

**Magnet Organic Chemistry
Laboratory Manual**

Name: _____

INFORMATION ACKNOWLEDGEMENT

I have received, read and understand the class expectations and lab safety contract for Mrs. Zado's chemistry class. (Signatures from both parents/guardians requested, if possible. Thank you.)

Student Name (last, first middle) Printed

Block

Grade

Student Signature

Home Phone Number

Parent/Guardian Signature

Daytime Phone Number

Parent/Guardian Signature

Daytime Phone Number

Daytime E-mail address (1)

E-mail address (2)

STUDENT INFORMATION

Nicknames: _____

DOB: _____

Are you in a regularly scheduled after school activity? (sports, drama, band, job, etc.)

Do you have any physical problems that I should be aware of? If yes, please explain.

What was your last math class? _____ Grade earned: _____

How did you do in biology? _____ Grade earned: _____

Do you have access to a computer, printer and internet at home? _____

E-Mail address: _____

On the back please tell me something about yourself. Please write in proper English. Include something about yourself, your family, home life, your goals and ambitions. I am interested in learning about your hobbies, interests and anything else which would help me to know you better.

Thank you!
Mrs. Zado



Flinn Scientific's Student Safety Contract

PURPOSE

Science is a hands-on laboratory class. You will be doing many laboratory activities which require the use of hazardous chemicals. Safety in the science classroom is the #1 priority for students, teachers, and parents. To ensure a safe science classroom, a list of rules has been developed and provided to you in this student safety contract. These rules must be followed at all times. Two copies of the contract are provided. One copy must be signed by both you and a parent or guardian before you can participate in the laboratory. The second copy is to be kept in your science notebook as a constant reminder of the safety rules.

GENERAL RULES

1. Conduct yourself in a responsible manner at all times in the laboratory.
2. Follow all written and verbal instructions carefully. If you do not understand a direction or part of a procedure, ask the instructor before proceeding.
3. Never work alone. No student may work in the laboratory without an instructor present.
4. When first entering a science room, do not touch any equipment, chemicals, or other materials in the laboratory area until you are instructed to do so.
5. Do not eat food, drink beverages, or chew gum in the laboratory. Do not use laboratory glassware as containers for food or beverages.
6. Perform only those experiments authorized by the instructor. Never do anything in the laboratory that is not called for in the laboratory procedures or by your instructor. Carefully follow all instructions, both written and oral. Unauthorized experiments are prohibited.
7. Be prepared for your work in the laboratory. Read all procedures thoroughly before entering the laboratory.
8. Never fool around in the laboratory. Horseplay, practical jokes, and pranks are dangerous and prohibited.
9. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times. Bring only your laboratory instructions, worksheets, and/or reports to the work area. Other materials (books, purses, backpacks, etc.) should be stored in the classroom area.
10. Keep aisles clear. Push your chair under the desk when not in use.
11. Know the locations and operating procedures of all safety equipment including the first aid kit, eyewash station, safety shower, fire extinguisher, and fire blanket. Know where the fire alarm and the exits are located.
12. Always work in a well-ventilated area. Use the fume hood when working with volatile substances or poisonous vapors. Never place your head into the fume hood.
13. Be alert and proceed with caution at all times in the laboratory. Notify the instructor immediately of any unsafe conditions you observe.
14. Dispose of all chemical waste properly. Never mix chemicals in sink drains. Sinks are to be used only for water and those solutions designated by the instructor. Solid chemicals, metals, matches, filter paper, and all other insoluble materials are to be disposed of in the proper waste containers, not in the sink. Check the label of all waste containers twice before adding your chemical waste to the container.
15. Labels and equipment instructions must be read carefully before use. Set up and use the prescribed apparatus as directed in the laboratory instructions or by your instructor.
16. Keep hands away from face, eyes, mouth and body while using chemicals or preserved specimens. Wash your hands with soap and water after performing all experiments. Clean all work surfaces and apparatus at the end of the experiment. Return all equipment clean and in working order to the proper storage area.
17. Experiments must be personally monitored at all times. You will be assigned a laboratory station at which to work. Do not wander around the room, distract other students, or interfere with the laboratory experiments of others.
18. Students are never permitted in the science storage rooms or preparation areas unless given specific permission by their instructor.
19. Know what to do if there is a fire drill during a laboratory period; containers must be closed, gas valves turned off, fume hoods turned off, and any electrical equipment turned off.
20. Handle all living organisms used in a laboratory activity in a humane manner. Preserved biological materials are to be treated with respect and disposed of properly.
21. When using knives and other sharp instruments, always carry with tips and points pointing down and away. Always cut away from your body. Never try to catch falling sharp instruments. Grasp sharp instruments only by the handles.
22. If you have a medical condition (e.g., allergies, pregnancy, etc.), check with your physician prior to working in lab.
23. Any time chemicals, heat, or glassware are used, students will wear laboratory goggles. There will be no exceptions to this rule!
24. Contact lenses should not be worn in the laboratory unless you have permission from your instructor.
25. Dress properly during a laboratory activity. Long hair, dangling jewelry, and loose or baggy clothing are a hazard in the laboratory. Long hair must be tied back and dangling jewelry and loose or baggy clothing must be secured. Shoes must completely cover the foot. No sandals allowed.
26. Lab aprons have been provided for your use and should be worn during laboratory activities.

CLOTHING

23. Any time chemicals, heat, or glassware are used, students will wear laboratory goggles. There will be no exceptions to this rule!
24. Contact lenses should not be worn in the laboratory unless you have permission from your instructor.
25. Dress properly during a laboratory activity. Long hair, dangling jewelry, and loose or baggy clothing are a hazard in the laboratory. Long hair must be tied back and dangling jewelry and loose or baggy clothing must be secured. Shoes must completely cover the foot. No sandals allowed.
26. Lab aprons have been provided for your use and should be worn during laboratory activities.

ACCIDENTS AND INJURIES

27. Report any accident (spill, breakage, etc.) or injury (cut, burn, etc.) to the instructor immediately, no matter how trivial it may appear.
28. If you or your lab partner are hurt, immediately yell out "Code one, Code one" to get the instructor's attention.
29. If a chemical splashes in your eye(s) or on your skin, immediately flush with running water from the eyewash station or safety shower for at least 20 minutes. Notify the instructor immediately.
30. When mercury thermometers are broken, mercury must not be touched. Notify the instructor immediately.

HANDLING CHEMICALS

31. All chemicals in the laboratory are to be considered dangerous. Do not touch, taste, or smell any chemicals unless specifically instructed to do so. The proper technique for smelling chemical fumes will be demonstrated to you.
32. Check the label on chemical bottles twice before removing any of the contents. Take only as much chemical as you need.
33. Never return unused chemicals to their original containers.

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Flinn Scientific's Student Safety Contract

34. Never use mouth suction to fill a pipet. Use a rubber bulb or pipet pump.
35. When transferring reagents from one container to another, hold the containers away from your body.
36. Acids must be handled with extreme care. You will be shown the proper method for diluting strong acids. Always add acid to water, swirl or stir the solution and be careful of the heat produced, particularly with sulfuric acid.
37. Handle flammable hazardous liquids over a pan to contain spills. Never dispense flammable liquids anywhere near an open flame or source of heat.
38. Never remove chemicals or other materials from the laboratory area.
39. Take great care when transporting acids and other chemicals from one part of the laboratory to another. Hold them securely and walk carefully.

HANDLING GLASSWARE AND EQUIPMENT

40. Carry glass tubing, especially long pieces, in a vertical position to minimize the likelihood of breakage and injury.
41. Never handle broken glass with your bare hands. Use a brush and dustpan to clean up broken glass. Place broken or waste glassware in the designated glass disposal container.
42. Inserting and removing glass tubing from rubber stoppers can be dangerous. Always lubricate glassware (tubing, thistle tubes, thermometers, etc.) before attempting to insert it in a stopper. Always protect your hands with towels or cotton gloves when inserting glass tubing into, or removing it from, a rubber stopper. If a piece of glassware becomes "frozen" in a stopper, take it to your instructor for removal.
43. Fill wash bottles only with distilled water and use only as intended, e.g., rinsing glassware and equipment, or adding water to a container.
44. When removing an electrical plug from its socket, grasp the plug, not the electrical cord. Hands must be completely dry before touching an electrical switch, plug, or outlet.
45. Examine glassware before each use. Never use chipped or cracked glassware. Never use dirty glassware.
46. Report damaged electrical equipment immediately. Look for things such as frayed cords, exposed wires, and loose

connections. Do not use damaged electrical equipment.

47. If you do not understand how to use a piece of equipment, ask the instructor for help.
48. Do not immerse hot glassware in cold water; it may shatter.

HEATING SUBSTANCES

49. Exercise extreme caution when using a gas burner. Take care that hair, clothing and hands are a safe distance from the flame at all times. Do not put any substance into the flame unless specifically instructed to do so. Never reach over an exposed flame. Light gas (or alcohol) burners only as instructed by the teacher.
50. Never leave a lit burner unattended. Never leave anything that is being heated or is visibly reacting unattended. Always turn the burner or hot plate off when not in use.
51. You will be instructed in the proper method of heating and boiling liquids in test tubes. Do not point the open end of a test tube being heated at yourself or anyone else.
52. Heated metals and glass remain very hot for a long time. They should be set aside to cool and picked up with caution. Use tongs or heat-protective gloves if necessary.
53. Never look into a container that is being heated.
54. Do not place hot apparatus directly on the laboratory desk. Always use an insulating pad. Allow plenty of time for hot apparatus to cool before touching it.
55. When bending glass, allow time for the glass to cool before further handling. Hot and cold glass have the same visual appearance. Determine if an object is hot by bringing the back of your hand close to it prior to grasping it.

QUESTIONS

56. Do you wear contact lenses?
 YES NO
57. Are you color blind?
 YES NO
58. Do you have allergies?
 YES NO
If so, list specific allergies _____

AGREEMENT

I, _____,
(student's name) have read and agree to follow all of the safety rules set forth in this contract. I realize that I must obey these rules to ensure my own safety, and that of my fellow students and instructors. I will cooperate to the fullest extent with my instructor and fellow students to maintain a safe lab environment. I will also closely follow the oral and written instructions provided by the instructor. I am aware that any violation of this safety contract that results in unsafe conduct in the laboratory or misbehavior on my part, may result in being removed from the laboratory, detention, receiving a failing grade, and/or dismissal from the course.

