

Experiments and Exercises in Organic Chemistry: A Challenge Oriented Approach

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<i>Experiment 2</i>	RECRYSTALLIZATION: PURIFICATION OF SOLIDS <i>The most commonly used technique for the purification of solids, recrystallization, is used to purify vanillin and an unknown.</i>	E2-1
<i>Experiment 3</i>	MELTING POINTS AND RANGES <i>Melting points are used for identification and estimation of purity for vanillin and an unknown</i>	E3-1
<i>Experiment 4</i>	WÖHLER'S SYNTHESIS OF UREA <i>Melting points are used for identification, estimation of purity and to examine Wöhler's synthesis of urea.</i>	E4-1
<i>Experiment 5</i>	SAPONIFICATION USING REFLUX <i>Oil of wintergreen is refluxed in the presence of base to make an acid. The acid is recrystallized and identified.</i>	E5-1
<i>Experiment 6</i>	EXTRACTION: A SEPARATION AND PURIFICATION TECHNIQUE <i>Extraction is used to separate a mixture of acetylsalicylic acid, acetanilide and urea but an "unexpected" result provides a challenge.</i>	E6-1
<i>Experiment 7</i>	IDENTIFICATION OF AN ORGANIC UNKNOWN. I <i>A liquid unknowns is characterized and identified using ir, refractive index, boiling point and density using the Internet as a resource. Students compare properties of unknowns and determine which of the other students in the laboratory have the same unknown.</i>	E7-1
<i>Experiment 8</i>	SIMPLE AND FRACTIONAL DISTILLATION <i>The results of simple and fractional distillation of a mixture of cyclohexane and toluene are compared.</i>	E8-1
<i>Experiment 9</i>	PAPER AND COLUMN CHROMATOGRAPHY <i>Spinach extract is chromatographed and the elutants are analyzed with visible spectroscopy.</i>	E9-1
<i>Experiment 10</i>	IDENTIFICATION OF AN ORGANIC UNKNOWN. II <i>Liquid unknowns are characterized and/or identified using ir, nmr, refractive index, boiling point and density using the Internet as a resource. Students compare properties of unknowns and determine which of the other students in the laboratory have the same unknown. Also the properties listed above and polarimetry will be used to compare two compounds.</i>	E10-1

Experiment 11	VACUUM DISTILLATION, POLYMERIZATION OF STYRENE <i>Styrene is vacuum distilled and polymerized.</i>	E11-1
Experiment 12	BROMINATION OF CINNAMIC ACID <i>The stereochemistry of the addition of bromine to cinnamic acid is determined.</i>	E12-1
Experiment 13	FERMENTATION OF SUCROSE <i>The percentage of ethanol that results after distillation of the fermentation product is determined using gas chromatography and nmr.</i>	E13-1
Experiment 14	ADDITION OF HBr TO ALKENES <i>The orientation of addition of HBr to 2,4,4-trimethyl-1-pentene is studied.</i>	E14-1
Experiment 15	NUCLEOPHILIC SUBSTITUTION <i>Using equimolar amounts of chloride and bromide as nucleophiles, product ratios will be used to elucidate the mechanism of nucleophilic substitution.</i>	E15-1
Experiment 16	ELIMINATION REACTIONS <i>Product structures will be determined to test Saytzev's rule.</i>	E16-1
Experiment 17	NUCLEOPHILIC SUBSTITUTION WITH SACCHARIN <i>The ratio of N to O alkylation is determined.</i>	E17-1
Experiment 18	STEREOCHEMISTRY OF NUCLEOPHILIC SUBSTITUTION <i>The stereochemistry of a sequence of nucleophilic substitutions will be studied.</i>	E18-1
Experiment 19	REDUCTION WITH NaBH₄; STERIC AND CONJUGATION EFFECTS <i>The reduction of camphor, benzoic acid and/or cinnamaldehyde will be studied to determine the steric and conjugation effects on product structure.</i>	E19-1
Experiment 20	GRIGNARD SYNTHESIS <i>A Grignard reagent is prepared and reacted with D₂O. The percentage of deuterium in the product is determined. An additional option is the reaction of the Grignard reagent with carbon dioxide.</i>	E20-1
Experiment 21	ELECTROPHILIC AROMATIC SUBSTITUTION <i>The orientation on anisole is determined using benzoic acid as the electrophile.</i>	E21-1
Experiment 22	FISCHER ESTERIFICATION <i>The product of the reaction of adipic acid with ethanol is vacuum distilled and identified. One of the goals is to determine if a mono or diester is formed.</i>	E22-1
Experiment 23	TAUTOMERISM OF 2,4-PENTANEDIONE <i>Nmr is used to determine the keto-enol equilibrium constant of 2,4-pentanedione in different solvents. Also the rate of deuterium exchange is determined in D₂O.</i>	E23-1
Experiment 24	A DOUBLE ALDOL CONDENSATION <i>The reaction of diphenylacetone and benzil in the presence of base using traditional and/or a microwave oven as the energy source is studied.</i>	E24-1
Experiment 25	IDENTIFICATION OF LIQUID AND SOLID UNKNOWN <i>In addition to the use of techniques discussed earlier, the preparation of KBr disks will be introduced.</i>	E25-1

