Using MolView (molview.com, free). ¹³	 → Pieces can sometimes be hard to push on and pull off. Grip firmly. Try not to bend bonds. → Direct students' attention to the raised "SP3", "SP2", etc. notations on the pieces. → The model kit comes with a helpful guide which is also free to download on the Darling website. → MolView can also predict dipole moments, energies, and other variables.
4 Essential Oil Distillation ¹⁴	 → Using a glass canning jar as distillation pot instead of Erlenmeyer flask. → Drilling small hole in top of jar lid to allow vapors to rise instead of one-holed rubber adapter. → Using hot water bath on stovetop to heat instead of Bunsen burner. → Using bent straw (later revised to use clear vinyl tubing) to collect steam distillate. → Using Eppendorf vial in ice bath to collect distillate instead of test tube. 	 → Students who maintained heat on low did not collect distillate quickly but did after an hour. All students noted strong scents. Turn up the heat, and keep the ice bath icy. → Students tried fresh rosemary, mint, thyme, and cinnamon sticks. Rosemary and cinnamon gave the most successful student data. → Beware trapped bubbles in the pan rocking the glass jar. Emphasize not leaving the experiment, having oven mitts nearby, and wearing clothes and shoes that protect against small splashes of boiling water jumping out of the saucepan occasionally.

5 Candle Magic ¹⁰	→ Using dripping hot candle wax to secure birthday candle to the Petri dish so it stands safely upright before starting either demo.	 → Warn students of hot wax. It's easy to forget that the wax goes straight down, even if you hold it upside down or angled. → Make sure that the copper is coiled, not just placed above the candle in a straight line. Otherwise, it won't extinguish the flame immediately. → Wait a full minute for the candle to burn before quickly placing the inverted test tube over it and down into the water. If done immediately after lighting the candle, the phenomenon will not be observed.
6 Extraction ¹⁵	 → Allowing students to alter amounts, provided ratios remain constant. → Conducting only a solid-liquid extraction and gravity filtration of a very crude mixture of cinnamaldehyde from cinnamon to demonstrate the concept. Not engaging in subsequent reactions with bleach. 	 → Add water and food coloring (from other labs) to EtOAc vial, and shake to demonstrate immiscibility before conducting cinnamon extraction. → Coffee filter paper can get clogged with cinnamon. Use a toothpick to gently agitate mixture on filter.
7 Glowmatography ¹⁶	 → Directing students to focus on difference between fluorescing and non-fluorescing spots. → Using flashlight on/off in dark room to see separation of non-fluorescing dye. → Using 95 % denatured EtOH instead of 91 % <i>i</i>-PrOH due to perceived improvement in separation for the chalk and glowstick supplies on hand. 	 → The glowsticks used were SuperGlow from Amscan. → Blue was very faint on chalk and faded quickly. Green and purple lasted longer and were more vivid. → Elution can be very uneven. Focus on just distinguishing between fluorescing spots (lower) and non-fluorescing, colored spots (higher). → The original published procedure has very helpful extra information in their SI.
8 M&M TLC ¹⁷ a	 → Testing a couple samples at different loads before running M&M dyes to demonstrate importance of load amounts. → Adding two spots for the yellow food coloring reference spots instead of one (one on each end of the paper). → Not drying sample spots with compressed air (unnecessary). 	 → Ensure the food coloring is "Yellow #5" (tartrazine). → Blue and green can be very faint. Apply these at least twice as much as the other colors. → Add only the <i>tiniest</i> trace of food coloring, or dilute it before loading to prevent overloading. → Run a yellow food coloring spot on both right and left sides of filter paper in case of uneven development. → Printer paper is not a good substitute for filter paper (poor capillary action), but a paper towel can work.
 9 Column a Chromatography 18-20 	 → Using a 10 mL syringe as column support instead of 5 mL. → Drilling small hole at the 7 mL mark to enable pressure equalization. → Covering hole with fleshy part of thumb while compressing plunger to force mobile phase, then release thumb to uncover hole (releasing pressure to avoid cracking and drying column while removing plunger). → Preparing column with tapping and solvent flush to remove air bubbles. → Not using green marker dye, just food coloring (worked better). → Using ≥ 70 % <i>i</i>-PrOH instead of EtOH. 	 → The cotton can be hard to get to stay down in the syringe tip. Wet it with solvent first, then push firmly with a blunt pencil. → Do not place more than 4 mL of corn starch into the column; it's unnecessary and slows elution. → Gravity elution was too slow. Pressure is required. → Green food coloring separated clearly and immediately into blue (elutes first) and yellow. → Repeat the experiment again with other materials after using the food coloring. Try multicolored plant leaves (extract first).
Students wrote their	own procedures for Labs 8 and 9 with instructor guidance, an	iu merenore men own experimental designs varied some

^a Students wrote their own procedures for Labs 8 and 9 with instructor guidance, and therefore their own experimental designs varied some from the ideal procedures on a case-by-case. The instructor notes here are for instructors who provide students with complete procedures.

Lab 1: Unsafe Lab Practical Organic Chemistry (27 points)

Purpose: to (1) highlight standard safe and unsafe practices and (2) foster a detail-oriented habit of constructive critique

Introduction: The chemistry lab is an exciting but dangerous place. On the physical end, there are breakable glass items, moving machine parts, vacuums, flames, hot plates, etc. On the chemical end, there are corrosive, flammable, volatile, light-sensitive, shock-sensitive, water-sensitive, air-sensitive, fuming, acidic, basic, oxidizing, carcinogenic, teratogenic, toxic, etc. materials which take the form of solids, liquids, and gases (often invisible). From the early days of alchemy to the dawn of modern chemistry, laboratory accidents were common and often resulted in eyesight loss, impaired hearing, loss of fingers and other limbs, fires, explosions, etc.

Fortunately, an increased awareness and adherence to safety practices as well as improvements in safety equipment and workplace regulations in modern times have greatly enhanced the safety of the experimental chemist's job. Sacrificing bodily wellness to scientific advancement and exposure to untoward danger is no longer an expectation of the profession. However, occasional incidents remind us of the potential for harm inherent in "wet" techniques (aka handling chemicals) and the need to proactively train practitioners to maintain "safety culture". A crucial part of maintaining safety culture is to indoctrinate introductory students into standard safety practices early in their academic careers in order to safeguard their wellbeing as well as the wellbeing of those around them and avoid property damage. In this lab activity, you will analyze videos of a person working in a lab to draft a list of appropriate and questionable actions they undertake. You will reflect on appropriate behavior you can undertake for future labs as you consider the hazards and risks of various activities.

Due: by XXXX

Safety, Waste Disposal, & Clean-Up:

- 1. Wear goggles during the balloon portion. Gloves are not necessary.
- 2. Be careful when popping the balloon that you don't get hurt. Be prepared for a loud noise.
- 3. Do not involve siblings or other people in your experiment.
- 4. When done, throw out the popped balloon pieces; make sure to get all the pieces up so that a pet or infant doesn't find any later. Hold onto everything else.

Clean-Up Approval Signature (1 point)	Name of Signer:
	Relationship to Student:

Materials: Contact your instructor about alternative materials if you're missing an item.

- Balloon bag (warning: latex allergen)
 - If your hexanes has evaporated, there are alternative items you can try to substitute in (you should only need a few drops). Items which contain hexanes or toluene

should work very well. WD-40, orange/citrus (might/might not work), paint thinner, gasoline, stain remover, spray adhesive, and spray paint are good to try.

Instructions: Conduct the following procedure, complete the table, and answer all questions. Follow all "Safety, Waste Disposal, & Clean-Up" guidelines.

- 1. Read the ACS webpage "Hazards vs. Risks": https://www.acs.org/content/acs/en/chemical-safety/basics/hazard-vs-risk.html.
- 2. Reflect on the most ridiculous injury you or someone you know got, one where you really should have known better. Think about what could have been done to avoid the injury.
- 3. Watch the first video (< 1 min.). Take notes on anything you notice about lab space which seems like a good, safety-oriented idea or bad/questionable idea in terms of safety.
 - Watch the video full-screen and with sound to help notice details.
 - Keep in mind that sometimes the good, safety-oriented aspects are hard to notice because we take them for granted.
- 4. In Table 1, list four unsafe/questionable aspects and four good, safety-oriented aspects about the physical environment from the first video.

Table 1. Safety Observations from Firs	st video (0.5 points/box, 4 points total)
Unsafe/Questionable	Good, Safety-Oriented

Table 1: Safety Observations from First Video (0.5 points/box, 4 points total)

- 5. Watch the second (< 3 min.) and third (< 6 min.) videos (the videos show different angles). Take notes on anything which seems like a good idea or bad idea in terms of safety.
 - Some areas you might choose to focus on are the person's actions (or inactions), the physical set-up of the lab area, attentiveness, cleanliness, attire, obtaining and using supplies, safety equipment, and clean-up.
- 6. In Tables 2 and 3, list four unsafe/questionable aspects and four good, safety-oriented aspects from the second and third videos.

Table 2: Safety Observations from Second Video (0.5 points/box, 4 points total)

Unsafe/Questionable	Good, Safety-Oriented

Unsafe/Questionable	Good, Safety-Oriented		

Table 3: Safety Observations from Third Video (0.5 points/box, 4 points total)

- 7. Put on lab goggles.
- 8. Inflate one of the balloons from the Balloon bag with air (from your lungs). Get it as big as you can (without popping it...). Tie it off.
- 9. Place it somewhere where it won't roll around but also where it won't hurt you if it pops (aka don't hold it in your hand or hug it). Tell anyone around to prepare for a loud noise.
- 10. Dip a toothpick in water to wet it slightly. Then, insert the wetted end into the balloon *right next to* the spot you tied it off, aka the thick "bottom" of the balloon. Insert the toothpick halfway into the balloon, and leave it there. Record observations in Table 4.
- 11. Repeat step 6 with a second toothpick except, this time, insert the toothpick into the side.
- 12. Repeat step 5 to inflate the second balloon.
- 13. Dribble a few drops of water on the balloon.
- 14. Either use the plastic pipette to squirt the balloon with the hexanes OR (if the hexanes evaporated) spray/rub the balloon with a suggested alternative material.
 - See the Materials list for alternatives. Regardless of the alternative you use, be sure you are handling the item appropriately and that you clean up any mess afterward.
 - If the balloon doesn't pop after a few seconds of contact with a few drops of your chosen alternative material, it probably won't. Try something else. If nothing works or you don't have an alternative supply to try, email your instructor.

Toothpick in Bottom	Toothpick in Side	Water	Organic Solvent*	
Popped / Didn't pop				

 Table 4: Observations on Balloon Popping (1 point/box, 4 points total)

*Organic Solvent: ______ (hexanes or name one of the alternatives)

Collaborator(s):

Questions:

1. Define "hazard" and "risk".

Hazard =	
----------	--

Risk =

2. Provide an example from your everyday life of a hazard, the risk, and how you mitigate the risk.

Risk[.]

Hazard:

Risk Mitigation:

3. The balloon demonstration shows how "physical" (toothpick) and "chemical" (hexanes) methods can vary in the impact they have on a substance based on (1) *what* the methods are and (2) *how* they are used. Provide one example of a physical safety issue and one chemical you might encounter *when conducting labs at home*.

Physical:

Chemical:

4. Describe in 1 sentence what happened to your heart rate and emotions when the balloon popped.

(1 point)

(1 point)

(1 point)

(1 point)

(1 point)

(3 points)

5. How might your reactionary response to a popping balloon impact your actions in a laboratory?

(1 point)

6. Brainstorm one action you could take to calm yourself back down after something surprising or scary happens so that you still behave in a safe manner.

(1 point)

Example List of Good and Bad Aspects in Unsafe Lab Practical Videos

Below are examples of good/bad examples of lab safety practices shown in the Lab 1 videos. You may have noticed others or disagree with some based on the situation.

Video 1:

In order of appearance:

- 1. Food and drink at lab bench
- 2. Hot plate is on and currently heating (yet nothing on it)
- 3. Broken glass next to dropper bottles
- 4. Dropper bottles are capped and not in a spot where they'd be knocked over easily
- 5. Bunsen burner is on (very difficult to see in video)
- 6. Lab bench seems mostly clear of extraneous supplies
- 7. Sink is dripping
- 8. Blue solution on cart doesn't seem to have a label to identify it
- 9. White bottle of liquid on cart is uncapped
- 10. Broken glass on cart
- 11. Used gloves left on cart
- 12. Dustpan on cart for cleaning up broken glass and solid spills
- 13. Fire extinguisher present
- 14. Fire extinguish on floor where it could be tripped over
- 15. Paper towel dispenser next to sink
- 16. Dirty beaker next to edge of sink (could easily be knocked over)
- 17. Stir bar left in sink
- 18. Scrub brush next to sink
- 19. Hand soap next to sink
- 20. Hand soap pump bottle is practically empty
- 21. Container holding cleaned glassware
- 22. Squirt bottle lying on its side
- 23. Music playing and laptop at bench

Categorized:

Questionable/Unsafe	Good, Safety-Oriented		
• Food and drink at lab bench	• Dropper bottles are capped and not in a		
• Hot plate is on and currently heating	spot where they'd be knocked over		
(yet nothing on it)	easily		
• Broken glass next to dropper bottles	• Lab bench seems mostly clear of		
• Bunsen burner is on (very difficult to	extraneous supplies		
see in video)	• Dustpan on cart for cleaning up broken		
Sink is dripping	glass and solid spills		
• Blue solution on cart doesn't seem to	• Fire extinguisher present		
have a label to identify it	• Paper towel dispenser next to sink		
• White bottle of liquid on cart is	• Scrub brush next to sink		
uncapped	• Hand soap next to sink		

 Broken glass on cart Used gloves left on cart Fire extinguish on floor where it could be tripped over 	• Container dedicated to holding cleaned glassware
 Dirty beaker next to edge of sink (could easily be knocked over) Stir bar left in sink 	
• Hand soap pump bottle is practically empty	
• Squirt bottle lying on its side	
 Music playing and laptop at bench 	

Video 2:

In order of appearance:

- 1. Putting on gloves before starting work
- 2. Wearing long pants and covering midriff most of the skin is protected
- 3. Hair pulled back in a ponytail
- 4. Seems distracted by something further off
- 5. Doesn't have safety glasses or contacts on
- 6. Doesn't appear to have a supervisor around
- 7. Conducting experiment during the day and seems awake enough
- 8. Appears to have started to use a chemical before checking the label
- 9. Checks label on chemical and puts back, realizing not the desired chemical
- 10. Wearing flipflops
- 11. Seems distracted
- 12. Puts scoopula in jar when done weighing
- 13. Adjusts something with the hot plate, paying attention to experiment
- 14. Touching face and hair with gloves on
- 15. Taking down ponytail
- 16. Checks label of bottle before use
- 17. Wafts to smell instead of sticking nose directly over experiment and breathing in
- 18. Stops sink from dripping
- 19. Stays with experiment the whole time

Questionable/Unsafe	Good, Safety-Oriented	
• Seems distracted by something further off	Putting on gloves before starting workWearing long pants and covering	
• Doesn't have safety glasses or contacts on	midriff – most of the skin is protectedHair pulled back in a ponytail	
• Doesn't appear to have a supervisor around	1 1 2	
• Appears to have started to use a chemical before checking the label	• Checks label on chemical and puts back, realizing not the desired chemical	

 Wearing flipflops Seems distracted Puts scoopula in jar when done weighing 	 Adjusts something with the hot plate, paying attention to experiment Checks label of bottle before use Wafts to smell instead of sticking nose
Touching face and hair with gloves onTaking down ponytail	directly over experiment and breathing in
	Stops sink from drippingStays with experiment the whole time

Video 3:

In order of appearance:

- 1. Wearing gloves, ponytail, long pants
- 2. Put a test tube away in rack
- 3. Picked up Erlenmeyer to look at closely, paying attention to experiment
- 4. Drank and ate food at bench took off one glove to do so but didn't wash hands
- 5. Put safety glasses on after experiment started
- 6. Put safety glasses on, at least
- 7. Spilled a bunch of pipettes and left them out
- 8. Using a pipette instead of pouring
- 9. Tossed dirty pipette on table
- 10. Doublechecked dropper bottle label before use
- 11. Recapped the bottle after pipetting
- 12. Threw away the dirty pipette
- 13. Put the spilled pipettes back in the box
- 14. Having to walk around the cart
- 15. Fire extinguisher on floor where could be tripped over
- 16. Didn't clean scoopula before use took it straight from another bottle
- 17. Stored scoopula in open bottle
- 18. Reached over lab bench to grab bottle
- 19. Paying attention to experiment at first but then walked away and left it
- 20. Left bottles with caps off
- 21. Brought laptop over to lab bench and played music
- 22. Dancing
- 23. Dancing while holding an open Erlenmeyer with liquid in it
- 24. Cleaning Erlenmeyer and left upside down to dry
- 25. Knocked over beaker sitting on edge of bench
- 26. Cleaned up broken glass with a dustpan immediately
- 27. Reached over bench again
- 28. Capped the bottles and put them away on the cart
- 29. Doesn't seem to know what the blue solution is

Questionable/Unsafe	Good, Safety-Oriented
 Drank and ate food at bench – took off one glove to do so but didn't wash hands Put safety glasses on after experiment started Spilled a bunch of pipettes and left them out Tossed dirty pipette on table Having to walk around the cart Fire extinguisher on floor where could be tripped over Didn't clean scoopula before use – took it straight from another bottle Stored scoopula in open bottle Reached over lab bench to grab bottle Paying attention to experiment at first but then walked away and left it Left bottles with caps off Brought laptop over to lab bench and played music Dancing Dancing while holding an open Erlenmeyer with liquid in it Knocked over bench again Doesn't seem to know what the blue solution is 	 Wearing gloves, ponytail, long pants Put a test tube away in rack Picked up Erlenmeyer to look at closely, paying attention to experiment Put safety glasses on – better late than never Using a pipette instead of pouring Doublechecked dropper bottle label before use Recapped the bottle after pipetting Threw away the dirty pipette Put the spilled pipettes back in the box Cleaning Erlenmeyer and left upside down to dry Cleaned up broken glass with a dustpan immediately Capped the bottles and put them away on the cart

Lab 2: Hydrogels Organic Chemistry (42 points)

Purpose: to investigate the macroscopic effects of inter/intramolecular forces on a material's size

Introduction: Polymers are common in nature, and plastics are organic polymers made by chemists. Plastics are extremely useful and a ubiquitous presence in everyday life. Soft contact lenses, for example, are a "hydrogel" plastic, meaning they are hydrophilic ("water-loving") and ideal for use in aqueous mediums such as the surface of the human eye. The properties of hydrogels can be manipulated through changes to their environment. In this lab, you will investigate how changing the pH of a solution alters the electronic interactions between the functional groups of a contact lens and, consequently, the observable size of the lens.