Student Signature

Date

Dear Parent or Guardian:

We feel that you should be informed regarding the school's effort to create and maintain a safe science classroom/laboratory environment.

With the cooperation of the instructors, parents, and students, a safety instruction program can eliminate, prevent, and correct possible hazards.

You should be aware of the safety instructions your son/daughter will receive before engaging in any laboratory work. Please read the list of safety rules above. No student will be permitted to perform laboratory activities unless this contract is signed by both the student and parent/guardian and is on file with the teacher.

Your signature on this contract indicates that you have read this Student Safety Contract, are aware of the measures taken to ensure the safety of your son/daughter in the science laboratory, and will instruct your son/daughter to uphold his/her agreement to follow these rules and procedures in the laboratory.

Parent/Guardian Signature

Date

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Periodic Table of the Elements

IA 1 H 1.008											VIIIA 2 He 4.003							
3 Li 6.941	IIA 4 Be 9.012										VIIA 9 F 19.00							
11 Na 23.00	12 Mg 24.30										VIA 8 O 16.00							
19 K 39.10	20 Ca 40.08	IIIB 21 Sc 44.96						22 Ti 47.88	23 V 50.94	24 Cr 52.00	25 Mn 54.94	VIA 7 N 14.01	VIIA 6 C 12.01					
37 Rb 85.47	38 Sr 87.62	39 Y 88.91	40 Zr 91.22			41 Nb 92.91		42 Mo 95.94	43 Tc (98)	44 Ru 101.1	45 Rh 102.9	46 Pd 106.4	47 Ag 107.9	48 Cd 112.4	49 In 114.8	50 Sn 118.7	VIA 5 As 74.92	VIIA 4 Se 78.96
55 Cs 132.9	56 Ba 137.3	57 Lu 175.0	58 Hf 178.5	59 Ta 180.9	60 W 183.8	61 Re 186.2	62 Os 190.2	63 Pt 195.1	64 Au 197.0	65 Hg 200.6	66 Tl 204.4	67 Pb 207.2	68 Bi 209.0	69 Po (209)	70 At (210)	VIA 3 Te 127.6	VIIA 3 Br 79.90	
87 Fr (223)	88 Ra 226.0	89 Lr (260)	90 Rf (261)	91 Db (262)	92 Sg (263)	93 Bh (262)	94 Hs (265)	95 Mt (266)	96 Ds (281)	97 Rg (280)	98 Cn (285)	99 Uut (284)	100 Ff (289)	101 Uup (288)	102 Lv (293)	VIA 2 Se 32.07	VIIA 2 Cl 35.45	
																VIIA 1 S 32.07	VIIA 1 Ar 39.95	
																	VIIA 0 As 74.92	VIIA 0 Kr 83.80
																	VIIA -1 Ge 72.59	VIIA -1 Br 79.90
																	VIIA -2 Ga 69.72	VIIA -2 Kr 83.80
																	VIIA -3 Ge 72.59	VIIA -3 Kr 83.80
																	VIIA -4 As 74.92	VIIA -4 Kr 83.80
																	VIIA -5 Se 78.96	VIIA -5 Kr 83.80
																	VIIA -6 Te 127.6	VIIA -6 Kr 83.80
																	VIIA -7 Te 127.6	VIIA -7 Kr 83.80
																	VIIA -8 Te 127.6	VIIA -8 Kr 83.80
																	VIIA -9 Te 127.6	VIIA -9 Kr 83.80
																	VIIA -10 Te 127.6	VIIA -10 Kr 83.80

57 La 138.9	58 Ce 140.9	59 Pr 140.9	60 Nd 144.2	61 Pm (145)	62 Sm 150.4	63 Eu 152.0	64 Gd 157.2	65 Tb 158.9	66 Dy 162.5	67 Ho 164.9	68 Er 167.3	69 Tm 168.9	70 Yb 173.0
89 Ac 227.0	90 Th 232.0	91 Pa 231.0	92 U 238.0	93 Np 237.0	94 Pu (244)	95 Am (243)	96 Cm (247)	97 Bk (247)	98 Cf (251)	99 Es (252)	100 Fm (257)	101 Md (258)	102 No (259)

Electronegativities of the Elements

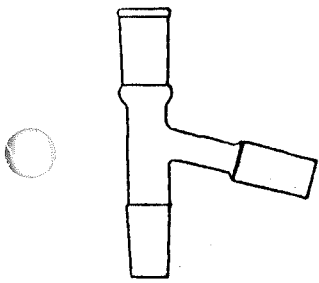
H 2.1																					
Li 1.0	Be 1.5															B 2.0	C 2.5	N 3.0	O 3.5	F 4.0	Ne -
Na 0.9	Mg 1.2															Al 1.5	Si 1.8	P 2.1	S 2.5	Cl 3.0	Ar -
K 0.8	Ca 1.0	Sc 1.3	Ti 1.5	V 1.6	Cr 1.6	Mn 1.5	Fe 1.8	Co 1.8	Ni 1.8	Cu 1.9	Zn 1.6	Ga 1.6	Ge 1.8	As 2.0	Se 2.4	Br 2.8	Kr -				
Rb 0.8	Sr 1.0	Y 1.2	Zr 1.4	Nb 1.6	Mo 1.8	Tc 1.9	Ru 2.2	Rh 2.2	Pd 2.2	Ag 1.9	Cd 1.7	In 1.7	Sn 1.8	Sb 1.9	Te 2.1	I 2.5	Xe -				
Cs 0.7	Ba 0.9	La 1.1	Hf 1.3	Ta 1.5	W 1.7	Re 1.9	Os 2.2	Ir 2.2	Pt 2.2	Au 2.4	Hg 1.9	Tl 1.8	Pb 1.8	Bi 1.9	Po 2.0	At 2.2	Rn -				
Fr 0.7	Ra 0.9	Ac 1.1																			

Activity Series of the Elements

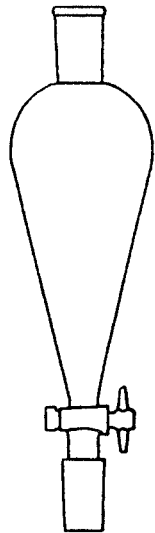
<u>Metals</u>	Decreasing Reactivity ↘ Decreasing Reactivity ↘ Decreasing Reactivity	<u>Nonmetals</u>
Lithium		Fluorine
Potassium		Chlorine
Barium		Bromine
Strontium		Iodine
Calcium		
Sodium		
Magnesium		
Aluminum		
Manganese		
Zinc		
Chromium		
Iron		
Cadmium		
Cobalt		
Nickel		
Tin		
Lead		
Hydrogen		
Antimony		
Arsenic		
Bismuth		
Copper		
Mercury		
Silver		
Palladium		
Platinum		
Gold		

Solubility Rules

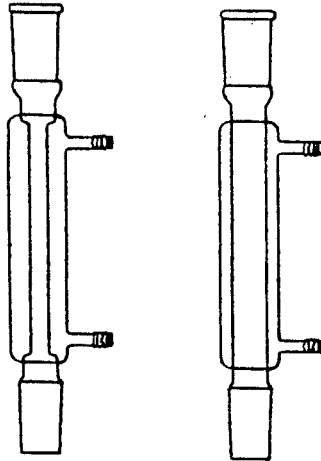
Ammonium (NH_4^+)	Salts of group IA elements are always soluble
Chlorides (Cl^-)	All soluble except AgCl , Hg_2Cl_2 , and PbCl_2 , which are insoluble
Bromides (Br^-)	All soluble except AgBr , Hg_2Br_2 , HgBr_2 , and PbBr_2 , which are insoluble
Iodides (I^-)	All soluble except AgI , Hg_2I_2 , HgI_2 , and PbI_2 , which are insoluble
Fluorides (F^-)	Are generally insoluble
Chlorates (ClO_3^-), Nitrates (NO_3^-), and Acetates ($\text{C}_2\text{H}_3\text{O}_2^-$)	All soluble
Sulfates (SO_4^{2-})	Are soluble except CaSO_4 , SrSO_4 , BaSO_4 , Hg_2SO_4 , HgSO_4 , PbSO_4 , and Ag_2SO_4 , which are insoluble
Phosphates (PO_4^{3-}), Carbonates (CO_3^{2-}) and Oxides (O^{2-})	Are insoluble except when with ammonium or Group IA elements
Hydroxides (OH^-)	Are insoluble except when with ammonium, Group IA elements and Group 2A elements from calcium down
Sulfides (S^{2-})	Are insoluble except when with ammonium, Group IA and 2A elements
Chromates (CrO_4^{2-})	Are generally insoluble



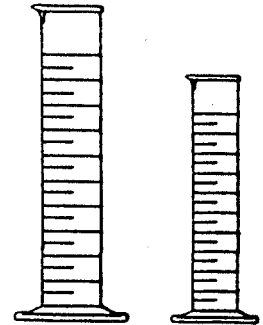
Distillation Adapter



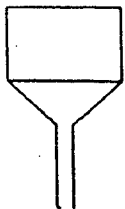
Separatory Funnel



Condensers



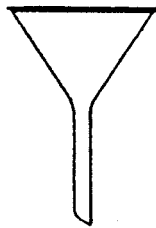
25 and 50 mL Graduated Cylinders



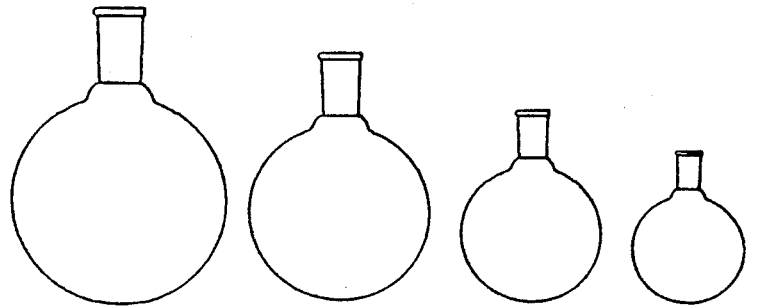
Buchner Funnel



Pennyhead Stopper



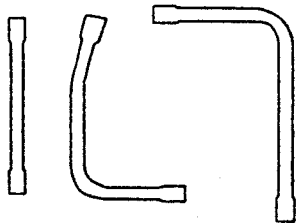
Glass Funnel



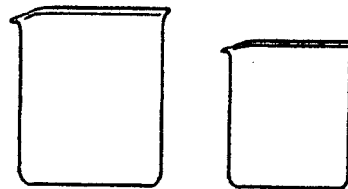
25mL, 50 mL, 100 mL, 250 mL Round Bottom Flasks