Experiment 26	MULTISTEP SYNTHESIS: CARBENE CHEMISTRY	E26-1
	<i>An elimination reaction to give an enyne followed by addition of a carbene is performed. Spectroscopy is used to determine if addition occurs at the double or triple bond.</i>	
Experiment 27	ANALYSIS OF A CARBOHYDRATE	E27-1
	<i>A scheme for determining the structure of a carbohydrate will be developed and tested using an unknown sugar.</i>	
Experiment 28	DIELS-ALDER REACTION	E28-1
	<i>The Diels-Alder reaction between cycloheptatriene and maleic anhydride is complicated because of the equilibrium between cycloheptatriene and norcaradiene. Determination of the product structure should determine the structure of the reactive diene.</i>	
Experiment 29	MICROWAVE REACTIONS: A RESEARCH PROJECT	E29-1
	<i>After performing a literature search, students will attempt to run a reaction of their choice using the microwave as the heat source rather than a traditional heat source.</i>	
Experiment 30	SYNTHESIS AND USE OF A DYE	E30-1
	<i>A synthesis of the dye rhodamine is attempted. The identity of the product is checked using visible absorption spectroscopy and qualitatively with fluorescence. The dye properties of rhodamine are tested on a multifabric cloth.</i>	
Experiment 31	SOME POLYMER EXPERIMENTS	E31-1
	<i>A series of polymer experiments including identification of household plastics using ir, cross linking to form slime and plastic worms and the synthesis of nylon are performed.</i>	
Experiment 32	SYNTHESIS OF A TRIBOLUMINESCENT COMPOUND	E32-1
	<i>The determination of the acylation product of anthranilic acid and a study of the triboluminescence of the product.</i>	
Experiment 33	STEAM DISTILLATION: SEPARATION OF BIOACTIVES	E33-1
	<i>Cloves are steam distilled and the structure and herbicidal activity of the oil is determined.</i>	
Experiment 34	A FATTY ACID FROM NUTMEG	E34-1
	<i>A fatty acid is obtained from nutmeg and separated, purified and identified.</i>	
Experiment 35	ACIDITY OF BENZOIC ACIDS: A LINEAR FREE ENERGY GRAPH	E35-1
	<i>The pK_a values of a series of substituted benzoic acids is determined and used to determine the reliability of the Hammett equation.</i>	
Experiment 36	A GREEN WITTIG REACTION	E36-1
	<i>A commercially available ylide is reacted with benzaldehyde and the stereochemistry of the product determined using nmr.</i>	
Experiment 37	ACID CATALYSIS - REACTION OF PINACOL	E37-1
	<i>The acid catalyzed reaction of pinacol will be studied and the structure of the product of the reaction determined.</i>	
Experiment 38	ENANTIOMERS OF IBUPROFEN	E38-1
	<i>An attempt is made to separate racemic ibuprofen into its enantiomers. Ibuprofen is reacted with (S)-(-)-1-phenethylamine to form diastereomers which can then separated as a result of different solubilities in water.</i>	
Experiment 39	SYNTHESIS OF COPPER(II) GLYCINATE	E39-1
	<i>Cis-copper(II) glycinate monohydrate is synthesized and isomerized to trans-copper(II) glycinate monohydrate. The ir spectra of the isomers are compared.</i>	