Due: by XXXX

> Allocate roughly half an hour to complete the hands-on experiment, including reading time.

Safety, Waste Disposal, & Clean-Up:

- 1. Wear splash goggles. Gloves are not necessary. Wash your hands at the end of lab.
- 2. Work in a clear, clean space during daylight. Do not involve siblings or other people in your experiment.
- 3. HCl is an acid, and NaOH is a base. Rinse off with cold water if you spill some on you. Immediately rinse out of eyes. Clean up spills with a damp paper towel.
- 4. When done (and after confirming results make sense), return the HCl and NaOH lenses to the appropriate vials. Throw away the remaining lens and used toothpick in the trash.
 - If you later discover you messed up the measurements, you can redo the experiment with these lenses.

Clean-Up Approval Signature (3 points)	
	Name of Signer:
	Relationship to Student:

Materials: Contact your instructor about alternative materials if you're missing an item.

- Hydrogel bag (warning: 1 M HCl acidic and 1 M NaOH basic solutions)
- Student supplies:

Ruler (preferably cm) A piece of white paper A way to tell time (min.)

Instructions: Conduct the following procedure, complete the table, and answer all questions. Follow all "Safety, Waste Disposal, & Clean-Up" guidelines.

- 1. Remove a contact lens from its container using your fingers, and shake the neutral storage buffer solution off.
 - Contact lenses can be torn if you are rough with them. Handle gently throughout.
 - Shaking off the solution prevents diluting the HCl and NaOH in the following steps.

- 2. Drop the lens in the HCl vial. Close the vial lid securely, and shake. Ensure the lens is fully in the solution (it might have gotten plastered to the vial lid during shaking).
- 3. Repeat steps 1-2 for a second lens and the NaOH vial.
 - [Optional for better visualization and to help not mix up the lenses later] Dip a dry toothpick into the yellow food coloring vial from the M&M TLC bag and then into the HCl vial to tint the lens slightly yellow for better visualization. Then dip a dry toothpick into the green food coloring vial from the Column Chromatography bag and then into the NaOH vial.
- 4. Ten minutes or more after step 3, remove both lenses from the vials using the toothpicks (to avoid touching the acidic/basic solutions with your fingers) and the third lens from the buffer solution (the original container).
 - Be careful not to tear the lenses.
 - Keep close track of which lens is which! If you lose track, place the lenses back into each solution (acidic, basic, and neutral buffer) and wait 10 minutes again.
- Place all three lenses with the concave face upward (the open, widest part up), right-side-out (aka not inside-out), and close to each other in a row from acidic (HCl) → neutral buffer → basic (NaOH) on a piece of white paper.
 - Make sure the lenses are the right-side-out. It is possible that the lenses would give slightly different measurements if they are inside-out. A contact lens is meant to be worn in one direction and can get accidently flipped, much like wearing a shirt inside-out. Lenses often have printed, translucent letters/numbers on them to indicate the correct direction; for example, ^{ABC} (read when looking at the outside of the lens) would read flipped ^{OBA} if inside-out.
- 6. Measure the diameter of each lens carefully. Record in Table 1.
 - Recall significant figures for measurements. If the smallest gradation on a ruler is 0.1 cm (9 hash marks between 1 and 2 cm), estimate to the 0.01 cm. If the diameter falls between 1.1-1.2 cm, you might estimate it as 1.11/1.12/1.13/1.14/etc. cm.
 - Be careful not to smush the lenses with the ruler or you might not get an accurate measurement. Measure each lens the same way.

Table 1: Diameters of Contact Lenses (2 points/data box, 1 point/unit box)

Lens fromSolution	Acidic	Neutral	Basic
diameter of lens (unit:)			

- 7. Insert a picture of the lenses from (1) a birds-eye view and (2) a side-on view into Table 2.
 - Ensure there is some sort of label in the pictures to indicate which lens is which.

Tuble 2. Thetales of Contact Lenses (2 points/ box)		
Birds-Eye View	Side-On View	

Table 2: Pictures of Contact Lenses (2 points/box)

Collaborator(s): _____

Questions:

1. Provide examples from this lab of a specific hazard, its risk, and risk mitigation.

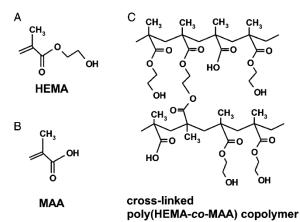
Hazard:

Risk:

(2 points)

Risk Mitigation:

2. HEMA and MAA monomers combine to make the cross-linked hydrogel polymer of the contact lenses, poly(2-hydroxyethyl methacrylate-*co*-methacrylic acid). Draw and name three fundamental functional groups which HEMA and MAA share in common.

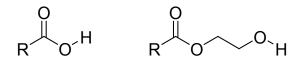


(3 points)

- 3. In addition to bonding, intermolecular forces (IMFs) result in attractions/repulsions between non-bonded atoms in the polymer. The major types of IMFs between components of the hydrogel polymer are London dispersion forces, dipole-induced dipole, dipole-dipole, and H-bonding. Of these, H-bonding is the strongest type (although it is not, despite the name, an actual bond, just an IMF).
 - a. What do the results in Table 1 suggest about the strengths of the attractive IMFs between the polymers when in acidic vs. neutral vs. basic conditions?

(2 points)

b. Draw in the implicit non-bonding electrons on the simplified structures below for MAA and HEMA. (1 point)

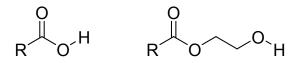


- c. Draw the individual dipole moments on the simplified MAA structure (assuming that the R group is carbon-based). Ignore any dipole for the C-R bond.
 - Recall that larger dipole moments are represented with larger and bolder arrows; ensure that the arrows you draw make sense relative to each other. (2 points)



- d. Circle the part(s) of the two simplified structures which can "accept" an H-bond. Box the parts which can "donate" an H-bond.
 - Only circle/box one atom at a time. Don't circle/box entire groups of atoms.

(3 points)



- 4. At different pH levels, the functional groups of HEMA and MAA change as they undergo protonation or deprotonation. In the acidic 1 M HCl solution, the carboxylic acid and alcohol are both singly protonated, as shown in the images above. In both the neutral buffer and basic 1 M NaOH solution, the carboxylic acid is deprotonated whereas the alcohol remains protonated (though a small percentage of the alcohol FGs will be temporarily deprotonated in the basic solution).
 - a. Draw the simplified structure of MAA in neutral/basic conditions. Show all nonbonding electrons and any formal charge(s).

(3 points)

b. How does the deprotonation of the MAA carboxylic acid affect its ability to participate in H-bonding?

(3 points)

c. If two MAA components of the hydrogel polymer in acidic conditions bumped up against each other, would they attract or repel each other? Explain your reasoning.

(3 points)

d. If two MAA components of the hydrogel polymer in neutral/basic conditions bumped up against each other, would they attract or repel each other? Explain your reasoning.

(3 points)

e. Does the data in Table 1 support your answers for 4c and 4d? Explain.

(1 point)

f. If a contact lens was placed in an extremely strongly basic solution so that the alcohol in HEMA was deprotonated, what effect would you expect this to have on the size of the contact lens? Explain in 1-3 sentences.

(2 points)

Lab 3: Modeling Organic Chemistry (54 points)

Purpose: to (1) build models, (2) connect structures and nomenclature to models, and (3) investigate molecular structures and their movements in a 3D medium

Introduction: Molecular modeling is one of the most important, low-cost, zero-waste, safe, practical tools at an organic chemist's disposal. All chemists use models routinely (a C to represent a carbon atom, a dot to represent an electron, etc.), but "molecular modeling" usually refers specially to physical 3D or computer-simulated models used to visualize (1) how molecules are arranged and (2) how they might interact with other molecules. Use of molecular models (especially high-powered computer models) has greatly enhanced understanding of molecular behavior for complex chemicals, diverse materials, protein interactions with drug candidates, intricate biochemical environments, atmospheric reactions, the energy requirements of reactions, etc. as well as fast-tracked the development of drugs, nanomaterials, diagnostic testing techniques, industrial chemical production plants, climate and pollution predictions, etc.

In this lab, you will practice molecular modeling to investigate the arrangement and behavior of molecules. This investigation will lead to a realization of certain complexities when analyzing structures and the need for in-depth analysis and classification of molecules according to their 3D spatial arrangements in addition to their bond connections and molecular formula.

Due: by XXXX

Safety, Waste Disposal, & Clean-Up:

1. The kit pieces are a choking hazard. Do not leave where a sibling or pet can access them. Put all pieces back in your kit in an orderly manner when you are done.

Clean-Up Approval Signature (1 point)	Name of Signer:
	Relationship to Student:

Materials: Contact your instructor about alternative materials if you're missing an item.

• Darling Models modeling kit Digital camera (phone is fine)

Instructions: Conduct the following procedure, complete the tables, and answer all questions. Due to the nature of the investigation, all questions are integrated into the procedure instead of in a dedicated Questions section. Follow all "Safety, Waste Disposal, & Clean-Up" guidelines.

- *Note:* You are not required to show lone pairs throughout this lab, just bonds and atoms.
- *Note:* If you run out of a specific color or marker ball, indicate what the color or ball is supposed to be when submitting your work.

Collaborator(s): _____

Part I: Making and Translating Models

- 1. Read the assigned pages from the model kit guide (p. 7-12, 14-20, 23).
- 2. Complete Tables 1 and 2 to identify important conventions when using model kits.

Table 1: Color Conventions (1 point/box)			
Black/Grey	White	Red	Blue

Table 2: Piece Conventions (1 point/box)

Piece	Description of Intended Representation(s) [ignoring color]

- 3. For Table 3...
 - a) Build a model for each bond-line structure shown. Insert a picture of each model into the table. Make sure to use the correct types of bonds and colors.
 - b) Name each molecule according to IUPAC convention.

Table 3: Models and Names from Bond-Line Drawings (2 points/model, 1 point/name)

Bond-Line Drawing	Picture of Model	IUPAC Name

ОЦН	

- 4. For Table 4...
 - a) Interpret the names shown to build a model of each molecule. Insert a picture of each model into the table.
 - b) Draw the bond-line structure according to standard organic chemistry conventions ("accurate, unambiguous, simple").

		· · ·
Table 4: Models and Bond-Line Drawings from Names (2 points/model,	1 point/structu	re)

Name	Picture of Model	Bond-Line Structure
ethanol		
methanamine		
but-3-ynal		

- 5. For Table 5...
 - a) Generate a good computer structure (made by you using <u>https://molview.org/</u>). Include wedge/dash stereochemistry where noted. Insert structures as screenshots.

b) Within MolView, generate an accompanying 3D-simulated model for each structure. Insert into the table as a screenshot.

Note on using MolView: The trashcan clears the drawing area. The Center button zooms in on your structure, the broom cleans up slight drawing issues, and "2D to 3D" translates your bond-line drawing into the 3D simulation. You can then move the simulation around by clicking and dragging. Play around with the tools listed under the "Jmol" tab to learn more about the molecule.

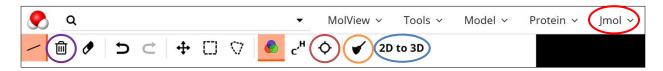


Table 5: Computer-Simulated Structures and Models from Kit (2 points/structure, 1 point/model)

Model	Computer Structure	Computer Model

Part II: Comparing Models

- 6. Build the following molecules with your kit, then wiggle them around to rotate the atoms in 3D space as much as possible *without* breaking the bonds (or breaking the model kit!).
 - a) Propane
 - b) Cyclopropane
 - c) Pentane
 - d) Cyclopentane
- 7. For step 6, circle the molecule in each pair which has a greater ability to wiggle around or "freely rotate" (in terms of the atoms' relationships to other atoms in the same molecule, not the molecule's ability to bob around as a whole in a solution or in the air).

(1 point/pair, 4 points total)

i. propane vs. cyclopropane	ii. pentane vs. cyclopentane
iii. propane vs. pentane	iv. cyclopropane vs. cyclopentane

8. For step 6, circle the molecule in each pair which had greater "strain" or stress/energy. (1 point/pair, 3 points total)

	•• • • •
1. propane vs. cyclopropane	11. pentane vs. cyclopentane
i propune vs. eyenopropune	in pentane vs. cycropentane

iii. cyclopropane vs. cyclopentane

9. Build pentane and 2,2-dimethylpropane, a set of constitutional/structural isomers. Compare their free rotations. Explain which one has worse free rotation.

(3 points)

10. There are two ways to make pent-3-enal. Build a model of the molecule. Then, without changing the *types* of bonds and the *atoms* which are connected, change something about the *spatial arrangement of the alkene* that makes the molecule fundamentally different from the first model you just made (i.e. the molecule can't just wiggle back into the original position). You will need to detach and reattach a bond to do this. You do not need to add or subtract any of the pieces you are using; the pieces you have from the first version will all be used to make the second version. Using your knowledge of molecular models, explain (using drawings and words) what is different about the two versions of pent-3-enal and why they are not considered the same molecule.

(3 points)

Real-Life Application: Historically, the Douglas Fir beetle invades and kills injured and old Douglas fir trees, thus making way for healthy, upcoming saplings to grow and playing a beneficial part in the life cycle of a forest. However, with climate change making (1) winters less severe and (2) droughts more severe in areas with evergreen forests, the beetle is increasingly capable of surviving winter and invading mature, otherwise-healthy trees which can't mount a sap-based response to the beetle invasion (due to the tree now being stressed and dry). Huge swathes of forestland are now "zombie forests" in part due to the beetle's climate change-fueled rampage. The beetle-killed trees are fantastic timber for wildfires, and many of the record-breaking wildfires from the past few years have been fueled by dry, beetle-killed timber.

As an organic chemist working with the United States Department Agriculture and the National Park Service, you are working with the four main anti-aggregation pheromones (Table 6) known for the Douglas Fir beetle. If you can make large batches of these molecules, put them in bags, and staple them to Douglas fir trees, the beetles will not attempt to colonize the trees because they will think that other beetles already live there. You will then have helped prevent widespread ecological damage and horrendous wildfires.

— 11 (

Table 6: Anti-Aggregation Pheromones for the Douglas Fir Beetl		
Common and IUPAC Names	Structure	
Frontalin 1,5-dimethyl-6,8-dioxabicyclo[3.2.1] octane		
Seudenol 3-methylcyclohex-2-en-1-ol	OH	
MCOL 1-methylcyclohex-2-en-1-ol	HO	
MCH 3-methylcyclohex-2-en-1-one	o	

11. However, in order to make frontalin, seudenol, and MCOL, you realize you need more information than drawn to make EXACTLY the correct molecule. What information is missing which you need to know EXACTLY what each molecule should look like?