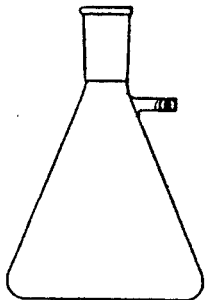
Thermometer



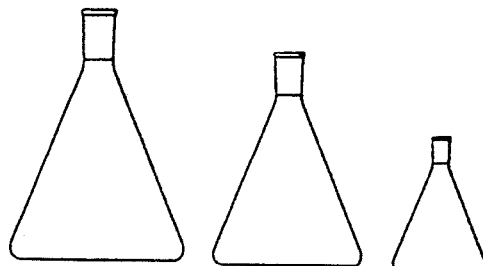
3 rubber hoses



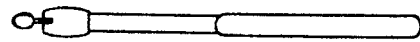
50 mL and 100 mL beaker



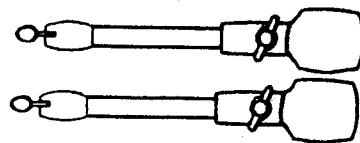
Filtering Flask



125 mL, 50 mL, and 25 mL Erlenmeyer Flasks



Iron Ring



Clamps

Other equipment: watch glass, scoopula, temperature controller, heating mantle, thermometer adapter



Organic Functional Groups

Class of Compound	Functional Group	General Formula	Example
halide (halocarbon)	-F (fluoro-) -Cl (chloro-) -Br (bromo-) -I (iodo-)	R-X (X represents any halogen)	CH ₃ CHClCH ₃ 2-chloropropane
alcohol	-OH	R-OH	CH ₃ CH ₂ CH ₂ OH 1-propanol
ether	-O-	R-O-R'	CH ₃ OCH ₂ CH ₃ methyl ethyl ether
aldehyde	$\begin{array}{c} \text{O} \\ \parallel \\ \text{-C-H} \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R-C-H} \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{CH}_3\text{CH}_2\text{C-H} \end{array}$ propanal
ketone	$\begin{array}{c} \text{O} \\ \parallel \\ \text{-C-} \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R-C-R}' \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{CH}_3\text{CCH}_2\text{CH}_2\text{CH}_3 \end{array}$ 2-pentanone
organic acid	$\begin{array}{c} \text{O} \\ \parallel \\ \text{-C-OH} \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R-C-OH} \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{CH}_3\text{CH}_2\text{C-OH} \end{array}$ propanoic acid
ester	$\begin{array}{c} \text{O} \\ \parallel \\ \text{-C-O-} \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R-C-O-R}' \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{CH}_3\text{CH}_2\text{COCH}_3 \end{array}$ methyl propanoate
amine	-N-	$\begin{array}{c} \text{R}' \\ \\ \text{R-N-R}'' \end{array}$	CH ₃ CH ₂ CH ₂ NH ₂ 1-propanamine
amide	$\begin{array}{c} \text{O} \\ \parallel \\ \text{-C-NH} \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R-C-NH} \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{CH}_3\text{CH}_2\text{C-NH}_2 \end{array}$ propanamide

R represents a bonded atom or group of atoms.

Organic Prefixes

Prefix	Number of Carbon Atoms
meth-	1
eth-	2
prop-	3
but-	4
pent-	5
hex-	6
hept-	7
oct-	8
non-	9
dec-	10

Homologous Series of Hydrocarbons

Name	General Formula	Examples	
		Name	Structural Formula
alkanes	C _n H _{2n+2}	ethane	$\begin{array}{c} \text{H} & \text{H} \\ & \\ \text{H}-\text{C}-\text{C}-\text{H} \\ & \\ \text{H} & \text{H} \end{array}$
alkenes	C _n H _{2n}	ethene	$\begin{array}{c} \text{H} & & \text{H} \\ & \diagdown & / \\ & \text{C}=\text{C} \\ & / & \diagdown \\ \text{H} & & \text{H} \end{array}$
alkynes	C _n H _{2n-2}	ethyne	$\text{H}-\text{C}\equiv\text{C}-\text{H}$

n = number of carbon atoms

4.2 How Do We Name Aromatic Compounds?

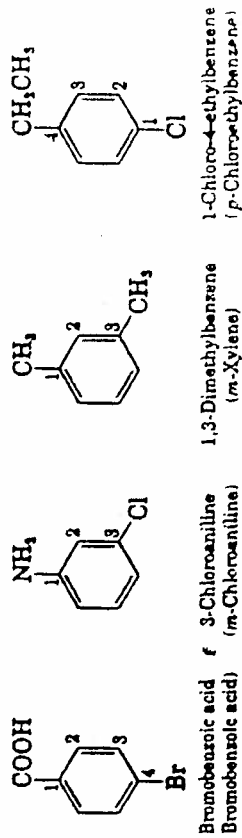
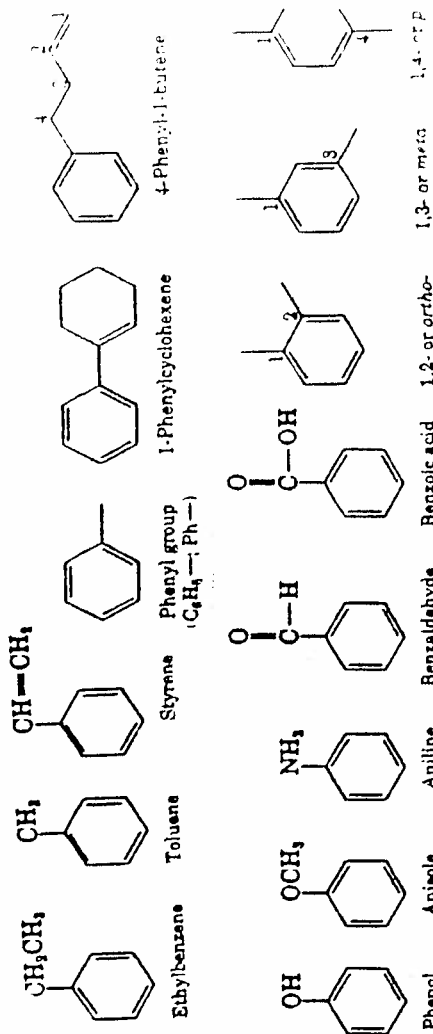


Table 2.1 Most Common Alkyl Groups

Name	Condensed Structural Formula	Name	Condensed Structural Formula
methyl	$-\text{CH}_3$	butyl	$-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$
ethyl	$-\text{CH}_2\text{CH}_3$	isobutyl	$-\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_3$
propyl	$-\text{CH}_2\text{CH}_2\text{CH}_3$	sec-butyl	$-\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_3$
isopropyl	$-\text{CH}(\text{CH}_3)_2$	tert-butyl	$-\text{C}(\text{CH}_3)_3$

Table 2.2 Most Common Alkyl Groups

Name	Condensed Structural Formula	Name	Condensed Structural Formula
methyl	$-\text{CH}_3$	butyl	$-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$
ethyl	$-\text{CH}_2\text{CH}_3$	isobutyl	$-\text{CH}_2\text{CH}(\text{CH}_3)_2$
propyl	$-\text{CH}_2\text{CH}_2\text{CH}_3$	sec-butyl	$-\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_3$
isopropyl	$-\text{CH}(\text{CH}_3)_2$	tert-butyl	$-\text{C}(\text{CH}_3)_3$

Table 2.3 Physical Properties of Some Unbranched Alkanes

Name	Condensed Structural Formula	Molecular Weight (amu)	Melting Point (°C)	Boiling Point (°C)	Density of Liquid (g/ml at 0°C)
methane	CH_4	16.0	-182	-164	(a gas)
ethane	CH_3CH_3	30.1	-183	-88	(a gas)
propane	$\text{CH}_3\text{CH}_2\text{CH}_3$	44.1	-190	-42	(a gas)
butane	$\text{CH}_3(\text{CH}_2)_2\text{CH}_3$	58.1	-138	0	(a gas)
pentane	$\text{CH}_3(\text{CH}_2)_3\text{CH}_3$	72.2	-130	36	0.626
hexane	$\text{CH}_3(\text{CH}_2)_4\text{CH}_3$	86.2	-95	69	0.659

Table 2.4 Water of Four Sets

Structural Formula	Name	Molecular Weight (amu)	Boiling Point (°C)	Solubility in Water
CH_3OH	methanol	32	65	infinite
$\text{CH}_3\text{CH}_2\text{OH}$	ethanol	30	-89	insoluble
$\text{CH}_3\text{CH}_2\text{CH}_2\text{OH}$	1-propanol	60	97	infinite
$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$	1-butanol	74	117	8 g/100 g
$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$	1-pentanol	72	96	insoluble

Name: _____ Block: ___ Date: _____

Organic Chemistry

Lab/topic	Did you feel comfortable with the test performed? Why/Why not? (if no, then come in for remediation	Remediation date	Student comments
The Tie that Bonds			
What's in a Name			
#1 Organic Chemistry Lab			
#2 Conformations of Alkanes Lab			
Functional Groups Activity			
#3 Distillation of Hexane			
#7 Cis-Trans Isomerism			
#8 Plastics Identification			
#9 Chemical Reactions of Alkanes and Alkenes			
Stereochemistry Activity			
#11 Electrophillic Aromatic Substitution: Nitration of Methyl Benzoate			

The Tie That Bonds

Purpose:

1. To review the process of bonding between atoms.
2. To be able to determine if a bond is ionic, polar covalent or non-polar covalent.
3. To be able to describe the difference between polar and non-polar covalent bonds.
4. To be able to determine if a sigma or pi bond has formed and the characteristics of each.