Experiment 40 **ALKYL PARABENS: SYNTHESIS AND DETECTION IN COSMETICS** E40-1
An alkyl paraben will be synthesized. Paper chromatography of alkyl parabens will be compared to paper chromatography of over-the-counter lotions to determine if parabens are present.

Experiment 41 **SYNTHESIS AND TESTING OF ANTIBIOTICS** E41-1
The synthesis of a penicillin and /or methyl salicylate is performed. References describe methods to perform biological assays of the potential antibiotics.

Exercises

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Exercise 5	Identification of Unknowns	X5-1
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Exercise 7	Choosing Laboratory Procedures	X7-1
Exercise 8	Mechanisms of Reactions	X8-1
Exercise 9	Petrochemicals and Climate	X9-1
Exercise 10	Solvents	X10-1
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Exercise 12	Controversial Chemicals: Issues and Synthesis	X12-1
Exercise 13	Material Selection	X13-1
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Exercise 15	Preparation of a Key for Reaction-Map of Organic Chemistry	X15-1

Appendices

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TECHNIQUES: DISCUSSION AND APPLICATIONS

	Discussion <i>Experiment</i>	Applications <i>Experiment</i>
Vacuum Filtration	1	most experiments
Recrystallization	2	2, 4, 6, 12, 21, 32, 34, 38, 39
Melting Point	3	3, 4, 5, 6, 12, 17, 18, 21, 24, 25, 28, 32, 34, 38
Reflux	5	5, 15, 20, 28, 32, 34, 40
Extraction	6	6, 11, 19, 20, 26, 33, 40
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Visible Spectroscopy	9	9, 24, 30
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Thin Layer Chromatography	9	40
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Steam Distillation	33	33
pH Measurement	35	35

EXPERIMENT: STATUS, TESTING AND IMPROVEMENTS NEEDED

Expt. #	Tested by Instructor	Student Tested	Success Implied by Source	Comments and Suggestions
1			x	While not tested and might need tweaking, these miscibility tests should work. Please report modifications needed.
2	x	x	x	Performed by many students for over 20 years.
3	x	x	x	Performed by many students for over 20 years.
4	x	x	x	Has had limited testing and might need improvement.
5	x	x	x	
6	x	x	x	
7	x	x		Boiling point determinations are problematical.
8	x	x	x	
9	x	x		
10	x	x		
11	x	x	x	
12	x	x	x	Bromination with Br ₂ works fine but questions about product structure remain when pyridinium tribromide used.
13	x	x	x	
14		x		
15			x	Experiment has been performed by students in our lab but untested changes have been made since then.
16	x	x	x	Probably needs further tweaking.
17	x	x	x	
18			x	
19	x	x	x	The camphor experiment has been performed in our laboratories. The other two parts are from references.

Expt. #	Tested by Instructor	Student Tested	Success Implied by Source	Comments and Suggestions
20			x	
21		x	x	
22	x	x	x	
23	x	x		
24	x	x	x	
25	x	x		
26	x	x		
27	x	x	x	
28		x	x	
29				This experiment involves research and possibly the first try at a synthesis using a microwave oven.
30		x	x	
31	x	x		
32	x	x	x	
33			x	
34			x	
35	x			
36			x	
37			x	
38			x	
39	x	x	x	The synthesis has been thoroughly tested but the isomerization is reported from the literature.
40				This experiment has yet to be tested and should provide a good research experience.
41			x	The bioassay might require the cooperation of a microbiologist.

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