(2 points)

12. Build a model of MCH. Twist one of the tetrahedral ring carbons up and down so that it rotates within the ring from being wedged to being dashed and vice versa. The chemical structure in Table 6 does not show any wedges or dashes. Does it need to show wedges and dashes in the ring for you to make the correct molecule? Defend your answer using the information gleaned from conducting this lab.

(2 points)

Lab 4: Essential Oil Distillation Organic Chemistry (57 points)

Purpose: to (1) collect a purified substance via distillation and (2) investigate intermolecular forces and physical properties based on sterics and electronics

Introduction: Living organisms produce complex cocktails of "secondary metabolites", molecules which are not directly necessary for survival. Secondary metabolites commonly include molecules involved in signaling/communication with other organisms, such as pollinator and mate attractants or deterrents against parasites, infection, predation, competition, etc. Humans can detect by smell and taste many volatile secondary metabolites produced by plants which are dispersed through the air as gases or secreted as liquids. Interestingly, many of these volatile molecules which comprise a plant's "essential oils" share certain steric and electronic features. In this lab, you will collect a fragrant plant's "essential oil" molecules via steam distillation and investigate the major components of essential oils.

Due: by XXXX

Allocate roughly 1.5-2 hours to complete the hands-on portion, including reading time.

Safety, Waste Disposal, & Clean-Up:

- 1. Wear splash goggles. Gloves are not necessary. Wash your hands at the end of lab.
- 2. Hot, boiling water will occasionally jump out of the saucepan and splatter on nearby surfaces. Wear long sleeves, long pants, and socks to prevent a potential hot water burn.
- 3. Ensure you have an adult supervisor present in the area in case of an emergency. Work in a clear, clean space in the kitchen during daylight and when the kitchen is not busy, being used to cook food, or will need to be used during your experiment. Do not involve younger siblings or other people in your experiment.
- 4. Make sure you only handle plant material that you are not allergic to!
- 5. The jar is thick, tough glass but can still break if knocked over or exposed to extreme temperature differentials.
 - If the jar breaks, sweep up the broken glass with a dustpan. Never use your hands to pick up broken glass. Wipe the area with a damp paper towel to collect remaining small shards (these may be quite hard to see).
 - Do not heat the jar directly on the stovetop; it may crack from the intense direct heat. Follow instructions to heat the jar by placing it in a "hot water bath" (i.e. the saucepan with hot water a saucepan can endure direct heat from the stovetop).
- 6. Exercise care when operating your stovetop. Never leave a hot stovetop unattended.
- 7. Treat any burns (including hot water burns) immediately with running cold water; depending on burn severity, you may need more medical attention.
- 8. Always use oven mitts or some other insulating material to touch potentially-hot items. Remember that you can't necessarily tell if an object is hot just by looking at it.
- 9. Concentrated essential oils are naturally irritating to the skin. Do not apply the collected liquid to your skin, eyes, mouth, other parts of the body, or clothing. Rinse off with cold water and soap in case of contact.

- Essential oils are *not* safe to use directly as perfume and can cause inflammatory skin reactions, even if you are not allergic to the plant it came from. They are *definitely not* safe to eat or drink. Do not add to food.
- 10. When done, throw out the used plant and netting in the trash. Clean the glass jar with soap and water (including the lid), and dry. Rinse the Eppendorf vial and tubing with water. Once your supplies are dry, put them back in the Ziploc bag in the lab bin.

Clean-Up Approval Signature (5 points)	Name of Signer:
	Relationship to Student:

Materials: Contact your instructor about alternative materials if you're missing an item.

- Essential Oil Distillation bag (warning: glass)
- Student supplies:

At *least* 8 inches of a fragrant plant's stem with leaves (ex: rosemary) or handfuls of fragrant petals

- > Best if fresh! Dried spices are questionable substitutes for this experiment.
- > The more of the fragrant plant you use, the easier the distillation.

Oven mittsStovetop ~ 1 tablespoon saltA chair to sit onA kitchen towelIce, a container (i.e. cup or bowl) to hold it in, & backup iceCup for pouring water

- Small ice pieces will be easier to use.
- > You will need to refresh your ice throughout the experiment.

A stovetop-safe saucepan which can hold water and the glass jar Knife for chopping

A saucepan with higher walls will enable you to add more hot water, but it must not be taller than the jar.

Instructions: Conduct the following procedure, complete the tables, and answer all questions. Follow all "Safety, Waste Disposal, & Clean-Up" guidelines.

• *Note:* Do not attempt to do this lab when someone else is using the kitchen. You should have a supervisor around for safety, of course, but a crowded kitchen is asking for a slip, trip, bump, spill, etc. type of accident!

Preparing the Distillation "Pot"

- 1. Remove the glass jar lid. Add water to the jar until the water is at least 2 inches high.
 - If you use a tall saucepan, you can add more water to the jar to help keep it stable. However, you still need space at the top for your plant material.
- 2. Remove the plant leaves/petals from the stem, and spend a minute chopping the fragrant part into smaller pieces. Throw out the denuded stem(s). Record logistical data about the plant in Table 1.

Name	Parts Used (i.e. petals)	Amount (i.e. tbsp)	Freshness?	

Table 1: Logistics about Plant (0.5 points/box)

3. Take a picture of the chopped plant material, and attach to Table 2. Record observations about the plant (ex: smell, color, intensity of smell, etc.).

Item	Observations	Picture
Plant		
Distillate		

 Table 2: Observations about Plant and Distillate Fractions (2 points/box)

- 4. Use the fabric/netting to make a little bag for the chopped leaves (layer if needed so the leaves don't fall through any cracks). Then wrap the green wire *securely and tightly* around two opposite ends of the "bag" so that, when it is dangling from the wire, none of the plant material will fall out.
- 5. Dangle the wrapped plant into the jar *without* letting it touch the water. Arrange it so that the leaves are (1) as high up as possible, (2) pressed against the lid when the lid is closed, and (3) directly in the middle so that the lid hole is over them.
- 6. Securely close the lid on top of the wires so that the lid is keeping the plant in the position specified above.
- 7. Insert one end of the tubing into the hole in the lid so that the tubing pokes into the jar interior by ~ 1 cm and is directly over the leaves. Let the other end dangle down for now, but don't let it get wet.
 - The tube should ideally be flush in the hole so that there are no/minimal gaps between the tube and the lid. If you need to enlarge the lid hole to accomplish this, rotate the tip of a pair of scissors in the hole to enlarge it (but do so cautiously so that you don't make the hole too big).

Preparing the "Hot Water Bath"

- 8. Place the saucepan on a stove burner that has clear counterspace next to it.
 - Your saucepan should be safe to use on the stove (i.e. is this something you use to cook with over a burner? If so, it's probably good).
- 9. Add the jar to the side of the saucepan nearest the counter. Add as much water as you safely can to the saucepan so that the jar is sitting in a "bath". Don't turn the heat on yet.
 - Don't add so much water that the jar starts to float or otherwise seems precarious! Also, don't completely top off the water, or else it'll make a mess when it boils.

Preparing the "Cold Water Bath"

- 10. Add ice to fill up the plastic condiment cup. Sprinkle ~ 1 teaspoon salt on the ice. Add a little cold water to make the cup into a "cold water bath" / "ice water bath" for the vial.
- 11. Carefully place the open vial in the ice water bath with the open cap hooked around the edge of the condiment cup to prevent it from wiggling around or falling in the bath.
 - The inside of the vial must be dry, but have as much of the exterior touching cold water as possible.
- 12. Insert the free end of the tubing all the way in the vial, not just touching at the lip.
 - You want the tubing to go as straight down as possible from its starting point on the jar (to help the distillate drip into the vial easily), but you don't want the ice bath to be directly next to the burner (heat) but rather at least a few inches away.
 - The tubing may be stiff and stuck in a curled position at first. As it warms up, it will become more flexible and relaxed.

Distilling

- 13. Doublecheck that (1) all parts of your set-up are <u>secure</u> and <u>stable</u>, (2) you have oven mitts to handle the saucepan and jar, (3) you have spare ice to replace any melted ice with as the experiment progresses, and (4) you have a towel to wipe up any spilled water with.
- 14. Take a picture of your entire set-up for your instructor to review later as Figure 1.

Figure 1: Distillation Set-Up (5 points)

- 15. Turn on the stovetop to high/medium-high. Get the saucepan water boiling but watch out that the vigorous boiling doesn't rock the jar around. Turn down the heat as needed to keep your apparatus stable, but you must maintain a hot temperature to encourage distillation.
 - Never leave your experiment alone when the heat is on.
 - You should see heavy condensation inside the jar within 5 minutes.
- 16. Monitor the tubing and vial for the appearance of distillate. Place your chair next to the experiment, sit down, and be patient.
 - *Note:* You'll need to replenish the ice several times during the experiment to maintain the ice water bath. You want as much ice as possible and a little water to

help conduct heat from the vial to the ice. As the ice starts to melt, use the pipette to remove water to make room for fresh ice without disturbing the set-up.

- *Note:* Routinely check that the hot water bath still has plenty of water; replace evaporated water with more tap water as needed.
- *Note:* By the 15 min. mark, you should see small droplets throughout the tubing which will get bigger with time, coalesce, and then drip into the vial. They should be large drops by the 30 min. mark. You can occasionally (and *carefully* without knocking over the vial) tap/flick/shake the tubing to help move the drops along. By the 1 hour mark, you should have at least a couple drops collected in the vial.
- 17. After you have obtained a few drops of distillate in the vial, you may stop the distillation whenever you wish. Turn off the stovetop.
- 18. Remove the tubing from the jar, hold it up straight over the vial, and (while holding the vial steady), shake the tubing to encourage any remaining droplets to fall into the vial.
- 19. Cap the vial so that nothing drips out. Wipe the exterior of the (capped) vial dry. Clean up your workspace according to the "Safety, Waste Disposal, & Clean-Up" instructions, but do NOT empty the vial until after you collect data in the following step.
- 20. Take a picture of the distillate in the vial, and attach to Table 2. Record observations about the distillate.
 - Think about characteristics such as color, transparency, smell, etc.
 - To smell the distillate, move to a new area that does not have a noticeable scent. Clear your nose, and take several deep breaths of clean air. Uncap the vial, and place your nose near the vial but not directly over it. Wave your hand slowly and gently over the vial to "waft" the scent toward your nose.

Collaborator(s):

Questions:

1. Provide examples from this lab of a specific hazard, its risk, and risk mitigation.

Hazard:

Risk:

Risk Mitigation:

2. Search for the definition of "essential oil" from Wikipedia. Copy down the definition in the space below.

(1 point)

(2 points)

3. Do you think that you successfully collected the plant's "essential oil"? Briefly present the observations that support your conclusion.

(2 points)

4. Comparing the smell of the plant versus the smell of the distillate, are there any similarities or differences?

(1 point)

5. What molecule do you think is likely the most abundant contaminant in the oil? Explain.

(1 point)

- 6. Based on your experience distilling the essential oil, sketch a "diagrammatic drawing" of a <u>generic</u> steam distillation (not just an <u>essential oil</u> steam distillation).
 - a. Include labels to identify in words <u>what</u> each part of the set-up is that you are drawing, especially if there is a keyword (ex: distillate) or sequence of events.
 - b. Include brief descriptions that explain the <u>purpose</u> of each part of the set-up.

(5 points)

- 7. Think about how the transfer of thermal energy from the stovetop to the essential oil molecules occurs.
 - a. What molecule is fundamentally primarily responsible for transferring the thermal energy, enabling the essential oil molecules to evaporate? (0.5 points)
 - b. What physical state is that molecule in when it transfers energy to the essential oil molecules? [*hint*: this process is called steam distillation] (0.5 points)
- 8. Why is heating the jar in a saucepan of water safer than heating the jar directly on the stovetop?

(2 points)

- 9. Describe in 2-3 sentences how the following alterations might affect distillate collection. Explicitly describe how you expect the results to be different *and/or* the same as you experienced.
 - a. If you dramatically shortened the tubing to $\sim 1-2$ in. length (so that the vial is connected to the tubing pretty much right after it leaves the jar)

(1 point)

b. If the tubing wasn't flush in the hole, if there were cracks in the tubing, or if the lid wasn't securely tightened

(1 point)

c. If the vial wasn't cold but, rather, also being heated

(1 point)

d. If the tubing was set up to let the distillate drip into the vial rather than physically inserting into the vial

(1 point)

e. If the plant was submerged in the water rather than hanging above the water

(1 point)

f. If the plant was to the side of the lid hole rather than directly under it

(1 point)

10. Research the molecule(s) which is/are the primary component(s) of your specific essential oil (Wikipedia is usually a good source for this). Some essential oils might only have one primary component whereas others might have multiple; a max of three slots are provided. Report the following information for each component: common name, structure (computer image is fine), boiling point (in Celsius), and ACS citation denoting where you got the information.

	 ients of the Essential off (0)	
Name		
Structure		
b.p. (°C)	 	
Citation		

Table 3: Components of the Essential Oil (6 points)

11. If you have multiple components, pick one to focus on (one that isn't 100 % C-C and C-H bonds): _____

a. Skim your source about the molecule. Report three separate facts about it.

3)

- 1) 2) (1 point) (1 point)
 - (1 point)b. Are there any safety considerations for this molecule? If so, report them. If not, try to find another source that has that information or write "data unavailable" if

(2 points)

c. Build either a physical model or a computer-simulated model (<u>www.molview.org</u>). Insert at least one clear image of the model; if your model is complex, consider attaching multiple images to show different angles. Move the molecule around to get a feel for shape and size.

(3 points)

Figure 2: Model and Dipole Moments

there do not seem to be any concerns.

d. On top of one picture of the model, draw individual dipole arrows for anything that isn't a C-C or C-H bond. Remember to keep arrow sizes relative. (2 points)

Note: Be prepared to share your answers to questions #9 and 10 with the class or for the instructor to screenshare your responses (*sans* grade) for class discussion to compare different molecules.

Lab 5: Candle Magic Organic Chemistry (44 points)

Purpose: to investigate how the presence of an invisible entity affects a visible reaction

Introduction: Most chemistry happens on a submicroscopic scale invisible to the naked eye. Even for reactions which produce a visible physical change in the macroscopic world, we gain limited information from our observations. This is especially true for organic chemistry, where most of the chemicals the organic chemist handles are either colorless, white, or yellow substances. The ability to detect submicroscopic changes to a chemical's identity is therefore of the utmost importance in determining the outcome of a reaction; it is the investigation into the *reasons* behind natural phenomenon which separates chemistry from magic.

The chemist is therefore often in the position of needing to figure out *how* to detect information and *what* experiment to do next to confirm or reject a hypothesis. Throughout the history of organic chemistry, the greatest breakthroughs have occurred thanks to the development of instruments which help the chemist detect and decipher changes in chemical make-up that a person could never determine on their own. In this lab, you will get a feel for the strengths and the limitations of relying solely on physical observation and critical thinking. This experience will prime you to more fully appreciate the historical developments which enable chemists to rapidly, easily analyze substances and which we often take for granted today. You will also gain experience planning to test a hypothesis

Due: by XXXX

> Allocate roughly 0.5 hours to complete the hands-on portion, including reading time.

Safety, Waste Disposal, & Clean-Up:

- 1. Wear splash goggles. Gloves are not necessary and should not be worn when dealing with open flame unless made of non-flammable, non-meltable material.
- 2. Tie back long hair to avoid catching on fire. Remove any bracelets, necklaces, or other dangling items you are wearing. Wear a shirt that does not have droopy sleeves.
- 3. Ensure you have an adult supervisor present in the area in case of an emergency. Work in a clear, clean space during daylight without any flammable items in your immediate vicinity. Do not involve younger siblings or other people in your experiment.
- 4. If the test tube breaks, sweep up the broken glass with a dustpan. Never use your hands to pick up broken glass. Wipe the area with a damp paper towel to collect remaining small shards (these may be quite hard to see).
- 5. Exercise care when lighting the candles and when manipulating the lit candles. Never leave an open flame unattended.
- 6. Treat any burns (including hot wax burns) immediately with running cold water; depending on burn severity, you may need more medical attention.
- 7. Always use oven mitts or some other insulating material to touch potentially-hot items. Remember that you can't necessarily tell if an object is hot just by looking at it.
- 8. When done, let the candle and test tube cool down to room temperature before putting them away. If you used food coloring, rinse off the Petri dish, test tube, and candle with water. Dry everything. If possible, wrap the dry test tube in a paper towel or toilet paper for

padding (add a little tape to prevent unraveling). Once your supplies are dry, put everything back in the Ziploc bag in the lab bin.