Review:

Indicate the number of valence electrons, bonding capacity and resulting bond angle (if applicable) for the following elements. Illustrate what the lewis dot diagram and ball and stick model would look like for that element.

<i>Element</i>	<i>Valence electrons</i>	<i>Bonding Capacity</i>	<i>Bond Angle</i>	<i>Lewis Dot Diagram</i>	<i>Ball and Stick Model</i>
Carbon					
Hydrogen					
Oxygen					
Nitrogen					
Phosphorous					
Sulfur					
Halogens					

1. What creates an ionic bond? A polar covalent bond? A non-polar covalent bond?
2. How is the type of bond determined between atoms? (describe thoroughly, be sure to ask for help if you don't remember)

3. Determine the type of bond that occurs between the following elements, using the reference chart given.

Type of bond	Electronegativities difference
Ionic	≥ 1.7
Polar covalent	0.5 to 1.6
Non-polar covalent	≤ 0.4

Bonded Elements	Numerical Difference	Type of Bond
Carbon and Hydrogen		
Carbon and Carbon		
Carbon and Oxygen		
Oxygen and Hydrogen		

Practice #1:

- Using the ball and stick models create and illustrate the following molecules.
- Determine the type of bonds that exist through the molecule.
- For polar covalent bonds use a highlighter illustrate the movement of electrons in the bond with an arrow pointing towards the element with the higher electronegative element. After all arrows have been applied determine if the overall molecule has polarity or not.

Illustration of Ball and Stick Model	Type of Bond(s)	Is Molecule Polar (state polar or non-polar)
CH ₄	C-H	
C ₂ H ₆	C-H	
	C-C	
C ₂ H ₄	C-H	
	C-C	
C ₂ H ₂	C-H	
	C-C	
CH ₃ OH	C-C	
	C-H	
	C-O	
	O-H	

CO ₂	C-O	
H ₂ O	O-H	
C ₃ H ₆ O	C-C	
	C-H	
	C-O	

Practice #2:

Materials: 7-Pipe-cleaners of any color except blue, 6-blue pipe-cleaners, 14 beads, 8 - cardboard disc.

1. Create 6 hydrogen atoms by placing hydrogen's single electron (bead) on a colored pipe-cleaner, make a tear drop shape with a pipe-cleaner, twist the ends of the pipe-cleaner together and put through the center hole of the disc, and fold twisted ends into a "knot" so that the pipe-cleaner cannot fall out.
2. Create 2 carbon atoms by placing one electron (bead) on a colored pipe-cleaner and one electron on each of three blue pipe-cleaners. Twist the ends of each pipe-cleaner to form a tear drop shape. Place the colored pipe-cleaner in the center hole and the blue pipe-cleaners in the holes around the outside. Be sure to fold twisted ends into a "knot" so that the pipe-cleaner cannot fall out.

What hybridization refers to the shape of the carbon atom? _____ What is the shape? _____

3. The single color pipe-cleaner will be used to represent the *s* orbital and the blue pipe-cleaners will represent the *p* orbitals. The first electrons to come together between the carbon are the *s* orbitals, they will fold over each other "blending" the pathways so that the electrons will now share an orbital, thus creating covalent bond. This bond can also be referred to as a sigma bond (σ). Please fold the *s* orbitals from the two carbon atoms together, clip them together with a hair clip. Notice that the *p* orbitals on each end of the bond still have free rotation.
4. The hydrogen *s* orbitals will now attach with each of the available carbon *p* orbitals. Notice the *s*-*p* connection still has free rotation and is therefore a sigma bond.

What type of bond is formed between the two carbon atoms? (single, double, triple) _____

5. Remove one hydrogen atom from each carbon atom. Without these hydrogens the carbon octet is not satisfied so the p orbitals will now come together sandwiching the existing sigma bond forming a pi bond (π). One lobe of the p orbital will go behind the sigma bond and the other will go in front. The diagram below will show this turned 90° so that the combination can be seen in more detail. The nuclei will be brought slightly closer together as a result. Notice that free rotation has now been eliminated.

What type of bond is formed between the two carbon atoms? (single, double, triple) _____

6. Remove another hydrogen atom from each carbon atom. The carbon atoms are in need of additional electrons so the p orbitals that are now available will fold over the existing sigma and pi bonds so that the sigma bond is now completely covered by p orbitals on all 4 sides. Please notice that the nuclei of the two atoms are closer together and the bond restricts free rotation. The nuclei will be brought slightly closer together as a result.

What type of bond is formed between the two carbon atoms? (single, double, triple) _____

7. Thinking back to your illustrations of water and carbon dioxide describe the bonds as sigma or pi in both of these molecules.

H₂O _____

CO₂ _____

8. Which of these two molecules would you consider more potentially active? Why? (please use facts to support your choice)

Name: _____ Block: ___ Date: _____

**What's In A Name -
A Review in Organic Structures and Nomenclature**

Purpose:

Review bonding capacity, molecular and empirical formulas, molecular shapes and structures. The lab activity will also introduce and review the nomenclature for organic compounds.

Introduction:

In this activity you will review the information you learned in your previous chemistry classes by creating organic structures out of candy and toothpicks. Each group will receive a bag of candy and toothpicks, a white board and expo marker. Using your knowledge of bonding, bonding capacity and structural formulas complete your pre-lab activity and illustrate the bonding capacity using the supplies provided.

Pre-Lab: Fill-in the answers in the chart provided.

- 1. What is the bonding capacity of the following elements?**
- 2. Designate a color for each element (recommended that hydrogen is the most abundant color and carbon is the second most abundant).**
- 3. Illustrate the arrangement of the bonding capacity by creating a model and then drawing it in the chart.**

Element	Bonding Capacity	Color	Illustration
Carbon			
Hydrogen			
Oxygen			
Nitrogen			
Phosphorous			
Sulfur			
Halogens			

4. What is the shape of a molecule made with carbon fully bonded at the center?
5. If the electrons are shared very close to equally in the bond between carbon and hydrogen, what type of bond is formed?

In organic nomenclature the name of the compound is actually a set of directions that tell the reader exactly what to build. The name is a series of prefixes and suffixes that explain everything from how many carbon have chained together, how they are bonded, what additional attachments may be there and where they are attached.

In the simplest hydrocarbons the prefix tells the number of carbon present and the suffix tells the type of bonds that exist between the carbon.

Prefixes for number of carbon:

Meth - 1	Hexa - 6	Undec - 11
Eth - 2	Hepta - 7	Dodec - 12
Prop - 3	Octa - 8	Tridec - 13
But - 4	Nona - 9	Tetradec - 14
Penta - 5	Deca - 10	Pentadec - 15

Prefixes for attachments: (substituents)

Bromo - bromine
 Chloro- chlorine
 Fluoro - fluorine
 Iodo - iodine
 yl - carbon branch (used in conjunction with
 Numeric prefix)

*a numeric indicator is used to indicate the carbon that has substituent attached

Prefix for shape: cyclo - indicates that the parent carbon chain forms a ring

Suffixes for bonding:

-ane for single bonds (comes from family alkane)
 -ene for double bonds (comes from family alkene)
 -yne for triple bonds (comes from family alkynes)

*a numeric indicator is used to give the position of the double or triple bond.

Using the information presented above and your knowledge of chemistry build the following compounds from your kit. When you complete the structure illustrate it below the name.

1-propyne	4-ethyl-2-methylheptane	1-bromo-cyclobutane

Please construct a compound of your choosing using 12 carbon or less. Please be sure that all bonding capacities are complete. Please leave your chart with color choices on the desk beside your molecule. Record an illustration of your molecule on your lab sheet in the appropriate box.

Name your molecule (properly).

Each group will rotate to the other teams record an illustration of their model and name the molecule.

1 _____ 	2 _____ 	3 _____
4 _____ 	5 _____ 	6 _____
7 _____ 	8 _____ 	9 _____
10 _____ 	11 _____ 	12 _____



Name: _____ Block: ____ Date: _____

Functional Groups Activity

Objective: In this activity you will investigate the organic functional groups, what they are called, their structure, their polarity, and how they change the function of the parent molecule.

Functional Group	Lewis Dot Diagram	Ball and Model	Skeletal model with bond angle	Polarity of functional group	Order of increasing polarity
Alcohol					
Ketones					
Aldehydes					
Carboxylic acids					
Carboxylic esters					
Ethers					
Amines					
Thiols					

Procedure:

We will be using the letter R to represent the remaining portion of a parent chain when illustrating a functional group. When building the model please use a purple ball to represent the R (parent chain).

1. Diagram the Lewis dot structure of each functional group, be sure to use different colored dots for each element's dots so that it is apparent when a bond is formed by the blending of electrons from two different atoms. (recommended coloring: carbon – black, hydrogen – yellow/orange, oxygen – red, nitrogen – blue, sulfur – green)
2. Create ball and stick model for each functional group.
3. Illustrate with colored dots to represent the atoms.
4. Illustrate the functional group with the skeletal model. Please label the resulting bond angles.
5. Examine the bonds, determine the type of bonds that exist and if there will be a region of polarity in the functional group.
6. Rank the functional groups in order of increasing polarity. (1 = low/no polarity, 8 = greatest polarity)

Discussion: (answer on a separate sheet of paper)

1. Which functional groups contain a carbonyl group?
2. Why are aldehydes and ketones separated into different functional groups? (please give a detailed answer.)
3. Why do you think it would be important to investigate the polarity of a functional group?
4. Which of the functional groups would you predict that would most easily react as an anion in an acid/base neutralization reaction? Illustrate the anion form of the functional group.
5. Alcohols and amines are classified as 1° (primary), 2° (secondary) and 3° (tertiary) based on the number of carbon attached to the carbon linked to the functional group. a) illustrate a secondary alcohol b) illustrate a tertiary amine
6. Alcohols and carboxylic acids go through a dehydration synthesis reaction to form an ester. Illustrate what you think this reaction would look like, using R for the parent chains, indicating where the water molecule comes from.
7. For each of the functional groups list a compound that contains that functional group and its common use.

Experiment 1: Introduction to Organic Substances

Purpose: 1) To examine the underlying chemical structure of foods. 2) To burn stuff.