Clean-Up Approval Signature (3 points)	Name of Signer:
	Relationship to Student:

Materials: Contact your instructor about alternative materials if you're missing an item.

- Candle Demos bag (warning: glass)
- Student supplies:

Digital camera (smartphone is fine)A way to safely light candles a few timesA non-flammable plate/bowlWater in a cupFood coloring [optional]Oven mitts or small towel or small tongs or large pliersFood coloring [optional]

You will use this to protect your hand as you hold a test tube briefly over a flame. Pick something that you won't accidentally crush the test tube in (might happen if using a bulky oven mitt or pliers with poor fine motor handling) nor accidentally trail into the candle's flame (more likely if using a fluffy or long towel). Choose wisely.

Fire extinguisher OR large cup of extra, stand-by water

Instructions: Conduct the following procedure, complete the tables, and answer all questions. Follow all "Safety, Waste Disposal, & Clean-Up" guidelines.

- *Note:* Do not attempt this lab in a crowded or messy area. Fire gets out of control *quickly*.
- *Note:* Choose a workspace that is not near a fire alarm nor any air drafts. A sensitive alarm may detect the candle smoke and start an unwarranted, nuisance alarm. An air draft may interfere with your experiment.
- 1. Set the larger part of the Petri dish down on a clean, flat surface with the concave part facing up. Set the smaller part back in the bin (don't need it). Ensure the non-flammable plate/bowl and stand-by water/fire extinguisher are ready for use.

Once you light the candle in the next step, you must quickly complete steps 2-4 before hot wax starts to accumulate and drip down the candle (onto your fingers – OUCH!). Always read the procedure beforehand so you know exactly what you are supposed to do.

Part I: Copper

- 2. Light one birthday candle.
 - Keep the candle upright both while lighting and after it's lit, or hot wax may drip. Remember, wax falls according to gravity, not necessarily along the candle shaft.
 - If you use a match, make sure to blow out the lit match once you light the candle and then place it down on the non-flammable plate/bowl to cool before throwing it away! Trashcan fires have happened before in many chemistry labs.
- 3. *Immediately and carefully*, while holding the lit candle over the open Petri dish, deliberately tilt the candle so that hot wax drips down into the center of the Petri dish (WATCH OUT FOR YOUR FINGERS).

- 4. Hold the candle in its tilted position until you have accumulated several hot wax drops in the center of the Petri dish. Then, blow out the candle, set it down on the non-flammable plate/bowl, and immediately place the butt of the second candle in the hot wax. Press down so that the hot wax "glues" the candle upright on the dish as the wax cools.
 - Press down firmly for half a minute or until the candle remains upright once you let go.
 - If the candle breaks out of position and the wax has already cooled so that it will not let you manipulate it, repeat steps 2-4 to secure the candle to the dish.
- 5. Wait a couple minutes for the wax to completely cool. The Petri candle should be upright and stable. Meanwhile, $coil \sim 1/6$ th 1/4th of the copper wire into a tight vertical coil (less than 1 in. in diameter), keeping the rest of the wire straight to use as a handhold later on.
- 6. Take a picture of your Petri dish candle and the coiled wire for your instructor to reference later in Table 2.
- 7. Light the Petri candle.
- 8. Place the coiled end of the wire on top of the candle wick.
 - You may need to hold the wire with an oven mitt if it gets too hot for your hand.
 - If the flame doesn't extinguish within 15 seconds, remove the wire. Blow out the flame. Let the wire cool to room temperature, then make the coil a tighter coil. Repeat steps 7-9 again.
- 9. The moment the flame extinguishes, immediately remove the wire.
 - If the flame doesn't re-ignite within 5 seconds, gently blow a breath of air to disperse any smoke hanging around the candle. Record observations in Table 1.
- 10. If the candle reignites, put the wire back on the wick to extinguish the flame, and stay there for 30 seconds after it extinguishes. Then, remove the wire to see if the candle reignites. Record all data in Table 1 and other observations in Table 2.

Table 1: Copper's Effect on Flame (1 point/data box)

Did the flame go out?	Did the flame return? (1 st)	Did the flame return? (2 nd)
No / Yes (after ~ sec.)	No / Yes (after \sim sec.)	N/A / No / Yes (sec.)

Table 2: Set-Up and Observations about Copper Wire Experiment (2 points/box)

Set-Up for Part I	Observations (for candle, flame, copper, etc.)

Part II: Test Tube

- 11. Place the Petri dish on a plate or some other secondary container to catch spilled water. Carefully pour water into the Petri dish until the dish is full but not overflowing.
- 12. [Optional] Add a small drop of food coloring to help visualize the next part.

Read the next part completely before proceeding.

- 13. Light the Petri candle. Let it burn for a full minute without disruption, or you might not observe the phenomenon in the following steps.
- 14. Hold your digital camera in one hand, ready to take a picture. In your other hand, use your oven mitt/towel/tongs/pliers to hold the test tube *upside down* (i.e. the tube's open part facing toward the floor and the closed, curved part facing toward the ceiling).
 - Alternatively, you can have another person holding the camera so you can focus completely on the test tube.
 - Either keep the paper towel that the test tube came wrapped in or use a fresh paper towel later to re-wrap the test tube after the experiment for transport back to school.
- 15. After the candle has burned for the specified time range, *quickly but carefully* lower the upside-down test tube over the lit candle and down into the water. Hold on to the test tube and keep it steady.
 - You must completely encase the candle with the test tube, and you must do so quickly to see the results. The test tube must be fully touching the water and *almost* touching the Petri dish but *not actually pressed down* on the Petri dish. The tube should be swimming, so to speak, in the water, not standing.
- 16. Take a picture of the resulting phenomenon for instructor reference in Table 3.
- 17. Remove the test tube, and let both it and the candle sit for a few minutes to cool down before cleaning up.

Tuble 5. bet op und observations about	Copper whe Experiment (2 points/box)
Results for Part II	Observations (for candle, flame, water, etc.)
	1

 Table 3: Set-Up and Observations about Copper Wire Experiment (2 points/box)

Collaborator(s):

Ouestions:

Hazard:

1. Provide examples from this lab of a specific hazard, its risk, and risk mitigation.

Risk Mitigation:

2. In Part I, what is likely the biggest safety reason for why you were instructed to secure the candle to the Petri dish before conducting the copper wire experiment rather than just hold the candle in one hand and the copper wire in another?

(2 points)

(2 points)

- 3. Why do you think you were told to smush a second candle onto the hot wax on the dish rather than smush the first candle onto the hot wax after blowing out its flame?
- 4. Without reading any further past this question, ... (aka don't change your answer after reading the next question – just put down your first gut instinct – completion grade for this question).
 - a. Hypothesize what the copper might be doing to the flame to extinguish it.

(1 point)

b. State the information/observations that you considered to arrive at your hypothesis.

(1 point)

5. The following "fire triangle" depicts the three fundamental things needed for a flame to occur: fuel, oxygen, and heat.

> a. Considering this new information, when applying the copper wire to a flame, what do you think is the *likeliest* explanation for what the copper does to extinguish the flame? Reflect on your observations and what you know about copper, fire, etc. Explain your reasoning.

> > (2 points)

b. Did your answer to question #4 change after you saw the information about the fire triangle? Describe how it did/didn't change and any information you used to reconsider your original hypothesis.

(1 point)



(2 points)

Risk:

- 6. If you remove the copper wire fast enough, the candle might reignite. However, if you keep the wire on the flame long enough, the candle will not reignite. Explain why for both situations.
 - a. Why it might reignite soon after
 - b. Why it will not reignite after a time

7. Since you can't know for sure that your proposed answers to the above questions about copper's influence on the flame are correct or not, propose at *least* one experiment you might need to run to support or reject your hypothesis. Be explicit about what the potential results of the experiment would tell you about your hypothesis.

• If you would need to run more than one experiment to really support your hypothesis well, then propose more than one.

8. Instead of running back-up experiments to check your hypothesis, it would be great to have an instrument that could directly test your hypothesis. Describe any imaginary instrument that you would like to exist in order to eliminate the need to run additional experiments

- that you would like to exist in order to eliminate the need to run additional experiments (i.e. what would your imaginary instrument need to be able to detect?). (1 point)
- 9. For Part II, what is the most relevant reason for why it would not be safe to hold the test tube in your bare hand for this particular experiment. Assume that the experiment goes *as planned* and you aren't shattering the tube or accidentally missing the flame and putting your hand over the fire instead that's not what I'm asking you to predict.
- 10. In Part II, what do you think would happen if you...
 - a. pushed the test tube all the way down on the Petri dish?
 - b. did not make contact with the water soon after extinguishing the flame?

(1 point)

(1 point)

(1 point)

(1 point)

(3 points)

(2 points)

- 11. Without reading any further past this question, ... (aka don't change your answer after reading the next question just put down your first gut instinct completion grade for this question).
 - a. Hypothesize what is causing the water to rise up the tube.

(1 point)

b. State the information/observations that you considered to arrive at your hypothesis.

(1 point)

- 12. Recall from introductory chemistry that, as a gas gets hotter, it expands. As a gas gets cooler, it contracts. Reflect now on what happened when you put the test tube over the candle.
 - a. Considering this information, what do you think is the *likeliest* explanation for what causes the water to rise up the tube? Explain your reasoning.

(2 points)

b. Did your answer to question #11 change after you saw the information about the fire triangle? Describe how it did/didn't change and any information you used to reconsider your original hypothesis.

(1 point)

- 13. Since you can't know for sure that your proposed answers to the above questions about the rising water are correct or not, propose at *least* one experiment you might need to run to support or reject your hypothesis. Be explicit about what the potential results of the experiment would tell you about your hypothesis.
 - If you would need to run more than one experiment to really support your hypothesis well, then propose more than one.

(3 points)

14. Instead of running back-up experiments to check your hypothesis, it would be great to have an instrument that could directly test your hypothesis. Describe any imaginary instrument that you would like to exist in order to eliminate the need to run additional experiments (i.e. what would your imaginary instrument need to be able to detect?).

(1 point)

Lab 6: Extraction Organic Chemistry (37 points)

Purpose: to (1) conduct liquid-liquid extraction between aqueous and organic phases and (2) conduct solid-liquid extraction and filtration

Introduction: Organic chemists specialize in manipulating carbon-based molecules. Most of these manipulations fall into one of two categories: conducting reactions or conducting purifications. In chemical reactions, a chemical's identity is changed $(X \rightarrow Y)$. In purifications, one type of chemical is separated out from an impure mixture (X is still X but is now pure instead of being contaminated with traces of A, B, C, etc.). Most purification techniques in organic chemistry labs are "solution-phase", meaning that chemicals are dissolved into a liquid in order to conduct the purification. Solution-phase purifications are simple to conduct and enable the chemist to easily customize the technique for individual situations. In this lab, you will use liquid-liquid extraction, solid-liquid extraction, and gravity filtration to explore solution-phase purifications.

Due: by XXXX

Allocate roughly 2 hours to complete the hands-on portion spread over > 1 day, including reading time.

Safety, Waste Disposal, & Clean-Up:

- 1. Wear splash goggles.
- 2. Ensure you have an adult supervisor present in the area in case of an emergency. Work in a clear, clean space during daylight with good ventilation. Do not involve younger siblings or other people in your experiment.
- 3. Rubbing alcohol and ethyl acetate are flammable and volatile. Use in a well-ventilated area while wearing goggles. Do not shake or expose to flame.
- 4. If the glass breaks, sweep up the broken glass with a dustpan. Never use your hands to pick up broken glass. Wipe the area with a damp paper towel to collect remaining small shards (these may be quite hard to see).
- 5. Exercise care when operating your stovetop. Never leave a hot stovetop unattended.
- 6. Treat any burns (including hot water burns) immediately with running cold water; depending on burn severity, you may need more medical attention.
- 7. Always use oven mitts or some other insulating material to touch potentially-hot items. Remember that you can't necessarily tell if an object is hot just by looking at it.
- 8. When done, throw away the used cinnamon, plastic cup, toothpick, pipette(s), and coffee filter(s) in the trash. Wash the glass pan, glass cup, glass jar, and all used kitchen supplies with water and soap. Once your jar is dry, put it back in the Ziploc bag in the lab bin.

Clean-Up Approval Signature (5 points)	Name of Signer:
	Relationship to Student:

Materials: Contact your instructor about alternative materials if you're missing an item.

- Extraction bag (warning: flammable ethyl acetate)
- Yellow food coloring from M&M TLC bag
- Glass jar from Distillation bag
- Student supplies:

```
Digital camera (smartphone is fine)

Oven mitts or other insulation

<sup>1</sup>/<sub>4</sub> cup ground cinnamon*

<sup>1</sup>/<sub>2</sub> cup rubbing alcohol (70 % or higher)*

<sup>1</sup>/<sub>4</sub> Stovetop and pot

<sup>1</sup>/<sub>4</sub> recessary, you can alter the amounts of cinnamon and alcohol if the ratio is the same.
```

Instructions: Conduct the following procedure, complete the tables, and answer all questions. Follow all "Safety, Waste Disposal, & Clean-Up" guidelines.

Part I: Liquid-Liquid Extraction

- 1. Put on goggles.
- 2. Use the toothpick to transfer a *tiny* drop of yellow food coloring from the M&M TLC bag into the colorless Eppendorf vial containing ethyl acetate (EtOAc).
 - Just dip the toothpick into the food coloring vial, then dip it into the EtOAc vial. You may repeat with the other, clean end of the toothpick if you feel you need a slight bit more.
- 3. Use the pipette to carefully transfer 0.5 mL tap water into the Eppendorf vial. Don't spill anything.
- 4. Securely close the vial cap. Closely observe the contents of the vial. With your thumb on the cap to keep it closed, shake the vial vigorously in all directions for ~ 10 seconds.
- 5. Hold the vial upright and steady. Immediately start observing for ~1 uninterrupted minute what happens inside the vial as the solution settles down from the shaking.
- 6. Take a picture of the settled contents of the vial, and attach to Table 1. Record observations in Table 1.

Observations Before Shaking	Observations After Shaking	Picture of Vial After Shaking

Table 1: Liquid-Liquid Extraction (1 point/box)

- 7. Now, figure out a way to effectively physically *collect* the layer that contains the most food coloring. Write down your procedural plan, execute the plan, then report how well your attempt worked in Table 2. Take a picture of the extract, and attach it to Table 2.
 - Your goal is to have the food coloring extract in one container by itself.
 - Consider the supplies you have on hand, the small size of the solution, and your need to not spill anything.

Plan	Results	Picture of Extract

Table 2: Collection of Food Coloring Extract (1 point/box)

Part II: Solid-Liquid Extraction

- 8. Cut out a large hole in the bottom of the red plastic cup. The hole should be almost as large as the bottom of the cup itself.
- 9. Place the cup upside down on top of a glass drinking cup. Put a coffee filter in the hole.
- 10. Add ¹/₄ cup ground cinnamon powder to the glass jar. Add ¹/₂ cup rubbing alcohol.
- 11. Fill a small pot half full of water, and heat the water to boiling on the stovetop. Turn off the heat, and place the pot on a hot pad/oven mitt on the counter.
- 12. Place the glass jar (without a lid on) into the pot of hot water. Stir the cinnamon mixture with a spoon for 10 minutes (you don't have to continuously stir the entire time, just at least a few times per minute).
- 13. Slowly pour the warm cinnamon mixture into the center of the coffee filter.
- 14. Monitor the "filtrate" as gravity causes it to drip through the filter into the glass cup. If it stops dripping, gently stir the heterogeneous mixture in the filter with the spoon. If that does not help, pipette the remaining mixture back into the jar, replace the clogged filter, and continue collecting.
- 15. Pour the collected filtrate into a flat glass pan. Label the pan with "CHEMISTRY EXPERIMENT DO NOT TOUCH", and place it somewhere safe from siblings and pets.
 - A warm and sunny location will help the following evaporation occur more quickly. Do not cover the pan with a lid. The larger the pan, the more the solution can spread out (and therefore the faster the evaporation).
- 16. Record observations about the solid residue on the filter paper in Table 3 before discarding it in the trash. Be sure to smell it (via wafting technique).
- 17. After the solvent evaporates from the glass pan and leaves the filtrate dry (this should take at least a day), take a picture and record observations of the filtrate in Table 3. Be sure to smell it (via wafting technique).