Materials: crucible, clay triangle, two samples of one type of food. Place one sample in the crucible; keep the other at your lab bench.

Procedure:

1. Set up ring stand with iron ring clamp and clay triangle. Position Bunsen burner under iron ring.
2. Light the Bunsen burner. Begin heating the substance. When the reaction stops (no more smoke) turn off Bunsen burner. Leave sample to cool.
3. Place the second sample at the base of your ring stand (so other students can see what the original sample looked like before it was burned).
4. Circulate to the other lab stations to observe the effect of heating on other foods.
5. Clean up: Place foods in the trash. Clean crucible with steel wool if necessary. Return equipment.

FOOD ITEM	BEFORE AND AFTER OBSERVATIONS

Discussion:

1. What color did most food substances change? _____

2. Look at the elements at the front of the room. Which element most resembles the product from burning? _____. This element was present in the foods before they were burned. In many cases, burning/heating breaks down complex molecules (by oxidizing them), leaving only the element that formed the framework of the substance.

3. What substances were exceptions (it did not have the same reaction to burning)?

What elements are found in this compound? _____, _____,

_____. Thus, it did not turn the same color as other foods because it did not contain

_____.

4. List three items in this room that are carbon based and three items that are not (do not include foods from the lab).

5. How many bonds does a carbon atom form when in a molecule? _____

6. How many carbon atoms can be linked together in a chain? _____

7. How are the chains that carbon atoms form different from the chains that other elements

form? _____

8. What other unique property does carbon have (compared to other members of the carbon family)? _____

9. Define organic chemistry:

10. List all of the carbon compounds that are not considered organic

11. Which other elements are commonly found in organic compounds?

12. What part(s) of a human body are principally inorganic?

Experiment 2: Conformations of Alkanes

Procedure and Discussion

1. Construct a model of the ethane molecule and rotate one of the carbons until the model is in the staggered conformation. Draw the Newman projection, sawhorse formula and dash-wedge formula for the molecule.
2. Now rotate one carbon 60° to construct the eclipsed conformation of ethane. Draw the Newman projection, sawhorse formula and dash-wedge formula for the molecule.
3. In which conformation do the electrons in the C-H bonds on one carbon come closer to similar electrons on the other carbon?
4. Considering that like charges repel one another, which conformation of ethane is less stable?
5. What type of motion is required to convert from the less stable arrangement to the more stable arrangement?
6. Define the term conformation.
7. Replace one hydrogen on your model with a carbon and complete the structure to make a model of propane. Sight down the carbon-carbon bonds and rotate the end carbons until they are staggered with respect to the central carbon. This is referred to as fully staggered. Draw the Newman projection by sighting down one of the carbon-carbon bonds and representing the other methyl group as $-\text{CH}_3$.
8. Sight down the carbon-carbon bonds and rotate the end carbons until they are eclipsed with respect to the central carbon. This is referred to as fully eclipsed. Draw the Newman projection by sighting down one of the carbon-carbon bonds and representing the other methyl group as $-\text{CH}_3$.
9. Sight down one carbon-carbon bonds and rotate the end carbon until it is staggered with respect to the central carbon. Leave the other terminal carbon in the eclipsed conformation. This is referred to as half-staggered/half-eclipsed. Draw the Newman projection by sighting down one of the carbon-carbon bonds and representing the other methyl group as $-\text{CH}_3$.
10. Do you expect the energy difference between staggered and eclipsed conformations in propane to be larger than, smaller than, or equal to the energy difference in ethane? Explain.
11. Construct a model of butane and rotate the carbons until the model is fully staggered. Draw the dash-wedge formula for this conformation.
12. Sight down the C-2/C-3 bond and draw a Newman projection around this bond, representing the methyl groups by $-\text{CH}_3$.
13. Keeping the arrangement at C-3 constant, rotate C-2 clockwise 60° , 120° , and 180° , drawing the Newman projection after each rotation.
14. Label the Newman projection in step 12 "A" and each successive rotation "B", "C", and "D". Which formulas represent staggered conformations at the bond between C-2 and C-3? Which are eclipsed?
15. Arrange conformations A, B, C, and D in order of decreasing stability.
16. Construct the chair conformation of cyclohexane. Site down any carbon-carbon bond. Are the hydrogens staggered or eclipsed?
17. How many equatorial hydrogens are present? How many axial hydrogens lie above the plane of the molecule and how many lie below it?
18. Designate one of the carbons as C-1. Which other carbons have an axial hydrogen on the same side of the molecule as C-1?
19. Draw the chair conformation of cyclohexane indicating the equatorial and axial hydrogens.

Name: _____ Block: ____ Date: _____

Organic Chemistry Experiment 2: Conformation of Alkanes

Record your answers to the questions in the spaces provided below. Record your illustrations in the boxes provided on the back of this sheet.

3. _____ 4. _____ 5. _____

6. _____

10. _____

14. staggered: _____ eclipsed: _____ 15. _____

16. _____ 17. _____

18. _____ 20. _____

21. _____ 22. _____

23. _____ 24. _____

25. _____ 26. _____

27. _____

1. Newman Projection ethane staggered	Saw-Horse Formula ethane	Dash-Wedge Formula ethane
2. Newman projection ethane eclipsed	Saw-Horse Formula ethane	Dash-Wedge Formula ethane
7. Newman Projection propane fully staggered	8. Newman Projection propane fully eclipsed	9. Newman Projection propane half n half
11. Dash-Wedge Butane fully staggered	12. Newman Projection Butane C-2/C-3	
13. Newman Projection Butane C-2/C-3 60° rotation	Newman Projection Butane C-2/C-3 120° rotation	Newman Projection Butane C-2/C-3 180° rotation
19. chair conformation of cyclohexane	20. boat conformation of cyclohexane	21. chair conformation of chloro-cyclohexane

20. Construct the boat model of cyclohexane. Site down any carbon-carbon bond. Are the hydrogens staggered or eclipsed?

21. Construct the chair conformation of cyclohexane and replace one of the equatorial hydrogens with a chlorine. Draw the structure of this molecule showing the equatorial and axial bonds clearly.

22. Now flip the chair. Is the chlorine equatorial or axial? Which conformation is more stable, the one in this question or the original conformation?

23. Construct the chair conformation of 1,2-dichlorocyclohexane with both chlorines in the equatorial position. Are the chlorines cis- or trans- to each other?

24. Now flip the chair. Are the chlorines equatorial or axial? Are they cis- or trans- to each other?

Which of the two conformations of 1,2-dichlorocyclohexane is more stable?

25. Relocate the chlorines so that one is equatorial and one is axial, maintaining the 1,2 configuration. Are the chlorines cis- or trans- to each other?

26. Now flip the chair. Are the chlorines equatorial or axial? Are they cis- or trans- to each other?

27. Which of the four conformations of 1,2-dichlorocyclohexane that is most stable?

Experiment 3: Distillation of Hexane

Introduction

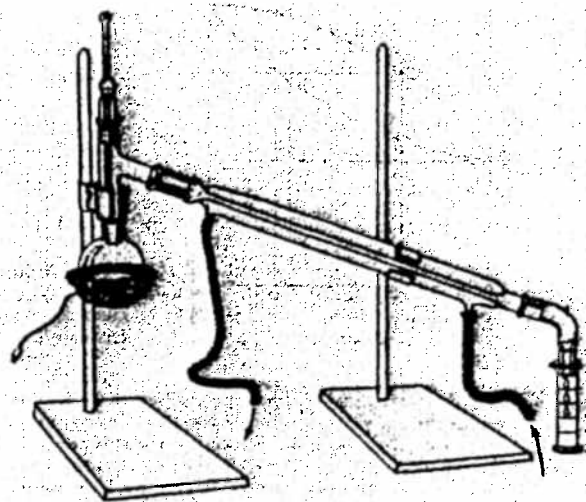
In a sealed container partially filled with a liquid, some molecules escape from the liquid's surface into the space above. In their random motion, molecules that have escaped to the vapor may strike the liquid surface again and stick to it. At equilibrium, the number of molecules that leave the surface equals the number of vaporized molecules that strike the liquid's surface and stick. The molecules in the vapor also strike the walls of the container and exert a pressure, which is called the vapor pressure of a liquid. If the temperature is raised, more molecules escape to the vapor until equilibrium is once again established. The vapor pressure of a liquid, therefore, increases with increasing temperature.

The boiling point of a liquid is that temperature at which the vapor pressure of the liquid becomes equal to the pressure exerted by its surroundings. If the liquid is open to the atmosphere, the boiling point is the temperature at which the vapor pressure becomes equal to the atmospheric pressure. The vapor pressure of a pure liquid rises steadily as the temperature is increased until the boiling point is reached.

A thermometer placed in the vapor of a boiling pure liquid registers that liquid's boiling point. The temperature remains constant throughout the distillation of a pure liquid. This is because, at the boiling point, vapor and liquid are in equilibrium; if the phase composition of the vapor and the liquid remains constant throughout the process, the temperature also remains constant. The boiling point (at a given pressure) is a characteristic property of a pure liquid, just as the melting point is a characteristic property of a pure crystalline solid.

When a mixture of two miscible liquids with different boiling points is heated, the vapor does not have the same composition as the liquid. Instead, the vapor is richer in the more volatile component. The boiling point gradually rises as the liquid becomes richer in the higher boiling component. Thus, in a simple distillation mixture, the first material to distill (called the first cut or first fraction) is rich in the more volatile or lower boiling component, while the last material (last cut, last fraction) is rich in the less volatile or higher boiling material.

In this experiment you will first distill a pure liquid and observe its boiling point. Then you will distill a two-component mixture and compare the boiling points of the two distillations.



Procedure

1. Set up the distillation apparatus as shown in the diagram above. Use a 50 mL round bottom flask. Be sure to place the thermometer bulb just below the side arm so that it measures the temperature of the vapor leaving the flask.
2. Remove the thermometer adapter and place 15 mL of hexane and a boiling stone in the flask. Replace the thermometer adapter.
3. After the teacher has checked your set up, start the water and turn on the heating mantle. Once the hexane begins dripping into the receiver, note the temperature. Take the temperature after 1, 3, 5, 7, 9, and 11 mL of distillate have been collected.
4. Once 11 mL of distillate have been collected, turn off the heating mantle. Note: never distill a liquid to dryness. An explosion may result.
5. Discard all the hexane in the waste beaker in the fume hood.
6. Carry out a second distillation, this time using a mixture of 10 mL of hexane and 10 mL of toluene. Record the temperature when 1, 2, 4, 6, 8, 10, 12, 14, and 16 mL of distillate have been collected. Discard the waste in the fume hood.
7. Look up the boiling points of hexane and toluene and record them in your data table.

Data Table

Literature values for boiling points: hexane _____
toluene _____

Quantity Distilled (mL)	Temperature (°C)
1	
3	
5	
7	
9	
11	

Quantity Distilled (mL)	Temperature (°C)
1	
2	
4	
6	
8	
10	
12	
14	
16	

Data Analysis

1. On a single graph, plot boiling point vs. volume of distillate, labeling each curve.
2. Compare the two curves plotted in the calculation section.
3. A pure liquid has a constant boiling point, but a liquid with a constant boiling point is not necessarily pure. Explain.
4. What effect does a reduction in atmospheric pressure have on the boiling point of a liquid?
5. Why doesn't a pure liquid in a distillation flask vaporize all at once when the boiling point is reached?
6. Why is it dangerous to heat a liquid in a distillation apparatus that is closed tightly at every joint and has no vent to the atmosphere?
7. Why is it important that the cooling water in a distillation apparatus enter the condenser jacket at the lower end and exit at the upper end, and not vice versa?
8. Why should a distilling flask be filled to not more than 2/3 of its capacity at the beginning of a distillation procedure?

Experiment 4: Recrystallization

Introduction

Impure crystalline substances can be purified by recrystallization from a suitable solvent. This process depends on two facts: most compounds are more soluble in hot solvents than in cold solvents, and impurities have solubilities different than those of the desired compound. Recrystallization involves (1) dissolving the impure material in a minimum amount of boiling solvent, (2) filtering the hot solution to remove impurities, (3) allowing the solution to cool slowly to deposit crystals of the compound, (4) filtering the crystals from the solution (called the mother liquor), (5) washing the crystals with a little cold solvent to remove the mother liquor, and (6) drying the crystals to remove the last trace of solvent.

If recrystallization is to be effective, the solvent must be properly selected. A good recrystallization solvent should (1) dissolve a moderate quantity of the substance being purified at an elevated temperature but only a small quantity at low temperatures, (2) not react with the substance being purified, (3) dissolve impurities readily at a low temperature or not dissolve them at all, and (4) be readily removable from the purified product. This last requirement usually means that the solvent should have a fairly low boiling point and evaporate readily. If a single solvent cannot be found to meet all these requirements, a mixture of two solvents may be used. Since many recrystallization solvents are flammable, a Bunsen burner should not be used for heating.

To obtain good recovery of purified material, it is best to avoid using unnecessarily large volume of solvent. Dissolving the substance in the smallest possible amount of hot solvent minimizes the amount of material lost by retention in the mother liquors. In practice, 3 – 5% more solvent than the minimum required is used so the hot solution will be not quite saturated. This helps to prevent separation of the crystals and clogging of the filter paper during the filtration of the hot solution.

Traces of coloring matter or resinous impurities can sometimes be removed with selective adsorbents such as finely divided charcoal. To do this, add a small amount of decolorizing charcoal to warm (not hot) solution before filtering it. Avoid using excess decolorizing agent, however, because it may also adsorb appreciable amounts of the substance being purified.

Some substances readily form supersaturated solutions, and crystallization may not occur spontaneously when the hot solution is cooled. In such situations, it is sometimes possible to initiate crystallization by scratching the walls of the container beneath the surface of the solution with a stirring rod. The best way to induce recrystallization is to “seed” the cold solution with one or two crystals of the substance being purified. Although some compounds crystallize readily, others may separate from solutions as oils, and it may take some time before they crystallize.

Filtration

To remove insoluble impurities and decolorizing charcoal, it is necessary to filter the solution while it is hot. Otherwise, when the solution cools, crystals deposit prematurely. Rapid filtration can be accomplished by using fluted filter paper (see figure 1) or by using a vacuum to increase filtration rate. Vacuum filtration is generally used to remove soluble impurities and solvents from the crystals of the purified substance. See figure 2 for the set-up of vacuum filtration apparatus. A piece of filter paper is placed inside the Buchner funnel and moistened with a small amount of solvent and vacuum is applied. The solution and crystals are transferred to the filter paper. The solution will pass through the paper while the crystals remain on the paper.

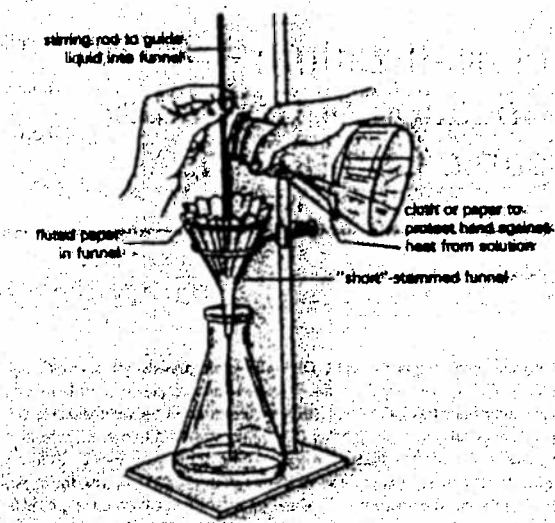


Figure 1

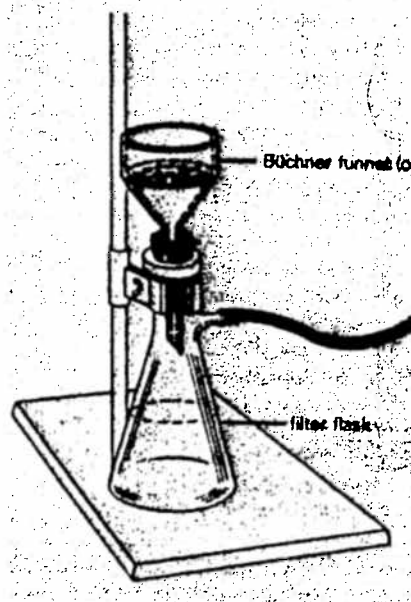


Figure 2

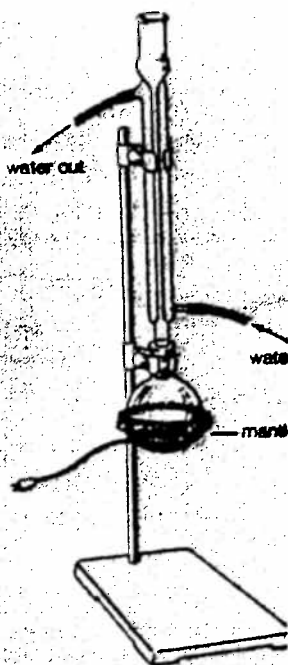


Figure 3

Reflux

It is sometimes necessary to heat a substance in a solvent for a long time without boiling away the solvent. This can be done by attaching a vertical condenser to the flask containing the boiling solution (see figure 3). The solvent vapor, upon cooling, condenses and returns as a liquid to the boiling solution. This process of continuous boiling, vaporization, cooling, and return of condensate is called refluxing. In recrystallization, it is sometimes necessary to use a reflux apparatus to obtain a solution of the material being purified because the dissolution process may be slow. We also use the reflux technique when recrystallizing large samples from volatile, flammable solvents.

In this experiment, a sample of impure acetanilide will be recrystallized from water. You will weigh the crude sample and the pure product and determine the melting point before and after recrystallization.

Procedure

1. Weigh out a 1.5 g sample of impure acetanilide and record the mass.
2. Place the sample in a 100 mL round bottom flask, connect the flask to a reflux condenser, and start the water through the condenser.
3. Add 35 mL of water to the flask through the top of the condenser. Bring the water to a boil using the heating mantle.
4. Continue to reflux the solution until no more solid appears to be dissolving. Remove the heating mantle.
5. While refluxing, fill a 250 mL beaker about half full of distilled water and bring to a boil.
6. While refluxing, determine the melting point of the impure sample and look up the melting point of acetanilide.
7. Allow the flask to cool for about 2 minutes. Remove the reflux condenser, add about 0.2 g of decolorizing charcoal to the flask, and replace the reflux condenser. Reflux the solution for an additional 5 minutes.
8. While solution is refluxing, set up the apparatus for filtration (see figure 1), using fluted filter paper and a 125 mL Erlenmeyer flask.
9. At the end of the 5 minutes of refluxing, pour 15 – 20 mL of boiling water through the funnel to warm it and wet the filter paper. Discard the water. Remove the condenser from the round bottom flask and, using the clamp as a handle; quickly pour the hot solution into the funnel.
10. If particles of charcoal pass through the filter paper return the filtrate to the round bottom flask, heat the solution to boiling, and filter again through the same piece of filter paper.
11. As the solutions cools, crystals will begin to form. As soon as crystals appear, place the Erlenmeyer flask in an ice bath to complete crystallization.
12. While the Erlenmeyer is sitting in the ice bath, set up for vacuum filtration. Place the filter paper in the funnel, turn on the vacuum, and wet the filter paper with 15 – 20 mL of cold water.
13. Pour the solution and crystals into the funnel and wash them with a few milliliters of cold water.
14. Remove the filter paper from the funnel and place on a watch glass. Let dry until next class period.
15. Find the mass and melting point of the dry crystals and record.

Data Table

Mass of impure acetanilide

Mass of filter paper

Mass of filter paper and purified acetanilide

Mass of purified acetanilide

Melting point of impure acetanilide

Melting point of purified acetanilide

Literature value for melting point of acetanilide

Data Analysis

1. Calculate the percent recovery of acetanilide (i.e. what percent of the crude substance was acetanilide?).
2. Give two reasons why vacuum filtration is sometimes preferable to ordinary (gravity) filtration.
3. Why is water a good solvent for the recrystallization of acetanilide?
4. Why must the funnel be heated before the hot acetanilide solution is filtered?
5. Assuming that wood shavings, charcoal and sugar are the main impurities in the sample of crude acetanilide, how were each removed from the impure sample?