Table 5. Fillation of Chinamon Extract (1 point/box)			
te Picture of Dry Filtrate			

 Table 3: Filtration of Cinnamon Extract (1 point/box)

Collaborator(s):

Questions:

1. Provide examples from this lab of a specific hazard, its risk, and risk mitigation.

Hazard:

Risk:

Risk Mitigation:

Part I: Liquid-Liquid Extraction

2. What do your observations tell you about the miscibility of EtOAc and water? Do they dissolve easily into each other or not? Explicitly state the observations that support your answer.

(1 point)

(2 points)

After shaking the vial, which "layer" was the EtOAc: top or bottom? To answer this, consider Table 4 below. Explain which layer in the vial is EtOAc and which is water.
 Table 4: Reference Densities (2 points)

	2011311105
Substance	Density (g/mL)
EtOAc	0.902
Solid H ₂ O, ice (0 °C)	0.917
Liquid H ₂ O (20 °C)	0.998

- 4. Chemists often conduct liquid-liquid extraction to move one chemical (X) in a mixture (X+Y+Z) from impure liquid A to pure liquid B, leaving the other contaminants (Y+Z) behind in liquid A. Liquid B can often then be evaporated to yield pure X all by itself. Suppose you were trying to use liquid-liquid extraction to purify yellow food coloring from a mixture in this manner. Based on your observations, explain how successful you would expect to be in the following scenarios:
 - a) The original mixture is water-based. You attempt to extract the food coloring using EtOAc.

(1 point)

b) The original mixture is EtOAc-based. You attempt to extract the food coloring using water.

(1 point)

Propose two, different important roles that shaking plays in liquid-liquid extraction.
 a)

(1 point)

b)

(1 point)

6. Propose a situation in which liquid-liquid extraction might *not* yield a pure compound but, instead, yields an impure extract of the desired compound *with* an original contaminant.

(2 points)

- 7. Reflect on the drug design project and the predictions of hydrophilicity and lipophilicity. Most organic chemistry liquid-liquid extractions use an "aqueous phase" (aka liquid water) and an "organic phase" (aka a liquid organic solvent). The aqueous phase is usually the most polar solvent a chemist uses; the organic phase is usually either nonpolar (i.e. hexanes) or moderately polar (i.e. EtOAc). If you were designing a liquid-liquid extraction procedure, how might you predict whether a chemical would best "partition into" the aqueous phase or the organic phase? Refer specifically to how you could use your knowledge of a molecule's structure to address this problem (since most organic molecules are colorless in solution, you can't see which phase they are actually in!).
- Part II: Solid-Liquid Extraction 8. What was the purpose of heating the cinnamon mixture before filtering? 9. What was the purpose of filtration? 10. trans-cinnamaldehyde is the primary molecule responsible for cinnamon's scent. a) Based on its structure, would you expect *trans*-cinnamaldehyde to be extracted in Ο this experiment? Explain.
 - b) Was trans-cinnamaldehyde extracted during the solid-liquid extraction or not? Explicitly refer to your observations to support your answer.

(1 point)

(2 points)

(3 points)

(1 point)

(1 point)

- 11. Wikipedia lists several physical properties for *trans*-cinnamaldehyde.
 - a) Determine whether your solid-liquid extraction and gravity filtration were fficient for purifying trans-cinnamaldehyde. Describe the data/observations you used to make your determination.

(1 point)

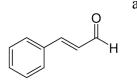
b) For (a) above, make a submicrocopic chemistry explanation for why the purification was/wasn't sufficient.

(1 point)

c) Propose one specific thing you might try to further purify your trans-cinnamaldehyde.

(2 points)

	Properties
Chemical formula	C ₉ H ₈ O
Molar mass	132.16 g/mol
Appearance	Yellow oil
Odor	Pungent, cinnamon-like
Density	1.0497 g/mL
Melting point	−7.5 °C (18.5 °F; 265.6 K)
Boiling point	248 °C (478 °F; 521 K)
Solubility in water	Slightly soluble
Solubility	Soluble in ether, chloroform Insoluble in petroleum ether Miscible with alcohol, oils
Magnetic susceptibility (x)	−7.48 × 10 ⁻⁵ cm ³ /mol
Refractive index (<i>n</i> _D)	1.6195



Lab 7: Glowmatography Organic Chemistry (69 points)

Purpose: to (1) separate components of a solution-phase mixture via chromatography and (2) observe macroscopic separation achievable by exploiting differences in polarity

Introduction: Very few molecules are found "pure" in nature or even in the chemistry lab. Instead, the world is covered in mixtures, and an entire subfield of chemistry is devoted to the science of separation, identification, and quantification (analytical chemistry). The experimental organic chemist also inevitably needs to purify and identify compounds as a routine part of the job, either before and/or after an experiment. Separation techniques such as chromatography enable both effective purification and preliminary identification of many organic molecules. In this lab, you will exploit differences in polarity to separate components of a glowstick solution using a rudimentary chromatographic technique.

Due:

- 1. Part I: Pre-Lab Baseline
 - by XXXX
- 2. Part II: Pre-Lab Reflections
 - by XXXX
- 3. Part III: Running the Experiment
 - by XXXX
 - Allocate roughly 0.5-1 hour to complete the hands-on portion, including reading time.
- 4. Part IV: Analyzing Results
 - by XXXX
- 5. Part V: Extensions
 - by XXXX

Safety, Waste Disposal, & Clean-Up:

- 1. Wear chemical splash goggles and the provided nitrile gloves throughout the experiment.
- 2. 95 % denatured ethanol is toxic by ingestion, flammable, and volatile. Do not shake, ingest, or expose to heat. Work in a ventilated area (no bedroom closets). Rinse off with cold water in case of contact. Rinse eyes for 15 minutes in case of eye contact, and remove any contact lenses. In case of ingestion, call the Poison Control Center Hotline (1-800-222-1222).
- 3. The inner tubes of the glowsticks are glass and are broken when the glowstick is bent for activation. Tiny fragments will get on your scissors when the glowstick is cut open. Wipe off your scissors with a damp paper towel afterward to remove the fragments.
- 4. The glowstick solution is non-toxic but may stain fabric. Rinse with water.
- 5. When finished, throw away the used glowsticks, chalk, pipettes, and plastic lids in the trash. Wipe down your work area with a damp paper towel to clean up any spills and to catch any missed glass. Return the dropper bottle to the lab bin, inside the Ziploc bag; ensure the top is securely attached and the bottle stands upright in the bin if there is any remaining ethanol.

Clean-Up Approval Signature (5 points)	Name of Signer:
	Relationship to Student:

Materials: Contact your instructor about alternative materials if you're missing an item.

Glowmatography bag (warning: 95 % denat. ethanol, glass inner tube, staining solution)
Student supplies:

Scissors Paper towels Pencil Flashlight (smartphone works) Digital camera

Note: Use a standard graphite pencil. A pen, marker, or colored pencil may compromise results.

Instructions: Complete the following steps, and answer all questions. Due to the nature of the investigation, there are questions for both before and after the experiment. Follow all "Safety, Waste Disposal, & Clean-Up" guidelines.

- You must complete Part I before starting Part II, Part II before starting Part III, etc.
- Not all the pages of this overall packet are shown at once in Formative; the additional pages will be added as you advance through the feedback and approval process.

Collaborator(s): _

• If you don't collaborate with someone at first but collaborate with someone by the time you finish the last part of this overall experiment, make sure to acknowledge them here.

Part I: Pre-Lab Baseline

1. Complete the separate assignment titled "Pre-Lab Baseline" before proceeding to Part II. The assignment is graded based on completion and will be revisited after the third chromatography experiment concludes.

(10 points)

□ Check this box when you have completed the "Pre-Lab Baseline" questions.

Part II: Pre-Lab Reflections

2. Identify the following terminology for this experiment (read procedure in Part III).

(1 point/term) a. The mobile phase is _____.

- b. The stationary phase is ______.
- c. The sample is .
- d. The developing chamber is

e. The vertical space that the sample moves along is called the

- f. The process of the mobile phase and sample moving along the stationary phase is called _____.
- g. The process of applying the sample to the stationary phase is called
- 3. Identify the underlying *physical property* that enables chromatographic separation.

(1 point) The physical property in question is called ______. It stems from the separation of charges, or dipole moments, between atoms of different electronegativities.

- 4. The stationary phase in this experiment is _____ (more / less) polar than the mobile phase [*hint*: refer to their chemical structures]. (1 point)
- 5. Describe the overall *chemical property* that explains *why* the physical property in question #3 will cause the sample to move along the stationary phase during the experiment.

(2 points)

- 6. The sample is an impure mixture of multiple types of molecules. Considering the relative polarities of the stationary phase vs. the mobile phase, the molecule which moves the furthest along the stationary phase in this experiment is (more / less) polar than those which move less [*hint*: think of "like dissolves like" and IMFs]. (1 point)
- 7. What should you do if you accidentally... (1 point each)

 - a. spill ethanol on your skin? ______b. splash ethanol into your eyes? ______
 - c. swallow ethanol?

□ Check this box to affirm you read the "Safety, Waste Disposal, & Clean-Up" section. (2 points)

Part III: Running the Experiment

Note: If you have access to a printer, it may be easier for you to print off the procedure rather than read off the computer so that you can (1) check off each step as you proceed in order to not get lost in the long procedure and (2) write down observations quickly.

- 8. Gather all materials. Prepare to thoroughly darken the room (don't darken it yet).
- 9. Put on splash goggles and nitrile gloves.
- 10. Carefully snap one of the chalk sticks in half. Leave the other two chalk sticks intact.
 - a. Handle the chalk carefully so that you don't make scratch marks or dig gouges into the surface.
- 11. Using a pencil, softly mark a circular line around each piece of chalk, ~1 cm from the "bottom" (designate the flattest end of each chalk stick as the "bottom"). The line should wrap around the chalk completely.
 - Do this for both the halved sticks and also the two intact sticks.
 - Don't dig into the chalk with the pencil. Draw lightly.
- 12. Approx. half a centimeter above that line, mark another circular line.
- 13. Place the empty plastic cup on a flat, stable surface. Lay down paper towels in between you and the plastic cup. Lay down the two plastic lids on the paper towel surface, with the "interior" side of the lids facing up.

After activating the first glowstick, don't stop. There's no need to rush, but don't dawdle. This experiment is time-sensitive from here onward. Do not proceed if you cannot allocate 30 minutes uninterrupted to finish the procedure.

Chalk Sticks #1 and #2 (Halved)

- 14. Activate one of the glowsticks by thoroughly bending every part of the glowstick.
 - Bend until you don't hear any crackling.
- 15. Shake the glowstick vigorously a few times to mix the ingredients thoroughly.
- 16. Over one of the plastic lids, cut the activated glowstick in half. Cut off one of the halves' ends. Use the lid to catch the glowing solution (it will immediately fall out of the glowstick once you cut off the end). Repeat for the other half.
 - The inner part of the glowstick is broken glass. Handle with care.
- 17. Using a clean pipette, draw up some of the glowstick solution. With *very steady hands*, place one drop of the solution *between the pencil lines* on one of the halved chalk sticks (Chalk #1).
 - Attempt to not go past the lines. You can alter the amount of pressure that you squeeze the pipette bulb with to expel either more or less solution.
- 18. With the same pipette, place more of the glowstick solution on the second halved chalk stick (Chalk #2). However, this time, deposit the glowstick solution completely around the stick (*while staying within the pencil lines*) instead of just in one spot.
- 19. Wait a moment for the solution to dry. Then repeat to deposit more solution on the chalk on top of the glowing band you just made.
- 20. Stand both halved chalk sticks upright in the plastic cup with each glowing band at the bottom.
 - Ensure the chalk sticks are level and not tilted.

- 21. Uncap the ethanol dropper bottle. Add ethanol to the plastic cup until the liquid is *just below* the height of the *lowest point* of the glowing bands.
 - Do NOT continue to add ethanol such that the ethanol touches any part of the glowing band. Cap the bottle, and place it upright on your work surface.
 - Throughout the experiment, you will need to periodically replenish the ethanol in the plastic cup as the ethanol soaks into the chalk. Keep an eye on the ethanol level so that the chalk doesn't dry out.
- 22. Thoroughly darken the room so you can easily see the glowing during the experiment.
- 23. Observe the ethanol (makes the chalk look damp) and the fluorescent, glowing solution travel up each chalk stick. Use a flashlight periodically to see if there are any colored, non-fluorescent spots moving up the chalk as well.
- 24. Record observations in Table 1. Take a picture of the two chalk sticks when the ethanol is ~ halfway up the chalk.
- 25. Remove the chalk sticks from the plastic cup once the ethanol reaches ~1 cm from the top OR when you can no longer see either any color or glowing (whichever occurs first).

Proceed to the next steps as soon as you finish jotting down your observations.

Table 1: Observations on separation of glowstick solution for halved chalk sticks (2 points/box)

	Chalk 1 Obs.	Chalk 2 Obs.	Picture During Elution
- 1		1	

Chalk Sticks #3 and #4 (Whole)

- 26. With the same used pipette and glowing solution from the plastic lid, "load" the glowing sample onto a new, longer chalk stick (Chalk #3) in the same manner as Chalk #2 (steps 18-19).
 - Load the sample at least four times, letting the chalk dry for a few seconds between each application so that the glowing solution doesn't seep across the pencil lines.
 - You can gently blow dry air onto the chalk to speed up the drying process.
 - Stop applying more sample when it seems like the solution is seeping across the pencil lines with each new application.
- 27. Place the loaded Chalk #3 into the ethanol cup, again being careful that the ethanol doesn't touch the glowing band.
 - If the ethanol is too high for this chalk, use the clean pipette to remove some of the ethanol. Add the ethanol from the pipette back in later as the ethanol level falls.

- You might need to add more ethanol from the dropper bottle, depending on how much the ethanol level dropped after running the two halved sticks.
- 28. Activate and drain the second glowstick into the second, clean plastic lid in the same manner as steps 14-16.
 - Make sure to clean off the scissors between glowsticks to prevent contamination. Wipe with a damp paper towel to remove glowstick solution and any glass shards.
 - Keep an eye on the "running" chalk stick as you work. By the time the color gets halfway up the chalk, you should have completed step 29, even if not done with this current step.
- 29. Flash your flashlight on the already-running Chalk #3 to observe the separation of the glowing sample into glowing and non-glowing parts. Take a picture when the ethanol is ~ halfway up the chalk (the chalk looks damp where the invisible ethanol is).
- 30. Record observations about Chalk #3 in Table 2; make sure to address the following:
 - Do you see any colored parts which aren't also glowing? If so, where are the nonglowing parts relative to the glowing parts on the chalk (i.e. higher or lower?)?
- 31. Load the final, unused chalk (Chalk #4) with the second, activated glowstick solution in the same manner as earlier. Place it in straight & upright in the ethanol cup *without knocking over Chalk #3*.
 - Continue to add ethanol from the dropper bottle to the plastic cup to maintain the liquid level slightly below the pencil bands.
- 32. Repeat steps 29-30 of observing and recording for Chalks #4 and #3 periodically until there is no more color or glowing noticeable OR the ethanol reaches ~1 cm from the top (whichever comes first).

Table 2: Observations on separation of glowstick solution for whole chalk sticks	(2	points/box)	
--	----	-------------	--

Chalk 3 Obs.	Picture During Elution	Chalk 4 Obs.	Picture During Elution

Color* of 1st glowstick:Color of 2nd glowstick:*Be unambiguous: Purple? Hot pink? Red?Yellow? Yellow-green? etc.

Note: Make sure to get Clean-Up Approval at the top of this packet.

Note: Crop your experiment pictures before uploading into Formative so that they fit in the tables.

Part IV: Analyzing the Results

33. Make a flowchart that diagrams the important steps/parts of the Glowmatography experiment procedure for Chalk #3.

(2 points)

- 34. Sketch the experiment's overall physical set-up for Chalk #3 (using diagrammatic drawing, not a picture taken from a camera).
 - Include labels to identify in words <u>what</u> each part of the set-up is that you are drawing, especially if there is a keyword at play (ex: mobile phase).
 - Include *brief* descriptions that explain the <u>purpose</u> of each part of the set-up.
 - Indicate how you loaded the sample.

(4 points)

35. There are two chemicals in each glowstick solution which are capable of being detected indirectly with the human eye (though we can't see the molecules themselves): (1) a dye molecule which gives off color and (2) an unstable, colorless "oxalate" molecule which causes the dye to glow. There are some other molecules in the solution as well which play important roles but which we are not concerned with here.

Did you gather any evidence that suggests separation occurred between the dye and the oxalate on either Chalk #3 or Chalk #4? Describe the experimental observations which either support or refute that separation occurred for the two glowing solutions.