Experiment 5: Extraction

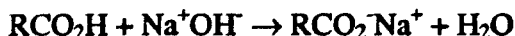
Introduction: In synthesis, the desired product must be separated from the by-products, excess reactants, impurities, and other substances that may be present in the reaction mixture. Similarly, substances in nature are always mixed with other substances. Extraction is the most common technique used to separate a desired organic product from a reaction mixture or to isolate an organic substance from its natural source. Extraction usually involves shaking a solution that contains the desired substance with an immiscible solvent in which the desired substance is more soluble than it is in the starting solution. Upon standing, the solvents form two layers that can be separated. Extraction may have to be repeated several times to effect complete separation.

Most commonly, one of the solvents is organic and the other aqueous. Inorganic compounds can usually be separated from organic compounds in this way since the former dissolve in the aqueous phase and the latter dissolve in the organic solvent. In such cases, a single extraction may suffice to effect satisfactory separation.

When choosing a solvent, many properties must be considered. The extraction solvent must readily dissolve the substance to be extracted, yet it must be only sparingly soluble in the solvent from which the desired substance is to be extracted. Also, it should only extract the desired substance or as small an amount as possible of any other substance present. It should not react chemically with the solute in any undesirable way and it should be easily separated from the desired solute after extraction. This last requirement can be met if the solvent is low-boiling and easily removed by distillation.

Common organic solvents that fulfill these requirements include many hydrocarbons and their chloro derivatives, such as benzene, petroleum ether (a mixture of low-boiling alkanes), dichloromethane, chloroform, and carbon tetrachloride. If benzene or chlorinated hydrocarbons are used, however, it is important to avoid breathing their vapors because these compounds are toxic and some are carcinogenic. They can be used safely if we carry out operations in an efficient fume hood and take care to avoid getting them on the skin. Diethyl ether (usually referred to as ether) is highly flammable and, upon standing in air, its solutions may develop dangerous concentrations of explosive peroxides. Furthermore, it is slightly water-soluble. Nevertheless, because most organic compounds are highly soluble in it and because of its low boiling point, ether is frequently used despite its drawbacks.

Sometimes we can use desirable, easily reversed chemical reactions such as acid-base reactions to effect separation by extraction. For example, dilute sodium hydroxide converts organic acids to their sodium salts:



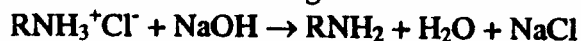
Although a particular acid may not be soluble in water, its more polar sodium salt usually is. When a mixture of a neutral compound and an acidic, water-insoluble compound in an organic solvent is shaken with dilute aqueous sodium hydroxide, the acid is converted to its sodium salt, which dissolves in the aqueous layer, and the neutral compound remains in the organic layer. After the layers are separated, the acid is recovered by acidifying the aqueous layer with a strong acid:



Dilute aqueous acid can be used to extract basic compounds, particularly amines, from neutral or acidic substances by converting them to water-soluble alkylammonium salts:



After separating the organic and aqueous layers, we can recover the amine from the aqueous layer by making the solution alkaline with a strong base:



Extractions are usually performed with a separatory funnel. Improperly handled, this moderately expensive piece of glassware is easily broken. When using one, follow these steps:

1. Support the funnel in an iron ring attached to a ring stand (see figure 1).
2. Close the stopcock and add the liquids to be separated.
3. Insert the stopper, lift it out of the ring, and invert the funnel holding the stopper with one hand and the stopcock with the other (see figure 2).
4. With the barrel pointing away from everybody, slowly open the stopcock. This will relieve any pressure build-up.
5. After the pressure is released, close the stopcock, shake the funnel gently two or three times, and again relieve the pressure. Repeat this two or three times.
6. Replace the funnel in the iron ring and remove the stopper immediately. Allow the funnel to stand until the layers separate.
7. Slowly draw off the lower layer through the stopcock into a flask or beaker of the appropriate size. Close the stopcock when the upper layer enters the stopcock bore.
8. Pour the upper layer out through the top of the funnel.

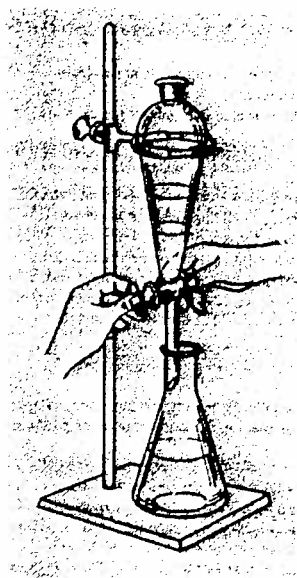


Figure 1

Sometimes, it is difficult to obtain a sharp separation of layers because an emulsion has formed. Gentle swirling of the funnel in an upright position, gentle stirring with a glass stirring rod, and addition of salt to the aqueous layer may overcome this difficulty.

In this experiment, a mixture of benzoic acid and naphthalene (a neutral substance) will be separated into its components by extraction.

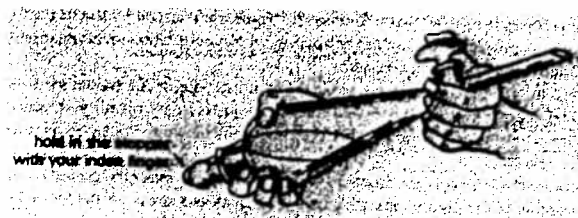


Figure 2

Procedure

1. Obtain 1.5 g sample of the mixture and record the exact mass. Grind in a mortar and pestle.
2. Dissolve the mixture in 14 mL of ether and pour into the separatory funnel. Measure the aliquots of liquid using glass pipettes and pumps.
3. To separate the acid, add 14 mL of 10% sodium hydroxide solution to the funnel and shake according to the directions in the introduction.
4. Draw off the lower, aqueous layer into a 125 mL Erlenmeyer flask and repeat step 3 two more times, combining the aqueous layers into the Erlenmeyer flask.
5. Extract the ether with 5 mL of water and add the water to the aqueous layers in the Erlenmeyer flask.
6. Pour the ether solution into a 25 mL Erlenmeyer flask. Add enough calcium chloride to cover the bottom of the flask, stopper the flask, and swirl the mixture occasionally for 15 minutes.
7. Mass a 50 mL beaker and record its mass.
8. Decant the ether into the 50 mL beaker and place the beaker in the fume hood until the ether has evaporated. Place the calcium chloride in the trash.
9. Find the mass of the remaining solid in the beaker.
10. Neutralize the combined alkaline extracts by adding concentrated HCl a small amount at a time until the solution is acid to litmus paper. (Approximately 4-5 mL until the solution is cloudy and then test for acid.)
11. Recover the precipitated benzoic acid by vacuum filtration. When finished, place the filter paper on a watch glass and allow to dry. Find the mass of benzoic acid recovered and its melting point.
12. Look up the melting point of benzoic acid and record on your data table.

Data Table

Mass of mixture	_____
Mass of beaker	_____
Mass of beaker and naphthalene	_____
Mass naphthalene recovered	_____
Mass of filter paper	_____
Mass of filter paper and benzoic acid	_____
Mass benzoic acid recovered	_____
Melting point of recovered benzoic acid	_____
Literature value for melting point of benzoic acid	_____

Data Analysis

1. Calculate the percent of naphthalene and benzoic acid recovered.
2. What are the advantages and disadvantages of using ether as an extraction solvent?
3. What is the practical advantage of having the organic solvent be denser than water when one is extracting an organic compound from an aqueous solution in a separatory funnel?
4. Why must the stopper on top of the separatory funnel be removed before liquid can be withdrawn through the stopcock? (don't forget question 5 on next page)

Experiment 6: The Extraction of Caffeine from Tea Leaves

Introduction

Caffeine is an alkaloid present in tea, coffee, cola nuts, and several other plants. It is a mild stimulant and may be used medically for this purpose. Structurally, caffeine belongs to a class of nitrogen bases called purines. It is a colorless, crystalline solid that melts at 235 – 236°C. It is moderately soluble in water but more soluble in common organic solvents.

In this experiment you will extract caffeine from tea leaves in hot water. This treatment also extracts tannins, another class of compounds found in tea. It will therefore be necessary to separate the two, which you will do with sodium carbonate. Tannins are acidic and are precipitated out by the sodium carbonate. The caffeine is extracted with dichloromethane, which is then evaporated, leaving crude caffeine.

Procedure

1. Measure 30 mL of water and 2.0 g of anhydrous sodium carbonate into a 150 mL beaker and place 2 tea bags in the beaker.
2. Cover the beaker with a watch glass and heat the water to boiling on a hot plate. Once boiling, turn the hot plate to "5" and boil for 20 minutes.
3. While boiling tea, place a 100 mL beaker of water on the hot plate.
4. Decant the hot liquid into a 50 mL Erlenmeyer flask. Wash the tea bags with 10 mL of hot water and add the wash to the tea extract.
5. Cool the tea to room temperature (20 - 25°) using a cool water bath.
6. Transfer the tea extract to a separatory funnel and extract with two 5 mL portions of dichloromethane, combining the dichloromethane extracts in a 25 mL Erlenmeyer flask.
7. Add 0.5 g of anhydrous sodium sulfate to the flask. Allow the flask to stand for 10 minutes, swirling occasionally.
8. Using the analytical balance, mass a 50 mL beaker and decant the contents of the 25 mL Erlenmeyer flask into the beaker. Try to leave as much solid as possible in the flask.
9. Place the beaker in the fume hood to evaporate off the dichloromethane.
10. Determine the mass of the caffeine.

Data Table

Mass of beaker

Mass of beaker and caffeine

Mass of caffeine

Theoretical yield of caffeine

Actual yield of caffeine

Data Analysis

1. Calculate the percent yield of caffeine.
2. How could the purity of the recovered caffeine be determined?
3. Why is sodium carbonate added to the water used to extract the tea?
4. Why was anhydrous sodium sulfate used?

5. Complete the flow diagram for this experiment showing the structures of the materials present at each stage.

Ether solution of benzoic acid and naphthalene

10% NaOH

Lower layer

Upper layer

Concentrated HCl

Experiment 7: Cis to Trans Isomerization

Purpose:

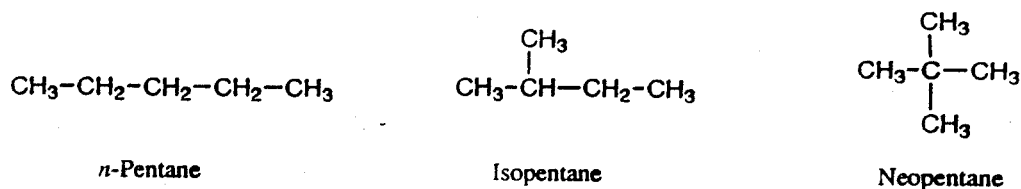
Isomerize the cis double bond in maleic acid to fumaric acid and study the difference in properties of the two isomers.

Background:

Many organic compounds have similar molecular formulas but different physical and chemical properties. These differences are primarily due to the structure of the molecule. When two or more compounds have exactly the same molecular formula, but different properties, they are called isomers. Isomers have different properties because the arrangement, or precise placement of specific atoms within the molecule, differs. Understanding the placement of atoms within a molecule will sometimes lead to a better understanding of its properties and reactivity.

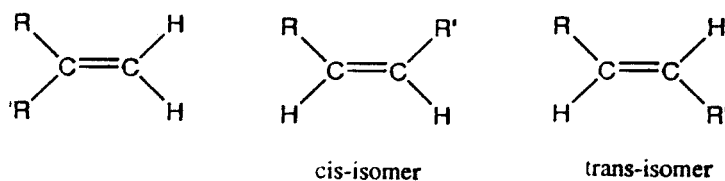
There are two main classes of isomers, structural isomers and stereoisomers. Structural isomers contain the same number and types of atoms but one or more bonds differ. Many structural isomers cannot be converted into one another because bonds have to be broken and reformed which requires a great deal of energy. For example, there are three structural isomers for the molecular formula C_5H_{12} : *n*-pentane, isopentane, and neopentane (Figure 1). These structural isomers are not easily interconverted to one another because a carbon-carbon bond would have to be broken and then reformed.

Figure 1



Stereoisomers have the same number and types of atoms, the same bonding arrangement, but the spatial arrangement of the individual atoms differ. One type of stereoisomers is called geometric isomers because the atoms or groups of atoms assume different geometric positions around a rigid bond or ring of atoms. Carbon-carbon double bonds are very rigid bonds and are common in organic compounds. There are three different arrangements that two different atoms or groups of atoms can take around a carbon-carbon double bond (Figure 2). [In many organic structural drawings, R and R' represent an atom or a group of atoms (e.g., OH, CH_3 , or C_6H_5).]

Figure 2



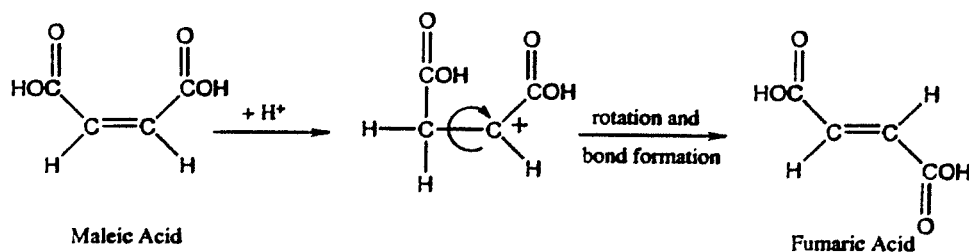
The isomer with the R and R' bonded to the same carbon is a structural isomer of the cis and trans isomers because a carbon-carbon bond would have to be broken to convert it into one of the other two isomers. The cis and trans isomers are stereoisomers because the atoms are identical, are bonded to the same atoms, but their geometry is different. Cis and trans isomers always have a hydrogen and a non-hydrogen atom bonded to each carbon of the double bond. The cis isomer is the isomer where both hydrogens are on one side of the double bond and the trans isomer has the hydrogen atoms on opposite sides.

In general, rotation about a carbon-carbon single bond occurs readily at room temperature, while rotation about carbon-carbon double bonds does not occur. Cis and trans isomers can be interconverted or isomerized under a variety of conditions depending on the molecule. Carbon-carbon double bonds are isomerized using heat, photolysis, or a catalyst. Common catalysts include enzymes, transition metal catalysts, and simple protic acids. Most carbon-carbon double bond isomerization processes involve a carbon-carbon single bond intermediate that can undergo a bond rotation to give either the cis or trans isomer. (Figure 3.)

Trans isomers are generally more stable than the corresponding cis isomer because the large "R" groups are farther apart and steric hinderance is minimized. Steric hinderance is due to the atoms in the "R" groups being too close to one another. Since the trans isomer is usually more stable, it is often the preferred product in an isomerization reaction. However, intramolecular interactions such as hydrogen bonding can sometimes favor the cis isomer. Most isomerization processes give some mixture of cis and trans isomers.

A simple example of a cis-to-trans isomerization is the conversion of maleic acid to fumaric acid. Maleic acid is *cis*-butendioic acid and fumaric acid is *trans*-butendioic acid. A proposed mechanism for the cis-to-trans isomerization reaction is an electrophilic addition of a hydrogen ion to form the carbonium cation followed by rotation about the carbon-carbon single bond to move the two acid groups as far away from each other as possible. Elimination of a hydrogen ion gives the trans isomer (Figure 3).

Figure 3

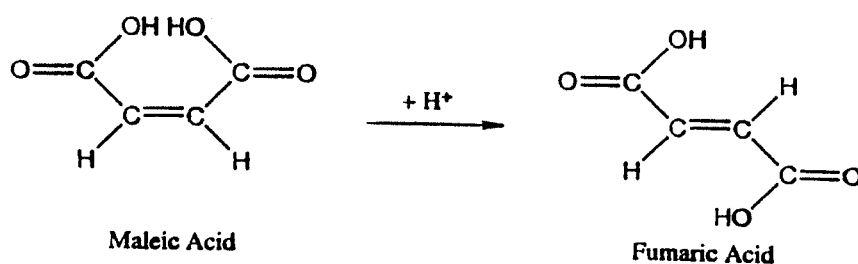


Cis and trans isomers usually differ in properties. Trans isomers generally have more symmetry, a smaller dipole moment, a higher melting point and lower solubility. Cis isomers are not as stable and normally have higher heats of formation (about 1–2 kcal/mol higher). For this reason, cis isomers can often be transformed into the trans isomer by heating. At higher temperatures, enough energy is available to break the carbon-carbon double bond. Rotation about the carbon-carbon single bond can occur and the molecule will prefer to be in a lower energy conformation so when the double bond reforms, the trans isomer is the predominate product.

Maleic Acid Isomerization

Reaction and Physical Properties

Figure 4



	Maleic Acid	Hydrochloric Acid	Fumaric Acid
Amount	0.4 g	3 mL	—
M.W.	116.07	36.46	116.07
Concentration	—	6 M	—
mmol	3	1.8	—

Safety and Chemical Hazards:

Hydrochloric acid is highly toxic by ingestion or inhalation and is severely corrosive to skin and eyes. Avoid all body tissue contact. Maleic acid is moderately toxic by ingestion and a body tissue irritant. Fumaric acid is an eye irritant. Always place the immersion heater in the water before plugging it in. Always wear chemical splash goggles, chemical-resistant gloves and a chemical-resistant apron.

Materials

- Small reaction vial
- Hot water setup
- Ice water setup
- Microtip pipet, 2
- Watch glass or weighing dish
- Small test tube with cork stopper, 2
- Test tube tongs
- Spatula
- Graduated cylinder, 10 mL
- Filter paper
- Copper wire
- Filter setup
- Melting point capillary tubes

Chemicals

- Maleic acid
- Hydrochloric acid, 6M
- Distilled or deionized water
- Cresol red indicator solution, 0.02%

Experimental Procedure

Procedure—Setup

1. Add approximately 300 mL of water to a 400- or 600-mL beaker. Place an immersion heater in the water, plug it in, and allow the water to come to a boil. Do not plug in the immersion heater until after it is placed in the water.
2. Place 0.4 g of maleic acid in a small reaction vial.
3. Add 3 mL of 6 M hydrochloric acid to the reaction vial.
4. Seal the reaction vial and shake vigorously for 1–2 minutes to dissolve the solid. If the solid does not completely dissolve, wait 1–2 minutes and shake again. Any remaining solid will dissolve when the solution is heated. Wrap a piece of copper wire under the cap to form a handle.
5. Using the copper wire handle or tongs, place the reaction vial in the beaker containing boiling water and the immersion heater. If at any time during the reaction a small stream of bubbles begins to flow out of the reaction vial cap, remove the vial from the boiling water, allow it to cool and tighten the cap.
6. After heating for about 10 minutes, a white solid will begin to appear. Observe how the crystals grow and then slowly fall to the bottom of the vial.
7. After a total of 25 minutes, remove the vial from the hot water using tongs or the copper wire handle and place it on the table top. Allow the vial to cool for a minute and then place it in an ice water bath for 2–3 minutes.

Isolate the product using a pipet, vacuum filtration or gravity filtration.

Isolation of Product—Filtration Procedure

8. Remove the copper wire handle. Shake the reaction vial to create a suspension. Quickly pour the suspension into a gravity filtration or vacuum filtration setup.
9. Add 2 mL of cold deionized or distilled water to the reaction vial. Place the cap back on the vial and shake the vial to dislodge any solid product. Pour the water and any solids into the filtration setup.
10. Rinse the solid twice more with 1–2 mL of cold water.
11. If using a vacuum filtration setup, allow the product to dry for a few minutes before transferring it to a watch glass or weighing dish to dry overnight.
12. If using a gravity filtration setup, remove the filter paper when all the filtrate has flowed through the filter paper. Open up the filter paper and place it on a paper towel or larger filter paper to help wick away as much water as possible from the product.
13. Allow the product to dry overnight. Drying can be hastened by placing the product under an incandescent lamp.

Purification and Analysis

14. After the product has dried, determine the mass of the product and calculate percent yield. Record in the data sheet.