Separation for Chalk #3? Yes / No for Chalk #4? Yes / No (3 points) Supporting Observations and Rationale:

36. Comparing the dyes and the oxalate, which type of molecule is likely more polar, based on your experimental observations and answers to questions #4 and 5? Briefly and explicitly mention the observations which support your conclusion.

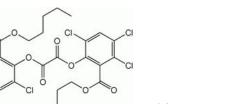
More polar: dyes vs. oxalate

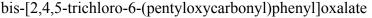
Supporting Observations and Rationale:

37. Although the glowstick company does not fully disclose the make-up of the glowstick solution, below are the likely structures for the oxalate and some of the possible dyes in the glowsticks. Explain or not whether your answer to the question above still makes sense considering these structures.

(1 point)

(3 points)







Part V: Extensions

38. In 1-2 sentences, propose one potential experimental issue concerning data collection that could result from working slowly or even pausing in the middle and coming back later.

(1 point)

39. If you allow the ethanol to touch the glowing band when you start the experiment, you will contaminate the reservoir of ethanol in the plastic cup. Predict what would happen to contaminate the ethanol when the band touches the ethanol.

(1 point)

40. Propose a reason why you must use a graphite pencil instead of a pen, marker, or colored pencil to mark the sample loading spot on the chalk sticks.

(1 point)

41. With Chalk #1 and Chalk #2, you loaded different amounts of the same sample onto the stationary phase. Based on your observations, why were you later instructed to load Chalk #3 and #4 in the same manner as Chalk #2?

(1 point)

- 42. Identify how the following situations could have affected your ability to gather meaningful data in your quest to gather evidence on the separation between the fluorescent and non-fluorescent molecules.
 - a) Putting only a tiny bit of glowing sample onto chalk

(1 point)

b) Putting 100 glowsticks'-worth of sample onto the chalk

(1 point)

43. If you loaded the glowing solution onto the stationary phase in a very messy manner (i.e. exceeding the pencil lines by a lot), do you think that the separation would be better or worse than you observed? Rationalize.

Separation if messy loading:	better	or	worse	
				(2 points)
Rationale:				

44. If different stationary and mobile phases were used such that the mobile phase was more polar than the stationary phase, which would elute more: the dye or oxalate?

(2 points)

Circle: dye or oxalate

Lab 8: Detection of Tartrazine Organic Chemistry (78 points)

Purpose: to (1) separate, compare, and identify components of a solid mixture and (2) build on previous chromatography experience to improve knowledge, competency, and independence

Introduction: By exploiting differences in chemical's properties, organic chemists can separate, identify, and isolate pure substances out of impure mixtures. Chromatography is therefore one of the most common and useful techniques an organic chemist employs. However, there are multiple types of chromatography, and each version has its advantages and disadvantages, tips and tricks, special set-ups or equipment, and different ways to properly conduct the experiment. In this lab, you will apply what you learned about chromatography in the Glowmatography experiment to build a procedure for conducting a different type of chromatography in order to analyze the components of food coloring in M&M candies.

Due:

- 1. Part I: Applying Chromatography Experience
 - by XXXX
 - on Formative
- 2. Part II: Writing the Procedure
 - by XXXX
 - on Formative (questions, instructions, grade) and Schoology (procedure template)
- 3. Part III: Running the Experiment
 - by XXXX
 - on Formative (questions, instructions, grade) and Schoology (procedure and data)
- 4. Part IV: Analyzing the Results
 - by XXXX
 - on Formative
- 5. Part V: Extensions
 - by XXXX
 - on Formative

Instructions: Complete the following steps, and answer all questions. Due to the nature of the investigation, the questions are integrated into the procedure instead of just in a dedicated Questions section.

- You must complete Part I before starting Part II, Part II before starting Part III, etc.
- Instructor feedback and approval is required before conducting the experiment.
- Not all the pages of this overall packet are shown at once in Formative; the additional pages will be added as you advance through the feedback and approval process.

Collaborator(s):

Part I: Applying Chromatography Experience

The Problem: The United States Food & Drug Administration (FDA) regulates food and related commercial activities such as food labeling and advertising. This includes any additives added to the food, such as food coloring, preservatives, flavorings, etc. Although food products must be tested and then approved by the FDA prior to entering the US market, it is wise to occasionally double-check what is on the grocery store shelf to ensure that companies are still following the rules laid out by the FDA (i.e. not adding in anything undeclared, food stays good until the expiration date, chemicals are shown to be safe to eat by humans, etc.).

In that vein, you will analyze a collection of M&M candies for the presence of an FDAapproved yellow dye known as "FD&C Yellow 5" (common name: tartrazine, IUPAC: ridiculously long). Tartrazine is a common food dye but can cause allergic reactions in certain people, so its presence must always be listed on a label. The FDA seizes any foods or medications containing undeclared tartrazine. An anonymous source has informed you that they found an M&M package without tartrazine on the label, and they sent a few M&Ms of each color to your lab for testing. You are investigating to see if any tartrazine is present.



- 1. Identify what your sample(s) is/are, specifically (i.e. will you be testing an entire M&M of each color, just the shells, just the inner chocolates, just certain M&M colors, etc.). Rationalize your decision.
 - Remember that your time and resources are precious, but you also must be able to confidently show whether the tartrazine is present in an M&M candy.

Sample(s):

Rationale:

2. When comparing your intended sample to the Glowmatography experiment's glowstick, do you have more or less quantity of sample to work with now?

(1 point)

(1 point)

3. Considering your answer above, do you think that a stick of chalk will be an appropriate stationary phase for the M&Ms? Explain why or why not.

(1 point)

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4. If you were to use a stationary phase other than chalk, would you want a thinner/flatter or a wider/deeper item to be the stationary phase for this experiment? Explain which you would prefer and why.

(1 point)

5. For Glowmatography, you pipetted a glowing solution directly onto the chalk. When thinking about testing the M&M candies, what is one practical issue you anticipate encountering in terms of physically loading your intended sample onto a stationary phase?

(1 point)

6. Considering your answer above, propose a way to work around this practical issue in order to effectively load your sample onto a stationary phase.

(1 point)

7. Suppose you loaded your samples onto an appropriate stationary phase and set up your chromatography experiment. The mobile phase is running, the samples' dyes are separating, and everything seems to be going smoothly until you realize that you didn't plan how you would determine if one of the dye components is tartrazine! Sure, tartrazine is yellow, but lots of yellow molecules are used as food dyes.

Propose something you could do to enable yourself to identify whether a yellow dye component of your samples is actually tartrazine. In addressing this hypothetical situation, you are limited to only chromatographic techniques but can have any chemical you can imagine.

(1 point)

8. Suppose you and a peer both run a tartrazine detection chromatography experiment, but your peer uses a stationary phase that's twice as long as yours. Because your stationary phases are different lengths, you are struggling to visually compare your two experiments afterward to see if you both got the same results. Brainstorm a way to use <u>numbers</u> to compare your results quantitatively to discover if you got the same results.

(1 point)

Part II: Writing the Procedure

9. Propose using clear, unambiguous language exactly what data/results/observations you will need to collect to determine whether a sample contains tartrazine (and be able to convince another person that you are right!).

(2 points)

- 10. Suppose your stationary phase was a piece of paper instead of chalk. Considering your reflections in Part I and in the Glowmatography experiment, sketch a diagrammatic drawing to propose a paper-based chromatography experimental set-up for your samples.
 - Remember that your primary goal is to identify whether a sample has tartrazine.
 - Include labels and any descriptive language necessary so that it is clear what each part of the set-up is.
 - Indicate how you will load the sample.

(4 points)

11. Make a flowchart that diagrams what you consider to be the important steps/parts of a tartrazine detection experiment.

(2 points)

- 12. Draft a procedure for this paper-based chromatography experiment (complete *just* the "Instructions" section for now do the other sections after this step).
 - Use the template provided on Schoology. The points earned will be recorded here in Formative. You will be able to continue editing your Schoology procedure in the future.
 - List (with numerical bullet points) all the steps you will take.
 - Be clear to avoid confusion. Include enough detail, tips, and reminders that you won't forget something important when running the experiment.
 - Your data collection steps should also be explicitly incorporated into the procedure. It would be a disaster to neglect collecting the data because you didn't have a procedural step that told you to record data!
 - In addition to your own data collection steps, include steps that instruct you to take a picture at the following points (for the instructor to view later):
 - i. The sample preparation process
 - ii. The sample loading process
 - iii. The loaded stationary phase before placing it in the mobile phase
 - iv. The stationary phase with the mobile phase halfway up the paper
 - v. The stationary phase after elution and analysis

(10 points)

□ Check this box when your "Instructions" section is ready for instructor review. The "Instructions" section will be marked approved by awarding the points.

- 13. After your rough draft "Instructions" section is approved, finish the rest of the procedure template.
 - Prepare a spot to record all data. This could be a table, a box for observations, a blank area for an uploaded picture, etc.
 - List all materials needed (but don't gather anything until the procedure is approved).
 - Propose appropriate "Safety, Waste Disposal, and Clean-Up" guidelines which must address, at a bare minimum, ...
 - i. the Personal Protective Equipment you will use
 - ii. hazards and risks of the items you will work with and any necessary risk mitigation
 - iii. how you will appropriately clean up afterward

(5 points)

□ Check this box when your entire procedure is ready for instructor review. The procedure will be marked approved by awarding the points.

Note:

- > Your proposal must be approved before you can begin the experiment.
- You might need to adjust your proposal according to instructor feedback before earning approval and points.
- > You may adjust your procedure again at any later time.

Part III: Running the Experiment

14. After your entire procedure is approved, conduct a trial run(s) of your experiment.

- Use a narrow slip of filter paper to test out your experiment with one or two samples. This will enable you to practice physically doing the steps and expose potential issues with your procedure and data collection plan.
- Be careful not to use up all your supplies in one run... An experimenter almost • always needs multiple rounds to finally get the experiment working well.
- Make any necessary adjustments to your procedure if you notice an issue (it would be unusual to *not* find something that should be tweaked...).
- It may be prudent to conduct another trial run after procedural adjustments to ensure vou have fixed any issues before doing the full experiment.

(5 points)

- □ Check this box after completing your trial run and making any adjustments. [Student] My adjustments were... (write "NA" if no adjustments)
- 15. Conduct the experiment.
 - Enter all your data into your Schoology procedure document. Record any • procedural deviations (intentional or accidental) or problems that you encountered during the experiment in the space below.
 - It is ok to intentionally deviate from your procedure if you have reason to believe that such a deviation is necessary or beneficial. It is also normal to accidentally deviate as well. You must, however, note any deviations.

(5 points)

□ Check this box when you have completed your experiment.

[Student] I experienced the following deviations from my plan: (or "NA")

Note: You should make sure you got the results you need before throwing out supplies.

Clean-Up Approval Signature (5 points)	Name of Signer:
	Relationship to Student:

16. Submit pictures of your experiment at the following stages:

- The sample preparation process i.
- The sample loading process ii.
- iii. The loaded stationary phase before placing in the mobile phase
- The stationary phase with the mobile phase halfway up the paper (1 point) iv. (1 point)
- v. The stationary phase after elution and analysis

(1 point)

(1 point)

(1 point)

Part IV: Analyzing the Results 17. The stationary phase for the experiment was (1 po						
18	3. The mobile phase t	for the experim	nent was			(1 point)
19	9. Was the mobile ph	ase polar or no	onpolar?			(1)
	polar	nor	polar			(1 point)
20	20. Paper is made of cellulose which is a polymeric matrix of rings containing ethers and alcohols (on the right). Is the paper polar or nonpolar?				НО	
	polar	nonpolar		HO OH OH	Loo L	
21	. Which M&M(s) co	ontained more	than one dye?		0 .40	(1 point)
red	orange	yellow	green	blue	brown	(1 point) none
22	2. Which M&M cont	ained the most	polar dye?			
red	orange	yellow	green	blue	brown	(2 points)
Explain how you know:						
23. Which M&M contained the least polar dye?					(2 points)	
red	orange	yellow	green	blue	brown	(- points)
	Explain how you know:					

24. Insert a picture of the analyzed stationary phase after elution (with all the circled spots in pencil). Write in the R_f values for each spot. Annotate the picture to show the lengths (their placement and their actual cm values) that you used in calculating R_f values.

(3 points)

25. For one of the spots, write the calculations you conducted to generate that R_f value (to provide a sample calculation for the instructor).

(1 point)

26. Did any of the M&M candies likely contain tartrazine? If so, circle them below. Explicitly refer to supporting data you collected during the experiment to rationalize your answer.

red	orange	yellow	green	blue	brown	(2 points) none
	Rationale:					

- 27. Propose one potential limitation surrounding the type of data you collected that could potentially weaken your conclusion by calling into question whether your data *reeaallly* proves tartrazine's presence/absence.
 - For this question, assume that nothing went wrong with the experiment and that you got great-looking data. This question is asking what could go wrong with interpretation and jumping to conclusions.

(2 points)

Part V: Extensions

28. If you left the paper in the mobile phase for an hour instead of removing it once the mobile phase is about the reach the top of the paper, what would your paper look like at the end of the hour? How would this affect your results?

(2 points)

29. If you wanted to switch the order of elution for the dyes based on polarity, how might you (in general terms) alter your experiment to accomplish that? Explain your reasoning.

(2 points)

(2 points)

30. If you did switch the order of elution described in the question above, which M&M's colored dye would likely be the highest up on the paper?

red	orange	vellow	green	blue	brown
104	orange	<i>j</i> e ¹¹ 0 <i>i</i>	Breen	orae	010 111

31. If you wanted to *collect* the tartrazine you separated in this experiment to use in a future experiment, do you think you would realistically be able to do that with the experiment you just ran? Explain your reasoning.

(2 points)

32. Related to the question above, if you wanted to collect sizable amounts of tartrazine from M&M's, brainstorm at least three theoretical changes you would need to make to your experimental set-up in order to realistically meet your goal. You can be somewhat vague.

(3 points)

Lab 9: Isolation from Plants Organic Chemistry (82 points)

Purpose: to (1) collect isolated colored molecules from a mixture and (2) build on previous chromatography experiences to independently solve both problems experimentally

Introduction: Chromatography enables separation, identification, and isolation of substances. Previously, you engaged in (1) a "glowmatography" experiment to observe separation of a glowstick solution and (2) a thin-layer chromatography experiment to identify tartrazine in M&M candies. In both cases, you conducted chromatography and observed separation, but neither experiment featured *collection* of the separated substances afterward. However, in the organic chemistry lab, experimenters often use chromatography not only to separate and identify molecules from a mixture but also to then collect the purified substance for future experiments. This process is often referred to either as "isolation" or "collection". In this lab, you will build upon previous chromatography experiments to independently design a complete chromatography experiment to solve a hypothetical problem regarding chromatographic isolation.

Due:

- 1. Part I: Applying Chromatography Experience
 - by XXXX
 - on Formative
- 2. Part II: Writing the Procedure
 - by XXXX
 - on Formative (questions, instructions, grade) and Schoology (procedure template)
- 3. Part III: Running the Experiment
 - by XXXX
 - on Formative (questions, instructions, grade) and Schoology (procedure and data)
- 4. Part IV: Analyzing the Results
 - by XXXX
 - on Formative

Instructions: Complete the following steps, and answer all questions. Due to the nature of the investigation, the questions are integrated into the procedure instead of just in a dedicated Questions section.

- You must complete Part I before starting Part II, Part II before starting Part III.
- Instructor feedback and approval is required before conducting the experiment.
- Not all the pages of this overall packet are shown at once in Formative; the additional pages will be added as you advance through the feedback and approval process.

Collaboration: no collaboration is allowed on this assignment unless specified – You may refer to the instructor, class notes, and returned graded work only.

Part I: Applying Chromatography Experience

The Problem: Throughout history and still in modern day, humans have obtained dyes, medicines, useful materials, etc. from nature. This often involves isolating desired substances (ex: the dye, the medicine, etc.) from raw natural materials (ex: tree bark, mineral deposits, fungi, etc.). You are an organic chemist working for a company which produces "organic", "natural" beauty products (shampoo from seaweed, perfumes from flowers, etc.). The company wants a new set of dyes to use in their beauty products, and they also need dyes with a variety of polarities for good mixing (i.e. a polar dye to mix with a polar beauty product vs. a nonpolar dye to mix with a nonpolar product). However, in keeping with the company's mission of using all-naturally sourced materials and no lab-produced substances, they want to obtain the new dyes 100 % from nature in order to appeal to their consumers and to keep their advertising honest. You decide to center your efforts on extracting dyes from plants, knowing that plants produce a wide range of colored molecules. The company leaders say that, in order to invest in any procedure you create, they need proof that the procedure is capable of producing real quantities (approximately a half teaspoon or more) of a purified dye.

1. State your end goal(s).

(1 point)

2. Explicitly identify how your goal(s) is/are different from the goals in the glowmatography and TLC experiments.

(1 point)

- 3. Propose using clear, unambiguous language exactly what data/results/observations you will need to collect AND why that data is necessary.
 - Think about both (1) the results you need at the end of the day to address your goal AND (2) what type of information you need to gather/learn *in order to conduct your experiment* to obtain those end results.
 - List as many types of data you think you might need.

(3 points)

4. Make a flowchart that diagrams the important steps/parts of your experiment.

(2 points)

- 5. Sketch a diagrammatic drawing(s) to propose an experimental set-up(s).
 - If you are unsure about what types of materials you might be able to use to conduct your experiment, just draw a hypothetical set-up that you would *like* to have, and your instructor may give you feedback to guide you regarding real supplies.
 - Include labels and any descriptive language necessary so that it is clear what each part of the set-up is.
 - Indicate how you will load the sample, run the experiment, and collect results.
 - Include all bits of detail that you can think of which might be important for you to address your goal.

(4 points)

Part II: Writing the Procedure

- 6. Draft a procedure, including the materials list, safety/waste disposal/clean-up, and data collection.
 - Use the template provided on Schoology. The points earned will be recorded here in Formative. You will be able to continue editing your Schoology procedure in the future.
 - In addition to your own data collection steps, include steps that instruct you to take a picture at the following points:
 - i. The sample preparation process
 - ii. The sample loading process
 - iii. The loaded stationary phase before adding the mobile phase
 - iv. The stationary phase with the mobile phase added, after a few minutes
 - v. The stationary phase after the experiment
 - vi. The collected dye(s)

(15 points)

□ Check this box when your entire procedure is ready for instructor review. The procedure will be marked approved by awarding the points.

Note:

- Your proposal must be approved for safety before you can begin the experiment, but it will not necessary be vetted for quality (meaning, your procedure is not guaranteed to work).
- You might need to adjust your proposal according to instructor feedback before earning approval and points.
- > You may adjust your procedure again at any later time.

Part III: Running the Experiment

- 7. After your entire procedure is approved, conduct a trial run(s) of your experiment.
 - Be careful not to use up all your supplies in one run... An experimenter almost always needs multiple rounds to finally get the experiment working well.
 - Make any necessary adjustments to your procedure if you notice an issue (it would be unusual to *not* find something that should be tweaked...).
 - If prudent, you may want to conduct another trial run after procedural adjustments to ensure you have fixed any issues before doing the full experiment.

(10 points)

□ Check this box after completing your trial run and making any adjustments. [*Student*] My adjustments were... (write "NA" if no adjustments)

- 8. Conduct the experiment.
 - Enter all your data into your Schoology procedure document. Record any procedural deviations (intentional or accidental) or problems that you encountered during the experiment in the space below.

(10 points)

 \Box Check this box when you have completed your experiment.

[Student] I experienced the following deviations from my plan: (or "NA")

Note: You should make sure you got the results you need before throwing out supplies.

Clean-Up Approval Signature (5 points)	
	Name of Signer:
	Relationship to Student:

9. Submit pictures of your experiment at the following stages:

The sample preparation process	(1 point)
The sample loading process	(1 point)
The loaded stationary phase before adding the mobile phase	(1 point)
The stationary phase with the mobile phase added, after a few minutes	(1 point)
The stationary phase after the experiment	(1 point)
The collected dye(s)	(1 point)
	The sample loading process The loaded stationary phase before adding the mobile phase The stationary phase with the mobile phase added, after a few minutes The stationary phase after the experiment

Part IV: Analyzing the Results

10. Did you successfully address the goal(s)? Provide evidence to support your answer.

(3 points)

11. Could one of your plant's dyes should be used with a polar beauty product? A nonpolar beauty product? Briefly rationalize.

(2 points)

12. Complete the separate assignment titled "Post-Lab Follow-Up" before meeting with your instructor in the next step. The assignment is graded based on completion.

(10 points)

□ Check this box when you have completed the "Post-Lab Follow-Up" questions.

- 13. Schedule a time to meet one-on-one with your instructor to discuss these past three experiments.
 - Your instructor will ask you questions during the meeting to prompt you to reflect on the process of experimental design and gaining laboratory competency. Your responses are not graded, but conducting the meeting is awarded completion points since self-reflection is a very important learning process for students.

(10 points)

 \Box Check this box when you have scheduled your meeting time.

Instructor Narrative about Student Performance

- 1. Lab 1: Of the students who submitted complete labs, all collected appropriate balloon-popping data, accurately defined hazards/risks from the assigned reading, and identified a variety of safe/unsafe situations in the videos. When asked to identify real-life examples of hazards and risks, they all accurately identified a hazard but 4 students also conflated risks with secondary hazards (for example, if the hazard was an "unattended candle", saying that the risk was "the candle will accidentally light something else on fire" instead of reporting the probability that the candle will catch something on fire). All students successfully proposed a risk mitigation step for their hazard and predicted physical and chemical hazards they could expect to encounter in upcoming labs. Every student also reported either an increased heart rate, surprise, jumping, and/or momentary fear when the balloons popped (despite expecting it) but shared in class that they learned something valuable by reflecting on their emotional response. This human reaction fueled a productive discussion on how most accidents in chemistry laboratories are slips, trips, and falls and that a calm response to an accident is of paramount importance. The first lab's role as a safety assignment hopefully reinforced that safety is the most important part of laboratory work. Later, on the following unit test, every student proposed appropriate ways to keep themselves safe in a chemistry laboratory.
- 2. Lab 2: All students collected reasonable data (though values fluctuated between students), and almost all students successfully used the data to reason out an appropriate response to a question about what the data suggested about the strengths of attractive IMFs for the hydrogel polymers when in acidic vs. neutral vs. basic conditions. Students successfully discussed how hydrogen bonding causes stronger attractions and therefore leads to shorter distances between polymers, resulting in observed contraction of the lens on a visible, macroscopic scale when in an acidic solution which features protonated carboxylic acids. A couple students presented incomplete discussions, though, of what happens in basic conditions to result in lens expansion (for example, discussing only the diminishing of hydrogen bonding due to deprotonation but not also discussing the resulting electrostatic repulsion of negatively charged species). Every student correctly predicted that, in a (theoretical) more strongly basic solution where all the polymer's alcohols can also be fully deprotonated, you should expect the lens to expand more.
- 3. *Lab 3:* Students correctly identified what the model kit pieces represented. Students created models accurately for the most part but occasionally forgot an atom or did not indicate stereochemistry appropriately. Almost every student correctly interpreted a set of models to discover the concept of free rotation and ring strain. Later, on the following unit test, students correctly (1) distinguished between structures with just rotated components versus structures which were stereoisomers, (2) described free rotation versus stereoisomerism, and (3) displayed good success as a group with naming and drawing molecules based on names, models, and structural drawings.
- 4. *Lab 4:* Since most students did not collect distillate due to the experimental problems previously discussed, the data collection and interpretation questions were altered to merely

record observations on the process (rather than compare distillate fractions) and predict what differences might have been noticed between different fractions. Students overwhelmingly correctly predicted potential issues that would arise due to hypothetical procedural alterations (such as not getting flush connections with the jar or not keeping the collection vial cold). In class, students compared structures of their plants' major essential oil components and identified structural and electronic similarities between their molecules as well as key differences when compared to other common plant molecules which are not volatile (such as cellulose).

- 5. *Lab 5:* Students collected reasonable data/observations, although not everyone saw reignition of the candle flame. Students overall showed an understanding of the dangers of hot wax dripping onto their fingers and made good attempts at rationalizing why the flames extinguished and why the water rose in the test tube. However, they failed to remember from general chemistry that copper is a conductive material, and so a few students tried to rationalize that a lack of oxygen was playing a role in extinguishing the flame instead of the copper wire. Most students correctly rationalized that the change in temperature and, consequently, pressure caused the movement of the water into the tube.
- 6. *Lab 6:* Students collected uniform results. They easily interpreted the results of the liquidliquid separation and the density table to decide miscibility and which layer was which, even though they had not been taught to do this interpretation in class. They accurately predicted limitations to liquid-liquid extraction, such as when the solvents are highly miscible or when the desired product and contaminant have similar solubilities. They accurately used both their observations of smell and knowledge of structure to determine that the cinnamaldehyde had been successfully extracted in the solid-liquid extraction.

Student performance with Labs 7-9 will be described separately in the future as a project on experimental design.

Student Surveys

Mid-Course Survey: administered between Labs 4 and 5 via an extra credit Google Forms survey – non-anonymous; lab feedback questions followed by general course feedback questions

Survey for Third Month of Organic Chemistry Class

Purpose: to see how you feel the semester is going and what you think about the labs

Instructions: Answer the questions with your gut instinct, and don't overthink anything. You can update your response until the due date if something else occurs to you after you submit. You do not need to answer in complete sentences, just make sure I can understand what you mean. Some questions are required, and some are not.

Optional extra credit: 5 points in Labs category Your email address is being collected to allocate extra credit.

Expected completion time: approximately 10 minutes

Due: by 11:59pm Friday, Nov. 20th
* Required

1. Email address *

Labs (Part I of II)

2. Rate your level of agreement regarding chemistry labs in general, regardless of whether at-school or athome labs. *

Mark only one oval per row.

	Strongly Disagree	Disagree	Neutral	Agree	Strongly Agree
I like doing l abs.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
I would rather do a problem set or other homework instead of a lab.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
I would rather do a computer simulation of a lab rather than do the physical lab itself.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
I would rather watch a video of someone else doing the lab or receive data from someone else doing the lab rather than do the physical lab myself.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
I'd be fine not doing any labs or looking at lab-related data/videos/simulations/etc. at all this semester.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc

3. Classify the labs done so far. You can check multiple boxes for each row/column or leave a row/column blank. *

Check all that apply.

	Fun	Difficu l t	Interesting	Worked well	Boring	Educational	Frustrating	Worthwhile
Lab 1 (Unsafe Lab Practica l)								
Lab 2 (Hydrogels)								
Lab 3 (Molecular Modeling)								
Lab 4 (Essential Oil Distillation)								

4. Rate your level of agreement regarding the at-home labs so far. *

Mark only one oval per row.

	Strong l y Disagree	Disagree	Neutral	Agree	Strong l y Agree	Not Applicable
I feel safe doing the labs.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
I can follow the lab procedures just fine.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
The labs do NOT feel relevant to the curriculum.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
My learning was benefited by completing the labs.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
I do NOT know what to do (or have trouble doing what I need to) to be safe during a lab.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
The labs are worth the effort.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc
The labs help me do better on non-lab assignments.	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc	\bigcirc

5. How often do you read the "Safety, Waste Disposal, & Clean-Up" section before starting a lab? *

Mark only one oval.

🔵 Always

Sometimes

Never

How are the labs going over	all?
---	------

Is there anything that I as your teacher should know about the labs?

End-of-Semester Survey: administered via Google Forms after Lab 9

Classify the labs. You can check multiple boxes for each row/column or leave a row/column blank. *

Check all that apply.

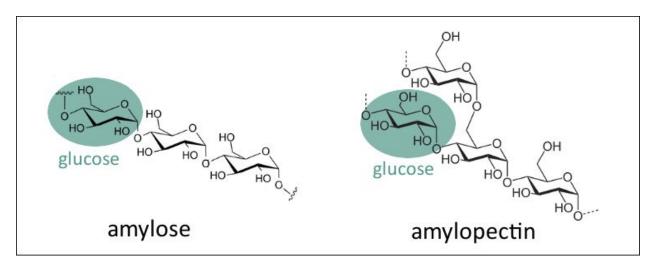
	Fun	Difficult	Interesting	Worked well	Boring	Educational	Frustrating	Worthwhile
Lab 5 (Candles)								
Lab 6 (Extractions)								
Lab 7 (Glowmatography)								
Lab 8 (Detection of Tartrazine)								
Lab 9 (Isolation from Plants)								

Lab 19: Polymerization from Potato Starch (37 points total)

Introduction

Plastics come from chemistry laboratories and are made from byproducts from petroleum refining. To reduce the world's reliance on petroleum, we need to devise effective ways to generate plastics which use eco-friendly, renewable building blocks. In this activity, you will make a bioplastic from potato starch. Potato starch contains two main polymers, amylose and amylopectin, which are long chains of glucose units joined together. On the first day, you will make a bioplastic. On the second day, you will test your bioplastic's physical properties and then videoconference with people from other groups to evaluate differences in types of plastics.

- Amylose: "straight" (aka unbranched) polymer chains of glucose units
- Amylopectin: "branched" polymer chains of glucose units



Due: by the start of the class after Day 2

> Allocate roughly 1 hour to complete the Day 1 hands-on portion, including reading time.

Safety, Waste Disposal, & Clean-Up

- 1. Wear splash goggles. Gloves are not necessary. Wash your hands at the end of lab.
- 2. Ensure you have an adult supervisor present in the area in case of an emergency. Work in a clear, clean space in the kitchen during daylight and when the kitchen is not busy, being used to cook food, or will need to be used during your experiment. Do not involve younger siblings or other people in your experiment.
- 3. Exercise case when operating your oven. Never leave the oven unattended.
- 4. Treat any burns immediately with running cold water; depending on burn severity, you may need more medical attention.
- 5. Always use oven mitts or some other insulating material to touch potentially-hot items. Remember that you often can't tell if an object is hot just by looking at it.
- 6. Do NOT put the aluminum dish in a microwave. Metal must NEVER go in a microwave. Dangerous arcing will occur.

- 7. NaOH and HCl are a strong base and strong acid, respectively, but are significantly diluted in this lab. The dilution lowers their hazard levels considerably, but they are still skin irritants. Rinse off with cold water and soap in case of contact. Rinse your eyes for several minutes with cold water if you get either in your eyes.
- 8. Glycerol is used in many cosmetics but can potentially be irritating to the skin when in concentrated form. Rinse off with cold water and soap in case of contact.
- 9. When done, rinse all kitchen supplies with water and dish soap. Put all supplies back clean and dry in a neat, organized manner. Throw away the bioplastic and the emptied Eppendorf vials in the trash.
- 10. Obtain Clean-Up Approval for each day of the experiment **before leaving the kitchen**. After Day 2, upload a picture of your supervisor's initials for both days.

Clean-Up Approva	Signature (5 points)	
<u>Day 1</u>	<u>Day 2</u>	Name of Signer:
		Relationship to Student:

Materials: Contact your instructor about alternative materials if you're missing an item.

All amounts are approximate since residual chemicals will remain in the containers. Try to get as much out as possible, but never touch chemicals with skin.

Everyone:	~2.5-3.0 g po	tato starch (given)	aluminum dish (given)		
	teaspoon	timer (minutes)	toaster oven/oven		
	blunt stirring	utensil (ex: wooden ch	opstick or blunt pencil but not a knife)		
	flat, oven-safe	e dish (ex: Pyrex)	small square of wax/parchment pape		

Group dependent:1.5 mL 0.20 M HCl_{aq} (given, blue vial, groups B/C/D)1.5 mL 0.20 M NaOH_{aq} (given, orange vial, groups B/C/D)~1.2-1.3 mL glycerol (given, green vial, groups C/D)gauze pad/toilet paper/tissue (group D)

Instructions: Conduct the following procedure, fill out the table, and answer all questions. Follow all "Safety, Waste Disposal, & Clean-Up" guidelines.

Assigned Group

<u>Day 1:</u> Use the procedure correspond to your group letter. Group A

- 1. Preheat a toaster oven or oven to 300 °F. Place wax or parchment paper on the bottom of an oven-safe dish.
- 2. Add \sim 5 teaspoons tap water to the aluminum dish. Add the potato starch. Use a thin, blunt stirring utensil to carefully mix them well.
- 3. Place the dish in the preheated oven.
- 4. For ~ 15 minutes, constantly and carefully watch the dish. Once every two minutes, open the oven door, and quickly but *carefully* stir the dish with the blunt utensil to homogenize the mixture.
 - a. Take great care not to spill the mixture.

- b. Be quick about this. Every moment the oven door is open, the temperature drops.
- c. If your mixture starts to bubble, turn down the temperature to 250 °F or lower. If your mixture stills looks extremely watery after 15 minutes, turn up the temperature to 350 °F and continue 5-10 more minutes. Do not open the door during this time.
- 5. Carefully remove the dish from the oven. *Thoroughly* stir the mixture until it looks homogenous.
- 6. *While the mixture is still warm*, pour it onto a clean, dry, smooth, flat oven-safe dish. Push the mixture around with your thin utensil so that it forms an even coating. Bake it in an oven at 250 °F until it looks mostly dry.
- Leave it to cool and finish drying overnight in a safe area where no one will touch it. Place a note next to the oven-safe dish reading "CHEMISTRY EXPERIMENT – DO NOT TOUCH".
- 8. Follow "Safety, Waste Disposal, & Clean-Up" guidelines. Obtain Clean-Up Approval.

Group B

- 1. Preheat a toaster oven or oven to 300 °F. Place wax or parchment paper on the bottom of an oven-safe dish.
- 2. Add \sim 5 teaspoons tap water to the aluminum dish. Add the potato starch. Use a thin, blunt stirring utensil to carefully mix them well.
- 3. Add the HCl. Stir to mix in well.
- 4. Place the dish in the preheated oven.
- 5. For \sim 15 minutes, constantly and carefully watch the dish. Once every two minutes, open the oven door, and quickly but *carefully* stir the dish with the blunt utensil to homogenize.
 - a. Take great care not to spill the mixture.
 - b. Be quick about this. Every moment the oven door is open, the temperature drops.
 - c. If your mixture starts to bubble, turn down the temperature to 250 °F or lower. If your mixture stills looks extremely watery after 15 minutes, turn up the temperature to 350 °F and continue 5-10 more minutes. Do not open the door during this time.
- 6. Carefully remove the dish from the oven. *Thoroughly* stir the mixture until it looks homogenous. Add the NaOH while stirring.
 - a. It is crucial to add the NaOH in order to neutralize the HCl.
- 7. *While the mixture is still warm*, pour it onto a clean, dry, smooth, flat oven-safe dish. Push the mixture around with your thin utensil so that it forms an even coating. Bake it in an oven at 250 °F until it looks mostly dry.
- 8. Leave it to cool and finish drying overnight in a safe area where no one will touch it. Place a note next to the oven-safe dish reading "CHEMISTRY EXPERIMENT DO NOT TOUCH".
- 9. Follow "Safety, Waste Disposal, & Clean-Up" guidelines. Obtain Clean-Up Approval.

Group C

- 1. Preheat a toaster oven or oven to 300 °F. Place wax or parchment paper on the bottom of an oven-safe dish.
- 2. Add \sim 5 teaspoons tap water to the aluminum dish. Add the potato starch. Use a thin, blunt stirring utensil to carefully mix them well.
- 3. Add the HCl and glycerol. Stir to mix in well.
- 4. Place the dish in the preheated oven or toaster oven.

- 5. For \sim 15 minutes, constantly and carefully watch the dish. Once every two minutes, open the oven door, and quickly but *carefully* stir the dish with the blunt utensil to homogenize.
 - a. Take great care not to spill the mixture.
 - b. Be quick about this. Every moment the oven door is open, the temperature drops.
 - c. If your mixture starts to bubble, turn down the temperature to 250 °F or lower. If your mixture stills looks extremely watery after 15 minutes, turn up the temperature to 350 °F and continue 5-10 more minutes. Do not open the door during this time.
- 6. Carefully remove the dish from the oven. *Thoroughly* stir the mixture until it looks homogenous. Add the NaOH while stirring.
 - a. It is crucial to add the NaOH in order to neutralize the HCl.
- 7. *While the mixture is still warm*, pour it onto a clean, dry, smooth, flat oven-safe dish. Push the mixture around with your thin utensil so that it forms an even coating. Bake it in an oven at 250 °F until it looks mostly dry.
- 8. Leave it to cool and finish drying overnight in a safe area where no one will touch it. Place a note next to the oven-safe dish reading "CHEMISTRY EXPERIMENT DO NOT TOUCH".
- 9. Follow "Safety, Waste Disposal, & Clean-Up" guidelines. Obtain Clean-Up Approval.

Group D

- 1. Preheat a toaster oven or oven to 300 °F. Place wax or parchment paper on the bottom of an oven-safe dish.
- 2. Add \sim 5 teaspoons tap water to the aluminum dish. Add the potato starch. Use a thin, blunt stirring utensil to carefully mix them well.
- 3. Add the HCl and glycerol. Stir to mix in well.
- 4. Place the dish in the preheated oven or toaster oven.
- 5. For ~ 15 minutes, constantly and carefully watch the dish. Once every two minutes, open the oven door, and quickly but *carefully* stir the dish with the blunt utensil to homogenize.
 - a. Take great care not to spill the mixture.
 - b. Be quick about this. Every moment the oven door is open, the temperature drops.
 - c. If your mixture starts to bubble, turn down the temperature to 250 °F or lower. If your mixture stills looks extremely watery after 15 minutes, turn up the temperature to 350 °F and continue 5-10 more minutes. Do not open the door during this time.
- 6. Carefully remove the dish from the oven. *Thoroughly* stir the mixture until it looks homogenous. Add the NaOH while stirring.
 - a. It is crucial to add the NaOH in order to neutralize the HCl.
- 7. *While the mixture is still warm*, place an unfolded gauze pad OR a leaf of toilet paper OR a tissue on a clean, dry, smooth, flat oven-safe dish. Pour the warm mixture on top of the gauze pad/toilet paper/tissue. Push the mixture around with your thin utensil so that it forms an even coating. Bake it in an oven at 250 °F until it looks mostly dry.
- 8. Leave it to cool and finish drying overnight in a safe area where no one will touch it. Place a note next to the oven-safe dish reading "CHEMISTRY EXPERIMENT DO NOT TOUCH".
- 9. Follow "Safety, Waste Disposal, & Clean-Up" guidelines. Obtain Clean-Up Approval.

Day 2: Before the class meeting

- 1. Take a picture of your *dry* bioplastic *before class*, and attach the picture to Table 1.
- 2. Evaluate the physical properties (ex: flexibility, elasticity, tensile strength, etc.) of your *dry* bioplastic *before class*. Record observations in Table 1.

Picture	Observations
	I am in Group

Table 1: Observations About Individual Bioplastic (2 points/box)

During the class meeting:

- 3. During class, video conference with other people in your group from your section.
 - a. Determine if you all observe the same properties or if there are discrepancies.
 - b. Make a note of similarities and differences in Table 2.
- 4. Then, you will video conference with people from other groups during class.
 - a. Show your collaborators your bioplastic. Demonstrate its properties.
 - b. Record observations about the bioplastics from each of the other groups.
- 5. Throw out the bioplastic, and clean up any remaining mess. Obtain Clean-Up Approval.

Table 2: Observations About Physical Properties of Groups' Bioplastics (2.5 points/observation box, 0.5 points/collaborator box)

Bioplastic	Observations
А	
Group A Co	ollaborator(s):
В	
Group B Co	ollaborator(s):
С	
Group C Co	ollaborator(s):

D

Group D Collaborator(s):

Collaborators (not including assigned groups): _____

Questions

1. What did each group do differently from Group A (control group)? (1 point each, 3 points total)

Group B:

Group C:

Group D:

2. *Succinctly* summarize the similarities and differences in the observed properties of the four bioplastics.

(4 points)

3. Postulate *how* the HCl treatment affects the bioplastic on a molecular level (think about the difference between amylose and amylopectin).

(3 points)

4. Postulate *how* the glycerol affects the bioplastic on a molecular level. Look up the structure of glycerol if you want, but think more about glycerol's physical properties.

(3 points)

5. Postulate *how* the gauze pad/toilet paper/tissue affects the bioplastic on a molecular level.

(3 points)

Lab 20: Solubility in the Kitchen (39 points total)

Introduction

Solubility is one of many physical properties which is highly dependent on intermolecular forces. The phrase "like dissolves like" arises from the tendency for solutes to dissolve in solvents with similar IMFs. But each solvent can "accommodate" only so much of a certain solute before it can no longer dissolve any more. In this lab, you will investigate (1) "like dissolves like" and (2) the phenomenon of supersaturation using simple household materials.

Due: by the start of class on XXXX

Allocate roughly ¼ hour to complete the balloon hands-on portion and ½ hour to complete the rock candy portion, including reading time.

Safety, Waste Disposal, & Clean-Up

- 1. Wear splash goggles. Gloves are not necessary. Wash your hands at the end of lab.
- 2. Ensure you have an adult supervisor present in the area in case of an emergency. Work in a clear, clean space in the kitchen during daylight and when the kitchen is not busy, being used to cook food, or will need to be used during your experiment. Do not involve younger siblings or other people in your experiment.
- 3. Treat any burns (including hot water burns) immediately with running cold water; depending on burn severity, you may need more medical attention.
- 4. Always use oven mitts or some other insulating material to touch potentially-hot items. Remember that you often can't tell if an object is hot just by looking at it.
- 5. Be careful when popping the balloon that you don't get hurt. Be prepared for a loud noise.
- 6. When done, rinse all supplies with water. Throw out the popped balloon pieces; make sure to get all the pieces up so that a pet or infant doesn't find any later. Put all supplies back clean and dry in a neat, organized manner.
- 7. Obtain Clean-Up Approval for each day of the experiment **before leaving the kitchen**. After Day 2, upload a picture of your supervisor's initials for both days.

Clean-Up Approval Day 1	Signature (5 points) Day 6/Last Day	Name of Signer:		
		Relationship to Student:		

Materials: Contact your instructor about alternative materials if you're missing an item. *Popping Balloon:* balloon (preferably a <u>water balloon</u> or <u>cheap</u> latex) clementine/orange

Rock Candy:3 glass cups/ceramic cups/canning jarssugar (\geq 3 cups)microwavetablespoonmeasuring cupspoonfood coloring (optional)wooden skewer/chopstick + clothespin OR thick thread + pencil + paperclip + tape

Instructions: Conduct the following procedures, fill out the tables, and answer all questions. Follow all "Safety, Waste Disposal, & Clean-Up" guidelines.

Part I: Balloon

- 1. Record what type of balloon you are using (water balloon, normal party balloon, etc.) in Table 1.
- 2. Inflate the balloon with air (from your lungs). Get it as big as you can (without popping it....). Tie it off.
- 3. Place it somewhere where it won't roll around but also where it won't hurt you if it pops (aka don't hold it in your hand or hug it). Tell anyone around to prepare for a loud noise.
- 4. Peel an orange.
 - You can also substitute other types of citrus for the orange, but oranges work best. A lemon is probably your next-best citrus. Record the type of citrus in Table 1.
- 5. Rub the inside part of the orange peel on a spot on the balloon until it pops. Be careful it doesn't pop on your hand and hurt.
 - If it doesn't pop after rubbing for half a minute, try the outer skin. Then try squeezing the orange above the balloon so that some juice drops onto the balloon.
- 6. If it doesn't pop after a minute, move on. Eat the orange. Yum yum.

Type of Balloon Type of Citrus Did it pop?						

Table 1: Balloon Data (1 point/box)

Part II: Rock Candy

- 1. Prepare three glass containers according to one of the methods below:
 - *Option 1*: Use a clothespin (perpendicular across the cup/jar opening) to suspend a wooden skewer/chopstick ~ 1 inch above the bottom of the cup/jar.
 - *Option 2*: Cut a length of thick thread roughly the height of the cup/jar. Tape it to a pencil. Attach a paperclip to the other end of the thread. Place the pencil on top of the cup/jar so that the weighted thread hangs ~1 inch above the bottom.
- 2. Tape onto one container a piece of paper reading "Solution 1". Do the same for the next one reading "Solution 2" and the next one "Solution 3".
- Record the type of sugar in Table 2. Spread ~ 1-3 tablespoons sugar on a clean, flat surface. Moisten each skewer/thread, and roll each in the sugar a couple times. Set the skewer/thread aside to dry during the next steps.
- 4. Add the water for each solution to their respective glass containers (Table 2).
 - [Optional] Add 2-3 drops of food coloring. Don't add any other flavorings though.
- 5. Let the water stand for \sim 5 minutes to let it equilibrate to room temperature.
- 6. Add the appropriate amount of sugar into each container.
 - *Note:* You may increase the amount of sugar proportionally if you want (aka if you double the sugar, double the water). You can also double the sugar without changing the water to get larger candy, *but record whatever alterations you make*.
 - *Note:* You may try sugar substitutes such as Splenda, but *record the substitution*.
- 7. Use a spoon to stir Solutions 1 and 2 for \sim 1-2 minutes. Then stop stirring, even if not dissolved.
- 8. Place the containers in a quiet area where no one will disturb it and, ideally, where there aren't many room vibrations from running feet, slamming doors, etc. Avoid areas exposed

to airborne dust, pollen, etc. Place a note next to the cups/jars reading "CHEMISTRY EXPERIMENT – DON'T DISTURB".

- 9. Remove the taped sign from Solution 3. Place the glass in a microwave. Heat the solution for a couple minutes, then stir it vigorously. Repeat until you've dissolved the sugar.
 - Tape can melt if put in a microwave, and metal will arc. Make sure everything is microwave-safe. Use oven mitts to handle the hot container.
- 10. Replace the taped "Solution 3" sign. Place the Solution 3 container next to the other glasses.
- 11. When all three containers are room temperature, add the skewer/thread apparatuses to each container.
- 12. Cover the tops of the glasses with a paper towel or something to keep dust from settling.
- 13. Record brief observations of the solutions' appearances in Table 3. Let the solutions sit for several days. Record observations each day (Day 1 = day of experiment).

Solution #	Amount of Sugar (cups)	Amount of Water (cups)	Water Temp.
1	0.5	0.5	Room temp.
2	1.0	0.5	Room temp.
3	1.5	0.5	Hot

Table 2: Sugar Dissolution Reference Ratios

Type of sugar (granulated, raw, confectioners, etc.):

Brand:			er info on nu	itritional label?
Did you alter the recipe?	Yes	/	No	(If Yes, describe in space below)
				(1 point for logistics)

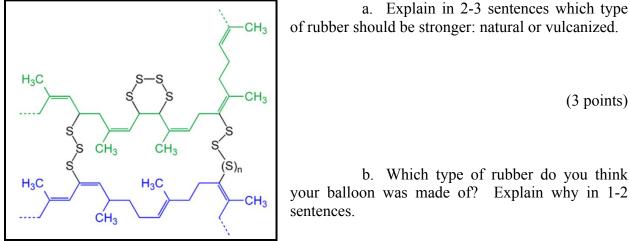
#	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6
1						
2						
3						
5						

 Table 3: Observations About Sugar Solutions (6 points total)

Questions

1. *cis*-Polyisopropene rubber is a polymer naturally made from latex, a type of sap or emulsion produced from certain plants. Latex rubber can be additionally processed via a technique called vulcanization which "cross-links" polymers together with sulfur-carbon bonds (Figure 1).

Figure 1: Vulcanized cis-polyisopropene



(2 points)

2. Limonene (on the right) is a major component of orange peel oil and contributes strongly to the distinctive citrus scent. When you rub an orange peel on a balloon, you rub significant amounts of limonene on the balloon. Rationalize in 2-3 sentences how limonene could cause a balloon to pop, given that limonene is *not* engaging in a chemical reaction.

(4 points)

3. Calculate how many milliliters of water you used for each solution in Part II, assuming that 1 cup of water = 237 mL.

(2 points)

4. Assuming that your sugar was pure sucrose, calculate approximately how many grams of sucrose you used for each solution. Show your calculations, and use appropriate significant figures.

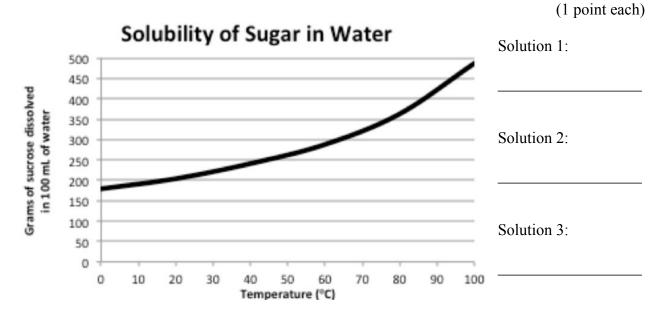
(1 point each)

Solution 1: _____ g

Solution 2: _____ g

Solution 3: _____ g

5. Reference the following solubility curve to determine approximately the type of saturation for each solution at room temperature (~ 20-23 °C).



6. Explain in 1-2 sentences why it was necessary to heat Solution 3.

(2 points)

7. What is the purpose of rolling the skewer/thread in sugar?

(2 points)

8. It is possible to see rock candy formation over time in solutions which aren't supersaturated to begin with. Hypothesize why in 1-2 sentences.

(3 points)